Quality Assurance Project Plan (QAPP)

Paerdegat Basin

Brooklyn, New York

Prepared By:
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860-368-5300

Revision 5
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Project No. 129600-1-1101

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INTRODUCTION

This Quality Assurance Project Plan (QAPP) is intended to integrate the technical and quality control aspects of a surface sediment investigation at the Paedegat Basin Site. This QAPP is supplemented by detailed information in the Surface Sediment and Tissue Sampling Work Plan and associeated addendums. This QAPP details the planning processes for collecting data and describes the implementation of the quality assurance (QA) and quality control (QC) activities developed for this program. The purpose of this QAPP is to generate project data that are technically valid and legally defensible. The QAPP consists of four main components:

- Project Management;
- Measurement and Data Acquisition;
- Assessment and Oversight; and
- Data Validation and Usability.

The above components will incorporate QA/QC requirements cited within the following United States Environmental Protection Agency (USEPA) and New York State Department of Environmental Conservation (NYSDEC) documents:

- USEPA *Requirements for Quality Assurance Project Plans*, USEPA QA/R-5, February 2006
- USEPA *Guidance for the Data Quality Objectives Process*, QA/G-4, August 2000
- USEPA, US Department of Defense, and US Department of Energy *Uniform Federal Policy (UFP) for Quality Assurance Project Plans*, Final Version March 2005
- NYSDEC. 2010, *DER-10 Technical Guidance for Site Investigation and Remediation*.
- Analytical Services Protocol (ASP). NYSDEC.

QAPP Worksheet #1 -- Title and Approval Page

Site Name/Project Name: Paedegat Basin Site

Site Location: Paedegat Basin, Brooklyn, New York

Document Title: *QAPP for Paedegat Basin*

Lead Organization: National Grid

Preparer's Name and Organization: Kimberly Bradley, GEI Consultants, Inc.

Preparer's Address, Telephone Number and E-mail Address:

455 Winding Brook Drive, Suite 201, Glastonbury, CT. 06033, kbradley@geiconsultants.com

Preparation Date: 03/19/13, Updated 7/14/2014

Organization	Name	Signature
Investigative Organization's Project Manager	Barry Giroux	
Investigative Organizations Project QC Officer	Brian Skelly	
Lead Organizations Project Manager	William Ryan	

Approval Signatures:_____

Printed Name/Title:_____

Approval Authority:_____

QAPP Worksheet #2 -- QAPP Identifying Information

Site Number/Code:

Operable Unit: Not Applicable

Contractor Name: GEI Consultants, Inc.

Contractor Number: Not Applicable

Contract Title: Not Applicable

Work Assignment Number: Not Applicable

1. **Identify guidance used to prepare QAPP:**
   Uniform Federal Policy for Quality Assurance Project Plans, NYSDEC Analytical Services Protocol (ASP), NYSDEC DER-10

2. **Identify regulatory program:** New York State Department of Environmental Conservation (NYSDEC) Administrative Order on Content

3. **Identify approval entity:** New York State Department of Environmental Conservation (NYSDEC)

4. This QAPP is project Specific.

5. Scoping Sessions occurred in October 2012/January 2013.

6. **List dates and titles of QAPP documents written for previous site work, if applicable:**

7. **List organizational partners (stakeholders) and connection with lead organization:**
   The primary project organizational partners include representatives from National Grid and GEI Consultants, Inc. National Grid will provide project and contract management guidance.

8. **List data users:** New York State Depatement of Environmental Conservation, National Grid, and GEI Consultants, Inc.

9. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion below:

QAPP Worksheet #2 -- QAPP Identifying Information (cont.)

Circle QAPP elements and required information that are not applicable to the project. Provide an explanation in the QAPP.

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
Project Management and Objectives		
2.1 Title and Approval Page	1	- Title and Approval Page
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2.3 Distribution List and Project Personnel Sign Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	3 4	- Distribution List - Project Personnel Sign-Off Sheet
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	5 6 7 8	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table

QAPP Worksheet #2 -- QAPP Identifying Information (cont.)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
2.5 Project Planning/Problem Definition		- Project Planning Session Documentation (including Data Needs tables)
2.5.1 Project Planning (Scoping)	9	- Project Scoping Session Participants Sheet
2.5.2 Problem Definition, Site History, and Background		- Problem Definition, Site History, and Background
	10	- Site Maps (historical and present)
2.6 Project Quality Objectives and Measurement Performance Criteria	11	- Site-Specific PQOs
2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process		
2.6.2 Measurement Performance Criteria	12	- Measurement Performance Criteria Table
2.7 Secondary Data Evaluation		- Sources of Secondary Data and Information
	13	- Secondary Data Criteria and Limitations Table
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2.8.2 Project Schedule		- Project Schedule/Timeline Table
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QAPP Worksheet #2 -- QAPP Identifying Information (cont.)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
Measurement/Data Acquisition		
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QAPP Worksheet #2 -- QAPP Identifying Information (cont.)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
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3.2.2 Analytical Instrument Calibration Procedures	24	- Analytical Instrument Calibration Table
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QAPP Worksheet #2 -- QAPP Identifying Information (cont.)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
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4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses	 30 31	- Assessments and Response Actions - Planned Project Assessments Table - Audit Checklists - Assessment Findings and Corrective Action Responses Table
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QAPP Worksheet #2 -- QAPP Identifying Information (cont.)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
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5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	33 34 35 36	- Verification (Step I) Process Table - Validation (Steps IIa and IIb) Process Table - Validation (Steps IIa and IIb) Summary Table - Usability Assessment
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QAPP Worksheet #3 -- Distribution List

List those entities to which copies of the approved QAPP, subsequent QAPP revisions, addenda, and amendments will be distributed.

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Jane O'Connell	NYSDEC Project Coordinator	NYSDEC DER (R2)	718.482.4599		jhoconne@gw.dec.state.ny.us	
Shaun Bollers	NYSDEC Project Contact	NYSDEC DER (R2)	718.482.4096	718.482.6358	snboller@gw.dec.state.ny.us	
William Ryan	Lead Organizations Project Manager	National Grid	516.545.2586		william.ryan@nationalgrid.com	
Barry Giroux	Investigative Organizations Project Manager	GEI Consultants	860.368.5300	860.368.5307	bgiroux@geiconsultants.com	
David Terry	Investigative Organizations In-house Consultant	GEI Consultants	860.368.5300	860.368.5307	dterry@geiconsultants.com	
Brian Skelly	Investigative Organizations Project Quality Control Officer	GEI Consultants	860.368.5300	860.368.5307	bskelly@geiconsultants.com	
Lorie MacKinnon	Data Validator	GEI Consultants	603. 974.0939		lmackinnon@geiconsultants.com	
Kimberly Bradley	Field Team Leader/Site Safety Officer	GEI Consultants	860.368.5300	860.368.5307	kbradley@geiconsultants.com	

Electronic copies of the final QAPP and related project documents will also be available in the project directory and the project database for personnel named in the organization chart provided as Figure 1 and other personnel who will be assigned to work on the project. Those names will be responsible for distributing the QAPP and related documents to others in their organization.
Note: Per the requirements of DER-10 Chapter 2.4(a)2.ii, the current resumes for Barry Giroux (PM) and Lori ManKinnon (Data Validator) are included in Attachment F.

QAPP Worksheet #4 -- Project Personnel Sign-Off Sheet

Have copies of this form signed by key project personnel from each organization to indicate that they have read the applicable QAPP sections and will perform the tasks as described. Ask each organization to forward signed sheets to the central project file.

Organization: GEI Consultants, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Barry Giroux	Investigative Organizations Project Manager	860.368.5300		
David Terry	Investigative Organizations In-House Consultant	860.368.5300		
Roy Stoecker	Investigative Organizations Biological Senior Consultant	631.751.4600		
Brian Skelly	Investigative Organizations Project Quality Control Officer	860.368.5300		
Lorie MacKinnon	Data Validation/Data Reviewer	603.974.0939		
Mary Beth Billerman	Field Team	631.751.4600		
Kimberly Bradley	Field Team Leader/ Project Safety Officer	860.368.5300		

QAPP Worksheet #4 Project Personnel Sign-Off Sheet (cont.)

Have copies of this form signed by key project personnel from each organization to indicate that they have read the applicable QAPP sections and will perform the tasks as described. Ask each organization to forward signed sheets to the central project file.

Organization: New York Department of Environmental Conservation

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Shaun Bollers	NYSDEC Project Contact	718.482.4096		

QAPP Worksheet #4 Project Personnel Sign-Off Sheet (cont.)

Have copies of this form signed by key project personnel from each organization to indicate that they have read the applicable QAPP sections and will perform the tasks as described. Ask each organization to forward signed sheets to the central project file.

Organization: National Grid

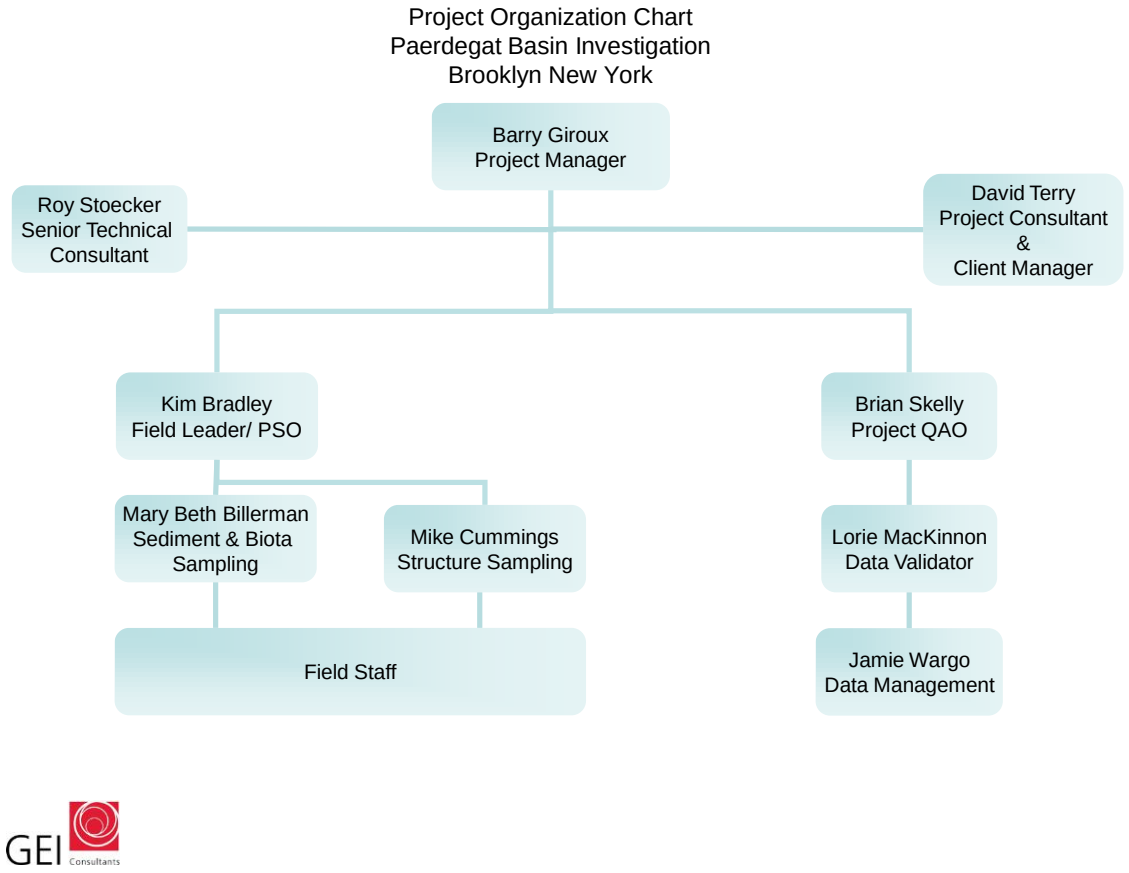
Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
William Ryan	Lead Organizations Project Manager	516.545.2586		

QAPP Worksheet #5 – Project Organizational Chart

Project Organization

The Organization Chart, provided as Figure 1, the description of project organization, and the roles of the team members are summarized below:

Figure 1. Project Organization Chart



Project/Task Organization Overview

The project management team will consist of representatives from National Grid, NYSDEC, and GEI Consultants, Inc. (GEI). GEI will provide technical oversight to the project during the planning and investigation, serve as the primary contractor, bear responsibility for developing and implementing the investigation, and provide project management for the other subcontractors.

Investigation Team Members

This section contains a description of the project organizational structure. The National Grid Project Manager will have contract management with responsibility for the

Paerdegat Basin Investigation. GEI, as the primary contractor, will be responsible for developing and implementing the investigation, and conduct project management for other subcontractors. Additional project team members from other companies may serve as subcontractors to GEI.

Project Manager (PM) – The PM is accountable to the PO throughout the duration of the project. The PM will be the primary point of contact with National Grid. The PM may delegate authority to expedite and facilitate the implementation of the project plan.

The PM is responsible for:

- Coordination with National Grid;
- Budget control;
- Subcontractor performance;
- Project coordination to implement Work Plans;
- Allocation of staffing and resources to implement the QA/QC program and the Health and Safety Plan (HASP); and
- Review of investigation, engineering, and interim reports.

Corporate Health and Safety Manager (CHSM) – The Corporate Health and Safety Manager is responsible for development and implementation of GEI's Health and Safety program. The CHSM serves as the administrator of GEI's Corporate Health and Safety program. The CHSM bears responsibility for:

- Proper training for GEI field personnel;
- Medical clearance of GEI field personnel;
- Field personnel having adequate experience with personal protective equipment;
- Providing guidance on data interpretation;
- Determining levels of worker protection; and
- Directing and assisting the Project Safety Officer (PSO).

Project Safety Officer (PSO) - The PSO is knowledgeable in safety and worker protection techniques as they relate to the project, as instructed and guided by the CHSM. Responsibilities include monitoring daily compliance of work to the HASP, having the ability and authority to make needed changes or additions to the HASP and providing technical assistance to the Project Manager on problems relating to work safety.

The PSO is responsible for the development and set-up of emergency procedures and personnel decontamination procedures. The PSO or designee will complete a daily diary of activities with health and safety relevance. If unsafe work conditions are encountered, the PSO is authorized to stop work. Resolution of all health and safety problems will be coordinated through the Technical PM.

Project Quality Control Officer – The Project QC Officer is responsible for project specific supervision and monitoring of the QA program and reports to the Project Manager. Additional responsibilities include:

- Ensuring that field personnel are familiar with and adhere to proper sample identification, and chain-of-custody procedures;
- Coordinating with the analytical laboratory for the receipt of samples, the reporting of analytical results, and recommending corrective actions to correct deficiencies in the analytical protocol or sampling.

Field Team Leader – The Field Team Leader will serve as the contact person GEI for field investigations and activities. The Field Team Leader will be responsible for the logistics of the field activities. The Field Team Leader will:

- Ensure that proper sampling procedures and field measurement techniques are performed,
- Inspect and replace equipment;
- Prepare daily activity reports;
- Prepare samples (in coordination with the Sample Management Officer) for shipment;
- Coordinate field activities; and
- Schedule sampling and other field activities.

QAPP Worksheet #6 -- QAPP Communication Pathways

Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Approval of Amendments to the QAPP	GEI Consultants, Inc.	Project Quality Control Officer) and Lead Organization's Project Manager	860.368.5300	Obtain initial approval from the Investigative Organization PM and submit documented amendments within 10 working days of initial approval
Document and Records Control	GEI Consultants, Inc.	Investigative Organization Project Manager	860.368.5300	Project Document Preparation and distribution. Document and records control posting procedure implemented within 5 working days of receipt by GEI Consultants, Inc.
Stop Work and Initiation of Corrective action	GEI Consultants, Inc.	Investigative Organization Project Manager	860.368.5300	The PM communicates within 24 hours of stop work to the project organization by phone, with confirming e-mail.
Real time modification, notifications and approval	GEI Consultants, Inc.	Field Team Leader	860.368.5300	Real time modification to the project will require the approval of the Project Quality Officer and PM (or designee) and will be documented using the Field Change Order Form in Attachment E within 5 working days.
Reporting of serious issues	GEI Consultants, Inc.	Project Managers	860.368.5300	Report any serious issues to National Grid and other concerned parties by e-mail or memo.
Meeting Minutes	GEI Consultants, Inc.	Investigative Organization Project Manager	860.368.5300	Post approved meeting minutes or distribute by email within 5 working days of meeting.
Corrective action, assessment finding	GEI Consultants, Inc.	Project Safety Officer/Quality Control Officer	860.368.5300	Problems or negative assessment findings are reported to the PM by e-mail within 3 days.

QAPP Worksheet #7 -- Personnel Responsibilities and Qualifications Table

Name	Title	Organizational Affiliation	Responsibilities	Years of Professional Experience	Education and Experience Qualifications
William Ryan	Project Manager	National Grid	Lead Organization's Project Manager	20 +	MS Public Health-Environmental & Occupational Health Science
Barry Giroux	Project Manager	GEI Consultants, Inc.	Investigative Organization's Project Manager	20+	P.E., BS Civil Engineering
David Terry	Project Manager	GEI Consultants, Inc.	Investigative Organization's In House Consultant	20	PG, MS Geology
Roy Stoecker	Senior Consultant	GEI Consultants, Inc.	Investigative Organization's In House Consultant	20+	PhD Botany
Mary Beth Billerman	Project Manager/Scientist	GEI Consultants, Inc.	Field Team Coordination	9	BS Environmental Science
Kimberly Bradley	Project Scientist	GEI Consultants, Inc.	Field Team Leader/ Site Safety Officer	6+	MS Environmental Sciences
Brian Skelly	Project Manager	GEI Consultants, Inc.	Project Quality Officer	10	MS Environmental Sciences
Kirk Young	Project Manager	Test America	Laboratory Manager	20 +	MS Environmental Sciences

QAPP Worksheet #8 -- Special Personnel Training Requirements Table

Project Function	Specialized Training – Title or Description of Course	Training Provider	Training Date	Personnel/ Groups Receiving Training	Personnel Titles / Organizational Affiliation	Location of Training Records/Certificates
Field Team	Safety and OSHA training and medical monitoring as specified in the HASP; Field Sampling training	GEI Consultants, Inc.	Training dates kept in company/ project training records	All field team members working on Project Properties.	All GEI Consultant and subcontractor personnel working on the Project Properties	GEI Consultant's Project Files; available upon request

QAPP Worksheet #9 -- Project Scoping Session Participants Sheets

Project Team Participants

Project Name: **Paerdegat Basin Spill** **Projected Date(s) of Sampling:** October 2012 **Project Managers:** W. Ryan, National Grid, B. Giroux, GEI Consultants	**Site Name:** Paerdegat Basin Site **Site Location:** Brooklyn, New York

Date of Session: October 15, 2012

Scoping Session Purpose: Scope Investigation

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Jane O'Connell	NYSDEC Project Manager	NYSDEC DER (2)	(718) 482-4599	jhoconne@gw.dec.state.ny.us	Project Manager
William Ryan	Lead Organization Project Manager	National Grid	(516) 545-2586	william.ryan@nationalgrid.com	Project Manager
David Terry	Investigative Organization In House Consultant	GEI Consultants	860-368-5412	dterry@geiconsultants.com	In-House Consultant
Roy Stoecker	Investigative Senior Principal	GEI Consultants	860-368-5414	rstoecker@geiconsultants.com	Senior Scientist

Many of the members of the NYSDEC, National Grid and GEI Consultants investigative team participated on the October 15, 2012 call. Full participation is listed on a contact sheet sent out by Jane O'Connell on October 17, 2012.

Comments/Decisions: The data quality objectives and data needs for the project were developed provided during communications
Action Items: Complete project planning documents (HASP, Work Plan, QAPP)

QAPP Worksheet #9 -- Project Scoping Session Participants Sheets

Project Team Participants

Project Name: **Paerdegat Basin Spill** **Projected Date(s) of Sampling:** To Be February/March 2013 **Project Managers:** W. Ryan, National Grid, B. Giroux, GEI Consultants	**Site Name:** Paerdegat Basin Site **Site Location:** Brooklyn, New York

Date of Session: February 4, 2013

Scoping Session Purpose: Scope Investigation Comments

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Jane O'Connell	NYSDEC Project Manager	NYSDEC DER (2)	(718) 482-4599	jhoconne@gw.dec.state.ny.us	Project Manager
William Ryan	Lead Organization Project Manager	National Grid	(516) 545-2586	william.ryan@nationalgrid.com	Project Manager
Barry Giroux	Investigative Project Manager	GEI Consultants	860-368-5412	bgiroux@geiconsultants.com	In-House Consultant
Roy Stoecker	Investigative Senior Principal	GEI Consultants	860-368-5414	rstoecker@geiconsultants.com	Senior Scientist

Other members of the NYSDECand associated agencies participated in the February 4, 2013 meeing. Full participation is listed on a contact sheet sent out by Roy Stoecker on February 5, 2013 .

Comments/Decisions: The data quality objectives and data needs for the project were developed provided during communications
Action Items: Complete project planning documents (HASP, Work Plan, QAPP)

QAPP Worksheet #10 – Problem Definition

The problem to be addressed by the project:

During abandonment of a 24-inch diameter gas transmission pipeline there was a release of gas condensate from a temporary standpipe pit that was initially installed as part of the gas line abandonment project. The release flowed from the standpipe along a paved road to a nearby stormwater catch basin and then into Paerdegat Basin. The New York Fire Department reportedly also flushed the gas condensate liquids pooled on the ground surface and caused the liquids to flow into the catch basin.

The standpipe pit is located along the southeast side of Seaview Avenue near the intersection with Paerdegat Ave North. During gas line abandonment services on September 27, 2012, cement grout was pumped into the gas line from the west side of Paerdegat Basin near the Hudson River Yacht Club. The gas line runs beneath Paerdegat Basin. At the standpipe pit location, as a result of the cement grout filling operation, residual gas condensate within the gas line was inadvertently forced through a vent installed at the standpipe.

The environmental questions being asked:

Has the release of gas condensate impacted sediments, surface water, biota or structures in Paerdegat Basin?

Observations from any site reconnaissance reports:

An oil-like sheen was visible on the surface water of Paerdegat Basin following the release of gas condensate. Sheen transport was observed on surface water in most areas in the Basin including the area of fixed and free-standing structures in the water (i.e., boats, docks, piers, and bulkheads). Wind action caused the sheen extent to spread primarily toward the headwaters of the basin from the release point. During immediate response actions undertaken following the release, boat hulls were cleaned and wipe tested for PCBs. However, other structures contacting surface water in the Basin such as recreational boat docks, piers, and bulkheads have not been tested for potential impact from the release and it is not known whether or not sediment, surface water and biota have been impacted. Sampling of sediment, surface water, biota, and structures and analysis for PCBs will be undertaken to assess potential impact from the release of the gas condensate fluid.

A synopsis of secondary data or information from site reports:

Upon being notified of the release, National Grid notified the National Response Center (NRC) and the NYSDEC. Emergency response activities were conducted under the direct supervision of the United States Coast Guard (USCG), NYSDEC and NYCDEP. Extensive emergency spill response efforts have been completed to recover the material released and work is ongoing to address residual impacts in upland areas.

The possible classes of contaminants and the affected matrices:

Based upon analytical results of the condensate oil collected from the standpipe, the condensate includes PCBs, volatile organic compounds and semi-volatile organic compounds.

The rationale for inclusion of chemical and nonchemical analyses:

The highest concentration contaminant is PCB Aroclor 1242. Given that this compound is present at the highest concentration, partitions strongly to sediments, is persistent in the environment and can bioaccumulate in biota, PCBs are the contaminants of concern for this Work Plan. Samples will be analyzed for PCB Aroclors. The PCB Aroclor analysis will be done on an expedited turn-around-time basis. If Aroclor 1242 is detected in a sample PCB congener analysis will be performed on that sample.

Project decision conditions:

QAPP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements

Who will use the data?

National Grid, NYSDEC and GEI will use this data.

What will the data be used for?

The data will be used to determine the presence of PCB Arolclor 1242 in the environment.

What type of data are needed (matrix, target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)?

Surface sediment at 42 sampling stations, surface water from five stations, 20 biological tissue (mussels) samples from five sampling stations, and 47 porous surface samples on docks and peirs in Paerdegat Basin. Evaluate each sample for the presence of Aroclor 1242 at an offsite laboratory (Test America Burlington). Sampling techniques are documents in the GEI Field SOPs and Work Plan.

How "good" do the data need to be in order to support the environmental decision?

The data quality objectives (DQOs) for this project have been established in accordance with *Guidance for the Data Quality Objectives Process* (USEPA, 2000), NYSDEC Analytical Services Protocol (ASP), and NYSDEC DER-10, and will provide technically defensible data established for the project as indicators of environmental quality. The DQOs were developed to obtain the necessary data to sufficiently assess risks to human health and the environment. The type, number, and location of samples as well as the sample analysis methods have been prescribed per the Sampling Analysis Plan (2010); therefore, the Quantitation Limits (QLs) achievable by the prescribed analysis will meet the DQOs for this project.

Worksheet 15, Reference and Evaluation Table, summarizes the analytical parameters and the associated Project Action Levels (PALs) and QLs.

How much data are needed (number of samples for each analytical group, matrix, and concentration)?

Surface sediment will be collected at 40 sampling stations, surface water at five sampling stations, 20 biological tissue (mussels) samples at five sampling stations, and 47 porous surface samples on docks and peirs in Paerdegat Basin are proposed.

Where, when, and how should the data be collected/generated?
Data will be collected as soon as possible (target of mid-to-late February 2013), using standard sampling approaches documented in the Work Plan.

Who will collect and generate the data?
GEI will collect environmental monitoring data and samples and tabulate and report field measurements. Test America will analyze samples for chemical analytical parameters and issue reports of analyses. GEI will conduct data validation and usability assessment.

How will the data be reported?
Test America, will submit reports of analyses to GEI, according to the requirements in Worksheet 29, including electronic data deliverables (EDD).

How will the data be archived?
GEI will maintain electronic and hard copies of the data. The data will be submitted to National Grid and NYSDEC. The availability of the data and the length of time the data is available will be at the discretion of the NYSDEC.

QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Sediment
Analytical Group	TOC –Lloyd Kahn
Concentration Level	unknown

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
PB-04	BR-WC-008	Sensitivity	< RL	Method Blank (MB)	A
		Accuracy/Bias	%R (75-125)	Lab Control Sample (LCS)	A
		Precision (lab)	50% RPD	Sample Duplicate (DP)	A
		Accuracy/Bias	%R (75-125)	Matrix Spikes (MS)	A

[2]Reference number from QAPP Worksheet #21 (see Section 3.1.2).

[3]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12 - Measurement Performance Criteria Table (cont.)

Matrix	Sediment
Analytical Group	Grain Size/ Bulk Density
Concentration Level	unknown

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
PB-04	BR-GT-006 BR-GT-018	NA	NA	NA	NA

[1]If information varies within an analytical group, separate by individual analyte.

[2]Reference number from QAPP Worksheet #21 (see Section 3.1.2).

[3]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12 - Measurement Performance Criteria Table (cont.)

Matrix	Sediment/Solid
Analytical Group	PCB Aroclors
Concentration Level	

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
PB-04 PB-09	BR-GC-005	Sensitivity and Accuracy	Less than CRQLs	Equipment Blank	S&A
		Precision	<RPD 50% for duplicate values greater than or equal to 5 times the CRQL	Field Duplicates	S&A
		Accuracy/Bias/ Precision	Per recovery and RPD% requirements of laboratory as listed in SOP	MS/MSD	A
		Accuracy/Bias	Per recovery requirements of laboratory as listed in SOP	LCS	A
		Sensitivity	MDLs	MDLs	A
		Sensitivity	Less than CRQLs	Method Blanks	A
		Completeness	> 90% sample collection, >90% laboratory analysis	Data Completeness Check	S&A

[2]Reference number from QAPP Worksheet #21 (see Section 3.1.2).

[3]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12 - Measurement Performance Criteria Table (cont.)

Matrix	Sediment/Solid
Analytical Group	Volatiles
Concentration Level	Low Level

Sampling Procedure[2]	**Analytical Method/SOP**[3]	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
PB-04 PB-09	BR-MV-006	Sensitivity and Accuracy	Less than CRQLs	Equipment Blank and Trip Blank	S&A
		Precision	<RPD 50% for duplicate values greater than or equal to 5 times the CRQL	Field Duplicates	S&A
		Accuracy/Bias/ Precision	Per recovery and RPD% requirements of laboratory	MS/MSD	A
		Accuracy/Bias	Deuterated Monitoring Compound recoveries per requirements	Deuterated Monitoring Compounds	A
		Sensitivity	MDLs	MDLs	A
		Sensitivity	Less than CRQLs	Method Blanks	A
		Completeness	> 90% sample collection, >90% laboratory analysis	Data Completeness Check	S&A

[2]Reference number from QAPP Worksheet #21 (see Section 3.1.2).

[3]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12 - Measurement Performance Criteria Table (cont.)

Matrix	Sediment/Solid
Analytical Group	PCB Congeners by 1668A
Concentration Level	Low

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
PB-04 PB-09 PB-12	002 - KNOX-ID-0013	Accuracy	50-150% for Toxics/LOCs	LCS	A
		Precision	0-50% RPD for Toxics/LOCs	Field Duplicate	S & A
		Bias/Contamination	No target analyte > EML	Method Blanks	A
		Completeness	90-100% valid data	Data Validation completeness check	S & A

[2]Reference number from QAPP Worksheet #21 (see Section 3.1.2).

[3]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12 - continued

Matrix	Aqueous
Analytical Group	PCB Aroclors
Concentration Level	

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
PB-04	011-SW846 8082 / BR-GC-005	Sensitivity and Accuracy	Less than CRQLs	Equipment Blank	S&A
		Precision	<RPD 30% for duplicate values greater than or equal to 5 times the CRQL	Field Duplicates	S&A
		Accuracy/Bias/ Precision	Per recovery and RPD% requirements of laboratory as listed in SOP	MS/MSD	A
		Accuracy/Bias	Per recovery requirements of laboratory as listed in SOP	LCS	A
		Sensitivity	MDLs	MDLs	A
		Sensitivity	Less than CRQLs	Method Blanks	A
		Completeness	> 90% sample collection, >90% laboratory analysis	Data Completeness Check	S&A

[2]Reference number from QAPP Worksheet #21 (see Section 3.1.2).

[3]Reference number from QAPP Worksheet #23 (see Section 3.2).

Matrix	Tissue
Analytical Group	PCB Aroclors
Concentration Level	

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
PB-08 PB-11	011-SW846 8082 / BR-GC-005	Sensitivity and Accuracy	Less than CRQLs	Equipment Blank	S&A
		Precision	<RPD 30% for duplicate values greater than or equal to 5 times the CRQL	Field Duplicates	S&A
		Accuracy/Bias/ Precision	Per recovery and RPD% requirements of laboratory as listed in SOP	MS/MSD	A
		Accuracy/Bias	Per recovery requirements of laboratory as listed in SOP	LCS	A
		Sensitivity	MDLs	MDLs	A
		Sensitivity	Less than CRQLs	Method Blanks	A
		Completeness	> 90% sample collection, >90% laboratory analysis	Data Completeness Check	S&A

[2]Reference number from QAPP Worksheet #21 (see Section 3.1.2).

[3]Reference number from QAPP Worksheet #23 (see Section 3.2).

Matrix	Solid
Analytical Group	SVOCs
Concentration Level	

Sampling Procedure[2]	**Analytical Method/SOP**[3]	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
PB-04 PB-09	SW-846 8270D/BR-MS-001	Sensitivity and Accuracy	Less than CRQLs	Equipment Blank	S&A
		Precision	<RPD 30% for duplicate values greater than or equal to 5 times the CRQL	Field Duplicates	S&A
		Accuracy/Bias/ Precision	Per recovery and RPD% requirements of laboratory as listed in SOP	MS/MSD	A
		Accuracy/Bias	Per recovery requirements of laboratory as listed in SOP	LCS	A
		Sensitivity	MDLs	MDLs	A
		Sensitivity	Less than CRQLs	Method Blanks	A
		Completeness	> 90% sample collection, >90% laboratory analysis	Data Completeness Check	S&A

[2]Reference number from QAPP Worksheet #21 (see Section 3.1.2).

[3]Reference number from QAPP Worksheet #23 (see Section 3.2).

Matrix	Solid
Analytical Group	TPH - 8015
Concentration Level	

Sampling Procedure[2]	**Analytical Method/SOP**[3]	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
PB-04 PB-09	SW-846 8015D/BR-GC-004_BR-GC-005	Sensitivity and Accuracy	Less than CRQLs	Equipment Blank	S&A
		Precision	<RPD 30% for duplicate values greater than or equal to 5 times the CRQL	Field Duplicates	S&A
		Accuracy/Bias/ Precision	Per recovery and RPD% requirements of laboratory as listed in SOP	MS/MSD	A
		Accuracy/Bias	Per recovery requirements of laboratory as listed in SOP	LCS	A
		Sensitivity	MDLs	MDLs	A
		Sensitivity	Less than CRQLs	Method Blanks	A
		Completeness	> 90% sample collection, >90% laboratory analysis	Data Completeness Check	S&A

[2]Reference number from QAPP Worksheet #21 (see Section 3.1.2).

[3]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #13 – Secondary Data Criteria and Limitations

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use

QAPP Worksheet #14 – Summary of Project Tasks

Sampling Tasks:

Collect surface sediment at 42 sampling stations, surface water at five sampling stations, 20 biological tissue (mussels) samples at five sampling stations, and 47 porus surface samples on docks and peirs in Paerdegat Basin. Evaluate each sample for soil, sediment, and tissue chemistry, specifically Aroclor 1242. All sample custody will be tracked through the use of Chains of Custody (Attachment D).

Work Plan Addendum 1 includes porus surface samples and wipe samples of tide gates, conctrete bulkhead structures and and sanitary manholes in and around Paerdegat Basin.

Determine if PCB Aroclor 1242 is present in soil, sediment, or biological tissue samples.

Analysis Tasks:

Analyze surficial soil and sediment samples from 42 sampling station plus field determined sampling locations for chemistry and characteristics:

- PCBs according to USEPA Method 8082A
- Target compound list volatile organic carbons (VOCs) at seven sample locations
- Total organic carbon (TOC) according to USEPA Method Lloyd Kahn
- Grain Size according to ASTM D422
- Samples will be archived with potential future analysis of PCB congeners according to USEPA Method 1668A
- Percent Moisture

Analyze surface water at five sampling stations for chemistry:

- PCBs according to USEPA Method 8082A
- Samples will be archived with potential future analysis of PCB congeners according to USEPA Method 1668A

Analyze 20 biological tissue (mussels) samples from five sampling stations for chemistry:

- PCBs according to USEPA Method 8082A
- Samples will be archived with potential future analysis of PCB congeners according to USEPA Method 1668A
- Percent moisture
- Percent lipids

Analyze semi-porous materials from 47 sampling stations with structures adjacent to the area of release for chemistry:

- PCBs according to USEPA Method 8082A
- Samples will be archived with potential future analysis of PCB congeners according to USEPA Method 1668A

Soils and semi-porous materials will be reported on a dry-weight basis, while surface water and mussel tissue will be reported on a wet-weight basis.

In addition, Work Plan Addendum 1 includes the following analysis:

Analyze semi-porous materials collected from tide gates, conctrete bulkhead structures, and and wipe tests from sanitary manholes associated with the NYCDEP CSO Facility for:

- PCBs according to USEPA Method 8082A
- Samples will be archived with potential future analysis of PCB congeners according to USEPA Method 1668A

A subset of two samples collected from tide gates and a reference tide gate sample will be analyzed for:

- VOCs according to USEPA Method 8260
- SVOC according to USEPA Method 8270
- Total Petroleum Hydrocarbons according to USEPA Method 8015

Quality Control Tasks:

The analytical and testing laboratories will be required to analyze QC samples. USEPA methods and the other documents and procedures are given in Worksheet 28. Quality control samples are shown by matrix and analytical group in Worksheet 20.

Secondary Data:
Information from previous investigations in the vicinity of Paergant Basin and Jamacia Bay.

Other Data:

Data Management Tasks:
All analytical data will be stored in a database on a server which will be maintained in the GEI office in Glastonbury, CT. All electronic data will be backed up. Hardcopies of data will also be stored in project files. See Worksheet 29 for discussion of data management.

Documentation and Records:
All hardcopy data (field notebooks, photos, hardcopies of Chain of Custody forms) will be stored at the GEI office in Glastonbury, CT and stored in the project files.

Assessment / Audit Tasks
The Work Plan and SOPs will be reviewed prior to the performance of tasks.

Data Review Tasks:
GEI will conduct verification of sampling and laboratory data. Chemical data that is generated will be validated by GEI data validators (see Worksheets 23, 28, 35 and 36).

QAPP Worksheet #15 -- Reference Limits and Evaluation Tables

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Finally, list the published and achievable detection and quantitation limits for each analyte.

Matrix: Sediment
Analytical Group: PCBs - USEPA 8082A
Concentration Level: Low to moderate

Analyte	CAS Number	NYDEC ER-L	NYDEC ER-M	Project Quantitation Limit Goal[2] (mg/Kg)	Laboratory-specific QLs (mg/Kg)	Laboratory-specific DLs (mg/Kg)
PCB-1016	12674-11-2	0.007	NE	0.017	0.017	0.0056
PCB-1221	11104-28-2	NE	NE	0.017	0.017	0.0043
PCB-1232	11141-16-5	NE	NE	0.017	0.017	0.0033
PCB-1242	53469-21-9	NE	NE	0.017	0.017	0.0067
PCB-1248	12672-29-6	0.03	NE	0.017	0.017	0.002
PCB-1254	11097-69-1	NE	NE	0.017	0.017	0.0028
PCB-1260	11096-82-5	0.005	NE	0.017	0.017	0.0024
PCB-1262	37324-23-5	NE	NE	0.017	0.017	0.0015
PCB-1268	11100-14-4	NE	NE	0.017	0.017	0.0014

Notes:
NE = Not established
NYDEC – New York Department of Environmental Conservation Sediment Screening Guidance
ER-L - Effects Range Low
ER-M - Effects Range Medium
ER-L and ER-M from "Sediments Classification Methods Compendium." Long and MacDonald 1992.

QAPP Worksheet #15 -- Reference Limits and Evaluation Tables (cont.)

Matrix: Tissue
Analytical Group: PCBs - USEPA 8082A
Concentration Level: Low to moderate

Analyte	CAS Number			Project Quantitation Limit Goal[2] (ug/Kg)	Laboratory-specific	
					QLs (ug/Kg)	DLs (ug/Kg)
PCB-1016	12674-11-2			34	34	4.6
PCB-1221	11104-28-2			34	34	2.4
PCB-1232	11141-16-5			34	34	4.2
PCB-1242	53469-21-9			34	34	2.8
PCB-1248	12672-29-6			34	34	1.4
PCB-1254	11097-69-1			34	34	6.6
PCB-1260	11096-82-5			34	34	4.4
PCB-1262	37324-23-5			34	34	2.4
PCB-1268	11100-14-4			34	34	0.96

Notes:

QAPP Worksheet #15 -- Reference Limits and Evaluation Tables (cont.)

Matrix: Aqueous
Analytical Group: PCBs - USEPA 8082A
Concentration Level: Low to moderate

Analyte	CAS Number	6 NYCRRs Part 703 SW(AA) (ug/L)	6 NYCRRs Part 703 SW(AC) (ug/L)	6 NYCRRs Part 703 SW(FC) (ug/L)	Project Quantitation Limit Goal[2] (ug/L)	Laboratory-specific	
						QLs (ug/L)	DLs (ug/L)
PCB-1016	12674-11-2	NE	NE	NE	0.5	0.5	0.031
PCB-1221	11104-28-2	NE	NE	NE	0.5	0.5	0.041
PCB-1232	11141-16-5	NE	NE	NE	0.5	0.5	0.065
PCB-1242	53469-21-9	NE	NE	NE	0.5	0.5	0.037
PCB-1248	12672-29-6	NE	NE	NE	0.5	0.5	0.034
PCB-1254	11097-69-1	NE	NE	NE	0.5	0.5	0.044
PCB-1260	11096-82-5	NE	NE	NE	0.5	0.5	0.03
PCB-1262	37324-23-5	NE	NE	NE	0.5	0.5	0.044
PCB-1268	11100-14-4	NE	NE	NE	0.5	0.5	0.02

Notes:

QAPP Worksheet #15 -- Reference Limits and Evaluation Tables (cont.)

Matrix: Sediment
Analytical Group: Volatiles - USEPA 8260B
Concentration Level: Low to moderate

Analyte	CAS Number	NYSDEC ER-L (mg/kg)	NYSDEC ER-M (mg/kg)	Project Quantitation Limit Goal2 (ug/Kg)	MDLs (ug/Kg)	Laboratory-specific QLs (ug/Kg)
1,1,1,2-Tetrachloroethane	630-20-6	NE	NE	5	0.13	5
1,1,1-Trichloroethane	71-55-6	NE	NE	5	0.7	5
1,1,2,2-Tetrachloroethane	79-34-5	NE	NE	5	0.26	5
Freon TF	76-13-1	NE	NE	5	0.33	5
1,1,2-Trichloroethane	79-00-5	NE	NE	5	0.34	5
1,1-Dichloroethane	75-34-3	NE	NE	5	0.41	5
1,1-Dichloroethene	75-35-4	NE	NE	5	0.37	5
1,1-Dichloropropene	563-58-6	NE	NE	5	0.93	5
1,2,3-Trichlorobenzene	87-61-6	NE	NE	5	0.15	5
1,2,3-Trichloropropane	96-18-4	NE	NE	5	0.3	5
1,2,4-Trichlorobenzene	120-82-1	NE	0.0048	5	0.2	5
1,2,4-Trimethylbenzene	95-63-6	NE	NE	5	0.18	5
1,2-Dibromo-3-Chloropropane	96-12-8	NE	NE	5	0.91	5
1,2-Dibromoethane	106-93-4	NE	NE	5	0.15	5
1,2-Dichlorobenzene	95-50-1	NE	0.013	5	0.22	5
1,2-Dichloroethane	107-06-2	NE	NE	5	0.62	5
1,2-Dichloropropane	78-87-5	NE	NE	5	0.29	5
1,3,5-Trimethylbenzene	108-67-8	NE	NE	5	0.18	5

1,3-Dichlorobenzene	541-73-1	NE	NE	5	0.15	5
1,3-Dichloropropane	142-28-9	NE	NE	5	0.19	5
1,4-Dioxane	123-91-1	NE	NE	250	23	250
1,4-Dichlorobenzene	106-46-7	NE	0.11	5	0.23	5
2,2-Dichloropropane	594-20-7	NE	NE	5	0.43	5
2-Butanone	78-93-3	NE	NE	5	1.5	5
2-Chloroethyl vinyl ether	110-75-8	NE	NE	5	0.53	5
2-Chlorotoluene	95-49-8	NE	NE	5	0.14	5
2-Hexanone	591-78-6	NE	NE	5	0.49	5
4-Chlorotoluene	106-43-4	NE	NE	5	0.35	5
4-Isopropyltoluene	99-87-6	NE	NE	5	0.12	5
4-Methyl-2-pentanone	108-10-1	NE	NE	5	0.6	5
Acetone	67-64-1	NE	NE	5	1	5
Benzene	71-43-2	0.34	NE	5	0.71	5
Bromobenzene	108-86-1	NE	NE	5	0.087	5
Bromoform	75-25-2	NE	NE	5	0.2	5
Bromomethane	74-83-9	NE	NE	5	0.74	5
Carbon tetrachloride	56-23-5	NE	NE	5	0.76	5
Chlorobenzene	108-90-7	NE	NE	5	0.076	5
Dibromochloromethane	124-48-1	NE	NE	5	0.11	5
Chloroethane	75-00-3	NE	NE	5	0.38	5
Chloromethane	74-87-3	NE	NE	5	0.26	5
Chloroform	67-66-3	NE	NE	5	0.32	5
cis-1,2-Dichloroethene	156-59-2	NE	NE	5	0.42	5
cis-1,3-Dichloropropene	10061-01-5	NE	NE	5	0.35	5
Cyclohexane	110-82-7	NE	NE	5	0.85	5
Dibromomethane	74-95-3	NE	NE	5	0.27	5

Bromochloromethane	74-97-5	NE	NE	5	0.37	5
Bromodichloromethane	75-27-4	NE	NE	5	0.21	5
Dichlorodifluoromethane	75-71-8	NE	NE	5	0.23	5
Methylene Chloride	75-09-2	NE	NE	5	0.55	5
Ethylbenzene	100-41-4	1.4	NE	5	0.056	5
Hexachlorobutadiene	87-68-3	NE	NE	5	0.17	5
Methyl iodide	74-88-4	NE	NE	5	0.31	5
Isobutyl alcohol	78-83-1	NE	NE	250	49	250
Isopropylbenzene	98-82-8	NE	NE	5	0.077	5
Methyl acetate	79-20-9	NE	NE	5	0.63	5
Methyl t-butyl ether	1634-04-4	NE	NE	5	0.3	5
m&p-Xylene	179601-23-1	NE	NE	5	0.7	5
Naphthalene	91-20-3	NE	NE	5	0.26	5
n-Butylbenzene	104-51-8	NE	NE	5	0.19	5
n-Propylbenzene	103-65-1	NE	NE	5	0.11	5
o-Xylene	95-47-6	NE	NE	5	0.061	5
sec-Butylbenzene	135-98-8	NE	NE	5	0.089	5
Styrene	100-42-5	NE	NE	5	0.1	5
tert-Butylbenzene	98-06-6	NE	NE	5	0.1	5
Tetrachloroethene	127-18-4	0.45	NE	5	0.11	5
Tetrahydrofuran	109-99-9	2.5	NE	50	6.1	50
Toluene	108-88-3	NE	NE	5	0.1	5
trans-1,2-Dichloroethene	156-60-5	NE	NE	5	0.37	5
trans-1,3-Dichloropropene	10061-02-6	NE	NE	5	0.13	5
Trichloroethene	79-01-6	1.6	NE	5	0.48	5
Trichlorofluoromethane	75-69-4	NE	NE	5	0.33	5
Vinyl acetate	108-05-4	NE	NE	5	0.7	5

Vinyl chloride	75-01-4	NE	NE	5	0.3	5
Xylenes, Total	1330-20-7	0.12	NE	5	0.73	5
Carbon disulfide	75-15-0	NE	NE	5	0.31	5
Methylcyclohexane	108-87-2	NE	NE	5	0.17	5

Notes:
NE = Not established
NYDEC - New York Department of Environmental Protection; Sediment Screening Guidance
ER-L - Effects Range Low
ER-M - Effects Range Medium
ER-L and ER-M from "Sediments Classification Methods Compendium." Long and MacDonald 1992.

QAPP Worksheet #15 -- Reference Limits and Evaluation Tables (cont.)

Matrix: Sediment
Analytical Group: PCB Congeners - USEPA 1668A
Concentration Level: Low

Sediment / PCB Congeners — Analyte	CAS	NYDEC ER-L	NYDEC ER-M	Project Quantitation Limit (mg/kg)	MDL (mg/kg)	QL (mg/kg)
2-Chlorobiphenyl	2051-60-7	NE	NE	0.00001	0.000008	0.00001
3-Chlorobiphenyl	2051-61-8	NE	NE	0.00001	4E-07	0.00001
4-Chlorobiphenyl	2051-62-9	NE	NE	0.00001	0.000009	0.00001
2,2'-Dichlorobiphenyl	13029-08-8	NE	NE	0.00002	0.000017	0.00002
2,3-Dichlorobiphenyl	16605-91-7	NE	NE	0.00001	0.000001	0.00001
2,3'-Dichlorobiphenyl	25569-80-6	NE	NE	0.00001	0.000001	0.00001
2,4-Dichlorobiphenyl	33284-50-3	NE	NE	0.00001	0.000002	0.00001
2,4'-Dichlorbiphenyl	34883-43-7	NE	NE	0.00002	0.000012	0.00002
2,5-Dichlorobiphenyl	34883-39-1	NE	NE	0.00001	0.000002	0.00001
2,6-Dichlorobiphenyl	33146-45-1	NE	NE	0.00001	0.000002	0.00001
3,3'-Dichlorobiphenyl	2050-67-1	NE	NE	0.00002	0.00001	0.00002
3,4-Dichlorobiphenyl	2974-92-7	NE	NE	0.00001	0.000003	0.00001
3,4'-Dichlorobiphenyl	2974-90-5	NE	NE	0.00001	0.000003	0.00001
3,5-Dichlorobiphenyl	34883-41-5	NE	NE	0.00001	0.000003	0.00001
4,4'-Dichlorobiphenyl	2050-68-2	NE	NE	0.00001	0.000018	0.00001
2,2',3-Trichlorobiphenyl	38444-78-9	NE	NE	0.00001	0.000004	0.00001
2,2',4-Trichlorobiphenyl	37680-66-3	NE	NE	0.00001	0.000009	0.00001
2,2',5-Trichlorobiphenyl	37680-65-2	NE	NE	0.00002	0.000017	0.00002
2,2',6-Trichlorobiphenyl	38444-73-4	NE	NE	0.00001	0.000004	0.00001
2,3,3'-Trichlorobiphenyl	38444-84-7	NE	NE	0.00002	0.000019	0.00002

2,3,4-Trichlorobiphenyl	55702-46-0	NE	NE	0.00001	0.000005	0.00001
2,3,4'-Trichlorobiphenyl	38444-85-8	NE	NE	0.00001	0.000009	0.00001
2,3,5-Trichlorobiphenyl	55720-44-0	NE	NE	0.00001	0.000005	0.00001
2,3,6-Trichlorobiphenyl	55702-45-9	NE	NE	0.00001	0.000005	0.00001
2,3',4-Trichlorobiphenyl	55712-37-3	NE	NE	0.00001	0.000005	0.00001
2,3',5-Trichlorobiphenyl	38444-81-4	NE	NE	0.00001	0.000008	0.00001
2,3',6-Trichlorobiphenyl	38444-76-7	NE	NE	0.00001	0.000006	0.00001
2,4,4'-Trichlorobiphenyl	7012-37-5	NE	NE	0.00002	0.000019	0.00002
2,4,5-Trichlorobiphenyl	15862-07-4	NE	NE	0.00001	0.000008	0.00001
2,4,6-Trichlorobiphenyl	35693-92-6	NE	NE	0.00002	0.000017	0.00002
2,4',5-Trichlorobiphenyl	16606-02-3	NE	NE	0.00002	0.000015	0.00002
2,4',6-Trichlorobiphenyl	38444-77-8	NE	NE	0.00001	0.000008	0.00001
2',3,4-Trichlorobiphenyl	38444-86-9	NE	NE	0.00001	0.000005	0.00001
2',3,5-Trichlorobiphenyl	37680-68-5	NE	NE	0.00001	0.000007	0.00001
3,3',4-Trichlorobiphenyl	37680-69-6	NE	NE	0.00001	0.000008	0.00001
3,3',5-Trichlorobiphenyl	38444-87-0	NE	NE	0.00001	0.000008	0.00001
3,4,4'-Trichlorobiphenyl	38444-90-5	NE	NE	0.00001	0.000013	0.00001
3,4,5-Trichlorobiphenyl	53555-66-1	NE	NE	0.00001	0.000008	0.00001
3,4',5-Trichlorobiphenyl	38444-88-1	NE	NE	0.00001	0.000009	0.00001
2,2',3,3'-Tetrachlorobiphenyl	38444-93-8	NE	NE	0.00001	0.000012	0.00001
2,2',3,4-Tetrachlorobiphenyl	52663-59-9	NE	NE	0.00001	0.000012	0.00001
2,2',3,4'-Tetrachlorobiphenyl	36559-22-5	NE	NE	0.00001	0.000006	0.00001
2,2',3,5-Tetrachlorobiphenyl	70362-46-8	NE	NE	0.00001	0.000009	0.00001
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	NE	NE	0.00001	0.000019	0.00001
2,2',3,6-Tetrachlorobiphenyl	70362-45-7	NE	NE	0.00001	0.000005	0.00001
2,2',3,6'-Tetrachlorobiphenyl	41464-47-5	NE	NE	0.00001	0.00001	0.00001
2,2',4,4'-Tetrachlorobiphenyl	2437-79-8	NE	NE	0.00001	0.000019	0.00001
2,2',4,5-Tetrachlorobiphenyl	70362-47-9	NE	NE	0.00001	0.000008	0.00001
2,2',4,5'-Tetrachlorobiphenyl	41464-40-8	NE	NE	0.00001	0.000011	0.00001

2,2',4,6-Tetrachlorobiphenyl	62796-65-0	NE	NE	0.00001	0.000006	0.00001
2,2',4,6'-Tetrachlorobiphenyl	68194-04-7	NE	NE	0.00001	0.000005	0.00001
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	NE	NE	0.00001	0.000019	0.00001
2,2',5,6'-Tetrachlorobiphenyl	41464-41-9	NE	NE	0.00001	0.000006	0.00001
2,2',6,6'-Tetrachlorobiphenyl	15968-05-5	NE	NE	0.00001	0.000012	0.00001
2,3,3',4-Tetrachlorobiphenyl	74338-24-2	NE	NE	0.00001	0.000012	0.00001
2,3,3',4'-Tetrachlorobiphenyl	41464-43-1	NE	NE	0.00001	0.00001	0.00001
2,3,3',5-Tetrachlorobiphenyl	70424-67-8	NE	NE	0.00001	0.000012	0.00001
2,3,3',5'-Tetrachlorobiphenyl	41464-49-7	NE	NE	0.00001	0.000013	0.00001
2,3,3',6-Tetrachlorobiphenyl	74472-33-6	NE	NE	0.00001	0.000006	0.00001
2,3,4,4'-Tetrachlorobiphenyl	33025-41-1	NE	NE	0.00001	0.000013	0.00001
2,3,4,5-Tetrachlorobiphenyl	33284-53-6	NE	NE	0.00002	0.000017	0.00002
2,3,4,6-Tetrachlorobiphenyl	54230-22-7	NE	NE	0.00001	0.000006	0.00001
2,3,4',5-Tetrachlorobiphenyl	74472-34-7	NE	NE	0.00001	0.000014	0.00001
2,3,4',6-Tetrachlorobiphenyl	52663-58-8	NE	NE	0.00001	0.000007	0.00001
2,3,5,6-Tetrachlorobiphenyl	33284-54-7	NE	NE	0.00001	0.000019	0.00001
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	NE	NE	0.00001	0.000016	0.00001
2,3',4,5-Tetrachlorobiphenyl	73575-53-8	NE	NE	0.00001	0.000015	0.00001
2,3',4,5'-Tetrachlorobiphenyl	73575-52-7	NE	NE	0.00001	0.000015	0.00001
2,3',4,6-Tetrachlorobiphenyl	60233-24-1	NE	NE	0.00001	0.000011	0.00001
2,3',4',5-Tetrachlorobiphenyl	32598-11-1	NE	NE	0.00002	0.000017	0.00002
2,3',4',6-Tetrachlorobiphenyl	41464-46-4	NE	NE	0.00001	0.000012	0.00001
2,3',5,5'-Tetrachlorobiphenyl	41464-42-0	NE	NE	0.00001	0.000016	0.00001
2,3',5',6-Tetrachlorobiphenyl	74338-23-1	NE	NE	0.00001	0.000016	0.00001
2,4,4',5-Tetrachlorobiphenyl	32690-93-0	NE	NE	0.00002	0.000017	0.00002
2,4,4',6-Tetrachlorobiphenyl	32598-12-2	NE	NE	0.00001	0.000006	0.00001
2',3,4,5-Tetrachlorobiphenyl	70362-48-0	NE	NE	0.00001	0.000017	0.00001
3,3',4,4'-Tetrachlorobiphenyl	32598-13-3	NE	NE	0.00001	0.000017	0.00001
3,3',4,5-Tetrachlorobiphenyl	70362-49-1	NE	NE	0.00001	0.000017	0.00001

3,3',4,5'-Tetrachlorobiphenyl	41464-48-6	NE	NE	0.00001	0.000017	0.00001
3,3',5,5'-Tetrachlorobiphenyl	33284-52-5	NE	NE	0.00001	0.000018	0.00001
3,4,4',5-Tetrachlorobiphenyl	70362-50-4	NE	NE	0.00001	0.000018	0.00001
2,2',3,3',4-Pentachlorobiphenyl	52663-62-4	NE	NE	0.00001	0.000013	0.00001
2,2',3,3',5-Pentachlorobiphenyl	60145-20-2	NE	NE	0.00001	0.000022	0.00001
2,2',3,3',6-Pentachlorobiphenyl	52663-60-2	NE	NE	0.00001	0.000012	0.00001
2,2',3,4,4'-Pentachlorobiphenyl	65510-45-4	NE	NE	0.00001	0.00001	0.00001
2,2',3,4,5-Pentachlorobiphenyl	55312-69-1	NE	NE	0.00001	0.000015	0.00001
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	NE	NE	0.00001	0.000015	0.00001
2,2',3,4,6-Pentachlorobiphenyl	55215-17-3	NE	NE	0.00001	0.000012	0.00001
2,2',3,4,6'-Pentachlorobiphenyl	73575-57-2	NE	NE	0.00001	0.000019	0.00001
2,2',3,4',5-Pentachlorobiphenyl	68194-07-0	NE	NE	0.00001	0.000024	0.00001
2,2',3,4',6-Pentachlorobiphenyl	68194-05-8	NE	NE	0.00001	0.000012	0.00001
2,2',3,5,5'-Pentachlorobiphenyl	52663-61-3	NE	NE	0.00001	0.000012	0.00001
2,2',3,5,6-Pentachlorobiphenyl	73575-56-1	NE	NE	0.00001	0.000022	0.00001
2,2',3,5,6'-Pentachlorobiphenyl	73575-55-0	NE	NE	0.00001	0.000012	0.00001
2,2',3,5',6-Pentachlorobiphenyl	38379-99-6	NE	NE	0.00001	0.000022	0.00001
2,2',3,6,6'-Pentachlorobiphenyl	73575-54-9	NE	NE	0.00001	0.000021	0.00001
2,2',3',4,5-Pentachlorobiphenyl	41464-51-1	NE	NE	0.00001	0.000015	0.00001
2,2',3',4,6-Pentachlorobiphenyl	60233-25-2	NE	NE	0.00001	0.000022	0.00001
2,2',4,4',5-Pentachlorobiphenyl	38380-01-7	NE	NE	0.00001	0.000022	0.00001
2,2',4,4',6-Pentachlorobiphenyl	39485-83-1	NE	NE	0.00001	0.000022	0.00001
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	NE	NE	0.00001	0.000024	0.00001
2,2',4,5,6'-Pentachlorobiphenyl	68194-06-9	NE	NE	0.00001	0.000022	0.00001
2,2',4,5',6-Pentachlorobiphenyl	60145-21-3	NE	NE	0.00001	0.000023	0.00001
2,2',4,6,6'-Pentachlorobiphenyl	56558-16-8	NE	NE	0.00001	0.000023	0.00001
2,3,3',4,4'-Pentachlorobiphenyl	32598-14-4	NE	NE	0.00001	0.000011	0.00001
2,3,3',4,5-Pentachlorobiphenyl	70424-69-0	NE	NE	0.00001	0.000014	0.00001
2,3,3',4',5-Pentachlorobiphenyl	70424-68-9	NE	NE	0.00001	0.000027	0.00001

2,3,3',4,5'-Pentachlorobiphenyl	70362-41-3	NE	NE	0.00001	0.000015	0.00001
2,3,3',4,6-Pentachlorobiphenyl	74472-35-8	NE	NE	0.00001	0.00001	0.00001
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	NE	NE	0.00001	0.000024	0.00001
2,3,3',5,5'-Pentachlorobiphenyl	39635-32-0	NE	NE	0.00001	0.000024	0.00001
2,3,3',5,6-Pentachlorobiphenyl	74472-36-9	NE	NE	0.00001	0.000025	0.00001
2,3,3',5',6-Pentachlorobiphenyl	68194-10-5	NE	NE	0.00001	0.000024	0.00001
2,3,4,4',5-Pentachlorobiphenyl	74472-37-0	NE	NE	0.00001	0.000012	0.00001
2,3,4,4',6-Pentachlorobiphenyl	74472-38-1	NE	NE	0.00001	0.000024	0.00001
2,3,4,5,6-Pentachlorobiphenyl	18259-05-7	NE	NE	0.00001	0.00001	0.00001
2,3,4',5,6-Pentachlorobiphenyl	68194-11-6	NE	NE	0.00001	0.00001	0.00001
2,3',4,4',5-Pentachlorobiphenyl	31508-00-6	NE	NE	0.00001	0.000019	0.00001
2,3',4,4',6-Pentachlorobiphenyl	56558-17-9	NE	NE	0.00001	0.000015	0.00001
2,3',4,5,5'-Pentachlorobiphenyl	68194-12-7	NE	NE	0.00001	0.000015	0.00001
2,3',4,5',6-Pentachlorobiphenyl	56558-18-0	NE	NE	0.00001	0.000021	0.00001
2',3,3',4,5-Pentachlorobiphenyl	76842-07-4	NE	NE	0.00001	0.000012	0.00001
2',3,4,4',5-Pentachlorobiphenyl	65510-44-3	NE	NE	0.00001	0.000015	0.00001
2',3,4,5,5'-Pentachlorobiphenyl	70424-70-3	NE	NE	0.00001	0.000027	0.00001
2',3,4,5,6'-Pentachlorobiphenyl	74472-39-2	NE	NE	0.00001	0.000015	0.00001
3,3',4,4',5-Pentachlorobiphenyl	57465-28-8	NE	NE	0.00001	0.000014	0.00001
3,3',4,5,5'-Pentachlorobiphenyl	39635-33-1	NE	NE	0.00001	0.000028	0.00001
2,2',3,3',4,4'-Hexachlorobiphenyl	38380-07-3	NE	NE	0.00001	0.000012	0.00001
2,2',3,3',4,5-Hexachlorobiphenyl	55215-18-4	NE	NE	0.00001	0.000021	0.00001
2,2',3,3',4,5'-Hexachlorobiphenyl	52663-66-8	NE	NE	0.00001	0.000014	0.00001
2,2',3,3',4,6-Hexachlorobiphenyl	61798-70-7	NE	NE	0.00001	0.000012	0.00001
2,2',3,3',4,6'-Hexachlorobiphenyl	38380-05-1	NE	NE	0.00001	0.000012	0.00001
2,2',3,3',5,5'-Hexachlorobiphenyl	35694-04-3	NE	NE	0.00001	0.000017	0.00001
2,2',3,3',5,6-Hexachlorobiphenyl	52704-70-8	NE	NE	0.00001	0.000013	0.00001
2,2',3,3',5,6'-Hexachlorobiphenyl	52744-13-5	NE	NE	0.00001	0.000011	0.00001
2,2',3,3',6,6'-Hexachlorobiphenyl	38411-22-2	NE	NE	0.00001	0.000009	0.00001

2,2',3,4,4',5-Hexachlorobiphenyl	35694-06-5	NE	NE	0.00001	0.00003	0.00001
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	NE	NE	0.00001	0.000021	0.00001
2,2',3,4,4',6-Hexachlorobiphenyl	56030-56-9	NE	NE	0.00001	0.00002	0.00001
2,2',3,4,4',6'-Hexachlorobiphenyl	59291-64-4	NE	NE	0.00001	0.00002	0.00001
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	NE	NE	0.00001	0.000009	0.00001
2,2',3,4,5,6-Hexachlorobiphenyl	41411-61-4	NE	NE	0.00001	0.000031	0.00001
2,2',3,4,5,6'-Hexachlorobiphenyl	68194-15-0	NE	NE	0.00001	0.000013	0.00001
2,2',3,4,5',6-Hexachlorobiphenyl	68194-14-9	NE	NE	0.00001	0.000017	0.00001
2,2',3,4,6,6'-Hexachlorobiphenyl	74472-40-5	NE	NE	0.00001	0.000032	0.00001
2,2',3,4',5,5'-Hexachlorobiphenyl	51908-16-8	NE	NE	0.00001	0.000018	0.00001
2,2',3,4',5,6-Hexachlorobiphenyl	68194-13-8	NE	NE	0.00001	0.000018	0.00001
2,2',3,4',5,6'-Hexachlorobiphenyl	74472-41-6	NE	NE	0.00001	0.000032	0.00001
2,2',3,4',5',6-Hexachlorobiphenyl	38380-04-0	NE	NE	0.00001	0.000018	0.00001
2,2',3,4',6,6'-Hexachlorobiphenyl	68194-08-1	NE	NE	0.00001	0.000033	0.00001
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	NE	NE	0.00001	0.000011	0.00001
2,2',3,5,6,6'-Hexachlorobiphenyl	68194-09-2	NE	NE	0.00001	0.000024	0.00001
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	NE	NE	0.00001	0.000013	0.00001
2,2',4,4',5,6'-Hexachlorobiphenyl	60145-22-4	NE	NE	0.00001	0.000011	0.00001
2,2',4,4',6,6'-Hexachlorobiphenyl	33979-03-2	NE	NE	0.00001	0.000034	0.00001
2,3,3',4,4',5-Hexachlorobiphenyl	38380-08-4	NE	NE	0.00001	0.000013	0.00001
2,3,3',4,4',5'-Hexachlorobiphenyl	69782-90-7	NE	NE	0.00001	0.000013	0.00001
2,3,3',4,4',6-Hexachlorobiphenyl	74472-42-7	NE	NE	0.00001	0.00001	0.00001
2,3,3',4,5,5'-Hexachlorobiphenyl	39635-35-3	NE	NE	0.00001	0.000035	0.00001
2,3,3',4,5,6-Hexachlorobiphenyl	41411-62-5	NE	NE	0.00001	0.000021	0.00001
2,3,3',4,5',6-Hexachlorobiphenyl	74472-43-8	NE	NE	0.00001	0.000035	0.00001
2,3,3',4',5,5'-Hexachlorobiphenyl	39635-34-2	NE	NE	0.00001	0.000035	0.00001
2,3,3',4',5,6-Hexachlorobiphenyl	74472-44-9	NE	NE	0.00001	0.000021	0.00001
2,3,3',4',5',6-Hexachlorobiphenyl	74472-45-0	NE	NE	0.00001	0.000014	0.00001
2,3,3',5,5',6-Hexachlorobiphenyl	74472-46-1	NE	NE	0.00001	0.000036	0.00001

2,3,4,4',5,6-Hexachlorobiphenyl	41411-63-6	NE	NE	0.00001	0.000012	0.00001
2,3',4,4',5,5'-Hexachlorobiphenyl	52663-72-6	NE	NE	0.00001	0.000011	0.00001
2,3',4,4',5',6-Hexachlorobiphenyl	59291-65-5	NE	NE	0.00001	0.000013	0.00001
3,3',4,4',5,5'-Hexachlorobiphenyl	32774-16-6	NE	NE	0.00001	0.000016	0.00001
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	NE	NE	0.00001	0.000016	0.00001
2,2',3,3',4,4',6-Heptachlorobiphenyl	52663-71-5	NE	NE	0.00001	0.000037	0.00001
2,2',3,3',4,5,5'-Heptachlorobiphenyl	52663-74-8	NE	NE	0.00001	0.000038	0.00001
2,2',3,3',4,5,6-Heptachlorobiphenyl	68194-16-1	NE	NE	0.00001	0.000037	0.00001
2,2',3,3',4,5,6'-Heptachlorobiphenyl	38411-25-5	NE	NE	0.00001	0.000019	0.00001
2,2',3,3',4,5',6-Heptachlorobiphenyl	40186-70-7	NE	NE	0.00001	0.000038	0.00001
2,2',3,3',4,6,6'-Heptachlorobiphenyl	52663-65-7	NE	NE	0.00001	0.000039	0.00001
2,2',3,3',4',5,6-Heptachlorobiphenyl	52663-70-4	NE	NE	0.00001	0.000014	0.00001
2,2',3,3',5,5',6-Heptachlorobiphenyl	52663-67-9	NE	NE	0.00001	0.000022	0.00001
2,2',3,3',5,6,6'-Heptachlorobiphenyl	52663-64-6	NE	NE	0.00001	0.000023	0.00001
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	NE	NE	0.00001	0.000014	0.00001
2,2',3,4,4',5,6-Heptachlorobiphenyl	74472-47-2	NE	NE	0.00001	0.00004	0.00001
2,2',3,4,4',5,6'-Heptachlorobiphenyl	60145-23-5	NE	NE	0.00001	0.00004	0.00001
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	NE	NE	0.00001	0.00004	0.00001
2,2',3,4,4',6,6'-Heptachlorobiphenyl	74472-48-3	NE	NE	0.00001	0.00004	0.00001
2,2',3,4,5,5',6-Heptachlorobiphenyl	52712-05-7	NE	NE	0.00001	0.00004	0.00001
2,2',3,4,5,6,6'-Heptachlorobiphenyl	74472-49-4	NE	NE	0.00001	0.000041	0.00001
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	NE	NE	0.00001	0.000019	0.00001
2,2',3,4',5,6,6'-Heptachlorobiphenyl	74487-85-7	NE	NE	0.00001	0.000023	0.00001
2,3,3',4,4',5,5'-Heptachlorobiphenyl	39635-31-9	NE	NE	0.00001	0.000018	0.00001
2,3,3',4,4',5,6-Heptachlorobiphenyl	41411-64-7	NE	NE	0.00001	0.000023	0.00001
2,3,3',4,4',5',6-Heptachlorobiphenyl	74472-50-7	NE	NE	0.00001	0.000042	0.00001
2,3,3',4,5,5',6-Heptachlorobiphenyl	74472-51-8	NE	NE	0.00001	0.000042	0.00001
2,3,3',4',5,5',6-Heptachlorobiphenyl	69782-91-8	NE	NE	0.00001	0.000014	0.00001
2,2',3,3',4,4',5,5'-Octachlorobiphenyl	35694-08-7	NE	NE	0.00001	0.000017	0.00001

2,2',3,3',4,4',5,6-Octachlorobiphenyl	52663-78-2	NE	NE	0.00001	0.000043	0.00001
2,2',3,3',4,4',5,6'-Octachlorobiphenyl	42740-50-1	NE	NE	0.00001	0.000043	0.00001
2,2',3,3',4,4',6,6'-Octachlorobiphenyl	33091-17-7	NE	NE	0.00001	0.000025	0.00001
2,2',3,3',4,5,5',6-Octachlorobiphenyl	68194-17-2	NE	NE	0.00001	0.00002	0.00001
2,2',3,3',4,5,6,6'-Octachlorobiphenyl	52663-73-7	NE	NE	0.00001	0.00002	0.00001
2,2',3,3',4,5',6,6'-Octachlorobiphenyl	40186-71-8	NE	NE	0.00001	0.000025	0.00001
2,2',3,3',4,5,5',6'-Octachlorobiphenyl	52663-75-9	NE	NE	0.00001	0.000044	0.00001
2,2',3,3',5,5',6,6'-Octachlorobiphenyl	2136-99-4	NE	NE	0.00001	0.000044	0.00001
2,2',3,4,4',5,5',6-Octachlorobiphenyl	52663-76-0	NE	NE	0.00001	0.000044	0.00001
2,2',3,4,4',5,6,6'-Octachlorobiphenyl	74472-52-9	NE	NE	0.00001	0.000045	0.00001
2,3,3',4,4',5,5',6-Octachlorobiphenyl	74472-53-0	NE	NE	0.00001	0.000045	0.00001
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	NE	NE	0.00001	0.000045	0.00001
2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl	52663-79-3	NE	NE	0.00001	0.000045	0.00001
2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl	52663-77-1	NE	NE	0.00001	0.000046	0.00001
Decachlorobiphenyl	2051-24-3	NE	NE	0.00001	0.000015	0.00001

Notes:

NE = Not established

NYDEC - New York Department of Environmental Conservation; Technical Guidance for Screening Contaminated Sediment

ER-L - Effects Range Low

ER-M - Effects Range Medium

ER-L and ER-M from "Sediments Classification Methods Compendium." Long and MacDonald 1992.

Matrix: Sediment
Analytical Group: TOC Lloyd Kahn and Black Carbon
Concentration Level: Unknown

Sediment / Analyte	TOC	CAS	NYDEC ER-L	NYDEC ER-M	Project Quantitation Limit (mg/kg)	MDL (mg/kg)	QL (mg/kg)
TOC		7440-44-0	NE	NE	1000	220	1000

Notes:
NE = Not established
NYDEC - New York Department of Environmental Conservation; Technical Guidance for Screening Contaminated Sediment
ER-L - Effects Range Low
ER-M - Effects Range Medium
ER-L and ER-M from "Sediments Classification Methods Compendium." Long and MacDonald 1992.

Matrix: Solid/Sediment							
Analytical Group: Semivolatile Organic Compounds							
Analyte	**CAS Number**	**NYDEC ER-L**	**NYDEC ER-M**	**Project Quantitation Limit Goal2 (ug/Kg)**	**Laboratory-specific**		
					QLs (ug/Kg)	**LODs (ug/Kg)**	**DLs (ug/Kg)**
1,1'-Biphenyl	92-52-4	NE	NE	330	330	330	15
1,2,4,5-Tetrachlorobenzene	95-94-3	NE	NE	330	330	330	40
2,2'-oxybis[1-chloropropane]	108-60-1	NE	NE	330	330	330	16
2,3,4,6-Tetrachlorophenol	58-90-2	NE	NE	330	330	330	37
2,4,5-Trichlorophenol	95-95-4	NE	NE	830	830	830	34
2,4,6-Trichlorophenol	88-06-2	NE	0.003	330	330	330	34
2,4-Dichlorophenol	120-83-2	NE	0.006	330	330	330	33
2,4-Dimethylphenol	105-67-9	NE	0.005	330	330	330	52
2,4-Dinitrophenol	51-28-5	NE	NE	830	830	830	140
2,4-Dinitrotoluene	121-14-2	NE	NE	330	330	330	24
2,6-Dinitrotoluene	606-20-2	NE	NE	330	330	330	30
2-Chloronaphthalene	91-58-7	NE	NE	330	330	330	43
2-Chlorophenol	95-57-8	NE	NE	330	330	330	34
2-Methylnaphthalene	91-57-6	NE	0.008	330	330	330	15
2-Methylphenol	95-48-7	0.07	0.67	330	330	330	40
2-Nitroaniline	88-74-4	NE	NE	830	830	830	37
2-Nitrophenol	88-75-5	NE	NE	330	330	330	37
3 & 4 Methylphenol	15831-10-4	NE	NE	670	670	670	74
3,3'-Dichlorobenzidine	91-94-1	NE	NE	330	330	330	48
3-Nitroaniline	99-09-2	NE	NE	830	830	830	38
4,6-Dinitro-2-methylphenol	534-52-1	NE	NE	830	830	830	97
4-Bromophenyl phenyl ether	101-55-3	NE	NE	330	330	330	18
4-Chloro-3-methylphenol	59-50-7	NE	NE	330	330	330	40
4-Chloroaniline	106-47-8	NE	NE	330	330	330	32
4-Chlorophenyl phenyl ether	7005-72-3	NE	NE	330	330	330	15

4-Nitroaniline	100-01-6	NE	NE	830	830	830	32
4-Nitrophenol	100-02-7	NE	NE	830	830	830	84
Acenaphthene	83-32-9	0.016	0.5	330	330	330	13
Acenaphthylene	208-96-8	0.044	0.64	330	330	330	15
Acetophenone	98-86-2	NE	NE	330	330	330	16
Anthracene	120-12-7	0.085	1.1	330	330	330	14
Atrazine	1912-24-9	NE	NE	330	330	330	16
Benzaldehyde	100-52-7	NE	NE	330	330	330	16
Benzo[a]anthracene	56-55-3	0.261	1.6	330	330	330	13
Benzo[a]pyrene	50-32-8	0.43	1.6	330	330	330	12
Benzo[b]fluoranthene	205-99-2	NE	1.8	330	330	330	23
Benzo[g,h,i]perylene	191-24-2	0.17	NE	330	330	330	13
Benzo[k]fluoranthene	207-08-9	0.24	NE	330	330	330	30
Bis(2-chloroethoxy)methane	111-91-1	NE	NE	330	330	330	15
Bis(2-chloroethyl)ether	111-44-4	NE	NE	330	330	330	15
Bis(2-ethylhexyl) phthalate	117-81-7	0.18216	2.64651	330	330	330	21
Butyl benzyl phthalate	85-68-7	NE	0.063	330	330	330	20
Caprolactam	105-60-2	NE	NE	330	330	330	35
Carbazole	86-74-8	0.384	2.8	330	330	330	15
Chrysene	218-01-9	0.063	0.26	330	330	330	18
Dibenz(a,h)anthracene	53-70-3	NE	NE	330	330	330	11
Dibenzofuran	132-64-9	NE	0.006	330	330	330	16
Diethyl phthalate	84-66-2	NE	NE	330	330	330	15
Dimethyl phthalate	131-11-3	NE	0.058	330	330	330	15
Di-n-butyl phthalate	84-74-2	NE	NE	330	330	330	15
Di-n-octyl phthalate	117-84-0	0.6	5.1	330	330	330	23
Fluoranthene	206-44-0	0.019	0.54	330	330	330	12
Fluorene	86-73-7	0.02	NE	330	330	330	15
Hexachlorobenzene	118-74-1	NE	0.0013	330	330	330	46
Hexachlorobutadiene	87-68-3	NE	NE	330	330	330	35

Hexachlorocyclopentadiene	77-47-4	NE	0.073	330	330	330	65
Hexachloroethane	67-72-1	0.2	NE	330	330	330	34
Indeno[1,2,3-cd]pyrene	193-39-5	NE	NE	330	330	330	14
Isophorone	78-59-1	0.16	2.1	330	330	330	42
Naphthalene	91-20-3	NE	NE	330	330	330	15
Nitrobenzene	98-95-3	NE	NE	330	330	330	41
N-Nitrosodi-n-propylamine	621-64-7	NE	NE	330	330	330	46
N-Nitrosodiphenylamine	86-30-6	NE	0.017	387	387	387	16
Pentachlorophenol	87-86-5	0.24	1.5	830	830	830	68
Phenanthrene	85-01-8	NE	0.13	330	330	330	12
Phenol	108-95-2	0.665	2.6	330	330	330	40
Pyrene	129-00-0	0.016	0.5	330	330	330	14
2,4,6-Tribromophenol (Surrogate)	118-79-6						
2-Fluorobiphenyl (Surrogate)	321-60-8						
2-Fluorophenol (Surrogate)	367-12-4						
Nitrobenzene-d5 (Surrogate)	4165-60-0						
Phenol-d5 (Surrogate)	4165-62-2						
Terphenyl-d14 (Surrogate)	1718-51-0						

Notes:

NE = Not established

NYDEC – New York Department of Environmental Conservation Sediment Screening Guidance

ER-L - Effects Range Low

ER-M - Effects Range Medium

ER-L and ER-M from "Sediments Classification Methods Compendium." Long and MacDonald 1992.

Matrix: Solid/Sediment
Analytical Group: TPH

Analyte	CAS Number	NYSDEC ER-L	NYSDEC ER-M	Project Quantitation Limit Goal2 (ug/Kg)	Laboratory-specific		
					QLs (ug/Kg)	LODs (ug/Kg)	D (ug
Gasoline Range Organics (C6-C10)	8006-61-9	NE	NE	1250	1250	330	3
Diesel Range Organics (C10-C28)	STL00143	NE	NE	6700	6700	340	3
a,a,a-Trifluorotoluene (Surrogate)	98-08-8						

Notes:
NE = Not established
NYDEC – New York Department of Environmental Conservation Sediment Screening Guidance
ER-L - Effects Range Low
ER-M - Effects Range Medium
ER-L and ER-M from "Sediments Classification Methods Compendium." Long and MacDonald 1992.

QAPP Worksheet #16 – Project Schedule/ Timeline Table

Activities	Organization	Anticipated Time Frame of Initiation	Anticipated Time Frame of Completion	Deliverable	Deliverable Due Date
Development Sampling Plan, Investigation Scope of Work, and QAPP	GEI	October 2012, Revised March 2013	March/April 2013	Final WP, QAPP	March/April 2013
Sampling Event	GEI	March -April 2013	April 2013	Interm Data Report	May 2013
Laboratory Analyses – All Events	Laboratories	Upon sample receipt	Dependant on Individual Laboratory Schedules	Laboratory Deliverable	Approximately 1 month after sample receipt
Work Plan Addendum 1	GEI	April 2014	July 2014	WP Addedum, QAPP Rev.,	July 2014
Work Plan Addendum 1 Sampling Activities	GEI	August 2014	September 2014	Data report	October 2014

QAPP Worksheet #17 -- QAPP Sampling Design and Rationale

Describe and provide a rationale for choosing the sampling approach:

This Work Plan was prepared by GEI Consultants, Inc. (GEI) for National Grid and outlines the scope of work for an investigation to determine if a release of gas condensate has impacted sediments, surface water, biota or structures in Paerdegat Basin. Paerdegat Basin is located in Brooklyn, New York as indicated Figure 1 in the current Work Plan.

This plan is based upon a field inspection conducted by GEI on October 16, 2012 and is also responsive to the NYSDEC comments of October 15, 2012, amendments from the GEI conference call of October 17, 2012, and review letter from NYSDEC received January 14, 2013 and follow up meeting held February 4, 2013. The scope of work includes the following:

- Intertidal sediment sampling
- Subtidal sediment and surface water sampling from shoreline structures including the CSO wall and the mouth of the Basin
- Subtidal sediment and surface water sampling from the survey boat; weather and ice permitting
- Biota sampling (mussels only)
- Structure sampling (piles, docks, and bulkheads)

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations):

The proposed field investigations include the collection of sediment at 42 sampling stations, 20 biological tissue (mussels) samples from five stations, surface water from five stations, and 47 porous structure samples on docks, piers and bulkheads in Paerdegat Basin. All samples will be analyzed for PCBs. Proposed sample locations are indicated on Figure 2 in the Work Plan. Actual sample locations may have to be adjusted based on field conditions at the time the field work is done. GEI will conduct the sampling activities for all media on behalf of National Grid.

Shoreline Survey and Contingent Sediment Sampling

The NYSDEC requested that a detailed shoreline survey be performed to document the potential presence of oily residues that may remain following the response action. Where possible, that survey will be performed by GEI staff walking the shoreline. This will likely be possible in areas such as the marinas and other easily accessible areas. However, because much of the shoreline consists of wetlands and mud-flat areas (including constructed wetlands currently being developed), large sections of the shoreline may not be accessible on foot. Therefore, GEI will also conduct a visual shoreline survey by boat of the entire shoreline

of the Basin. The objective will be to document presence/absence of visible product or oily residues, primarily by sheen or possibly by staining of the *Spartina* shoots in the restoration areas. The survey will be conducted around low tide. We will notify staff at NYSDEC's Division of Fish, Wildlife and Marine Resources so that they can participate in the survey if they are available.

GEI will document the shoreline conditions and observations of any sheen or oily residues using video photographs and field notes. GPS coordinates of any observed oily residues will be recorded. It is likely that other petroleum-related releases un-related to the gas condensate liquid release have caused oily residue or sheen impacts in the intertidal zone of the basin. Therefore, regardless of potential source, GEI will adjust its proposed sample locations to include the collection of soil/sediment samples for laboratory analyses from any accessible area of oily residue or sheen to evaluate the potential for the observed impact to be related to the gas condensate release. The documented observations will be included in GEI's sampling report.

Sediment, Surface Water and Biota Sampling

The sampling plan will be implemented over an approximate one week period. The scope of work for field investigation includes the following:

- Intertidal surface sediment sampling: this effort will involve 14 stations along the *Spartina* restoration side and other areas accessible on foot.
- Subtidal surface sediment sampling from shoreline structures: this will include 12 subtidal stations. This includes four from the CSO wall and floating docks, seven from marina docks and one at the mouth of the basin. A surface water sample will also be collected from the station located near the outfall.
- Subtidal surface sediment sampling from the survey boat (weather and ice permitting): this will consist of 14 stations using the research vessel including three reference stations to be taken in Jamaica Bay. Surface water samples will be collected from three of these stations within the basin and one from Jamaica Bay.
- Biota sampling for mussels: up to 20 samples from five stations, however, the actual number of stations will depend upon availability of mussels.

Intertidal Sample Locations

The objective of this sampling task is to characterize the extent and concentrations of PCBs in the intertidal zone. The study zone will range from the mean low water (MLW) to the mean high water line with stations as shown in Figure 2 in the Work Plan. Surficial sediment will be collected from eighteen stations with stainless steel trowels cleaned between stations as described in the QAPP. Single use, disposable sampling equipment may also be used to collect sample aliquots. Locations will be recorded

utilizing GPS. Sediment samples will be photographically recorded. Complete chain of custody records will be maintained.

Subtidal Sediment and Surface Water Sampling From Shoreline Structures

The objective of this element is to characterize the extent and concentrations of PCBs in the subtidal zones. Sediment samples will be collected from the 12 stations using a petite PONAR sampling device. Three surface water samples will be collected at locations shown on Figure 2 in the Work Plan. Limited surface water sampling is porposed due to the time that has elapsed since the initial release, the low solubility of PCBs, and because the analysis of samples conducted immediately after the release did not detect any PCBs. This particular protocol is for nearshore collections under and around hard structures such as floating docks and bulkheads. The exact sample locations may be modified in the field should obstructions be encountered. The PONAR sampler will be deployed manually at the sampling station until sufficient sediment sample volume is obtained for the desired analytical requirements (see below). Based on input from NYSDEC the primary focus is to characterize the upper 1-inch of the sediment surface. As a result efforts will be made to obtain sediment material from the top fraction of material in each retrieved PONAR sampler. The PONAR and all re-usable sampling equipment will be decontaminated between sample locations. Single use, disposable sampling equipment may also be used to collect sample aliquots from the PONAR device and transfer the sediments to the laboratory sample containers. The sediment sampling locations will be recorded with a GPS and the sediment samples will be photographically recorded. Complete chain of custody records will be maintained.

Subtidal Sediment and Surface Sampling Utilizing the Survey Vessel

Sediment samples will be collected from 15 stations using a petite PONAR sampling device and four direct surface water samples will be collected as shown on Figure 2 (within basin) and Figure 3 (within Jamaica Bay) in the Work Plan. GEI's sampling vessel, the RV Kingfisher will be used to access the offshore stations for sample collections (weather and ice conditions permitting). The exact sample locations may be modified in the field should obstructions be encountered. The PONAR sampler will be deployed at the sampling station until sufficient sediment sample volume is obtained for the desired analytical requirements (see below). Collection strategy will again be focused on characterizing the upper 1-inch of the sediment surface.Efforts will be made to obtain sediment material from the top fraction of material in each retrieved PONAR sampler. The PONAR and all re-usable sampling equipment will be decontaminated between sample locations. Single use, disposable sampling equipment may also be used to collect sample aliquots from the PONAR device and transfer the sediments to the laboratory sample containers. Surface water samples will be directly sampled. The sampling locations will be recorded with an on-board GPS and the sediment samples will be photographically recorded. Complete chain of custody records will be maintained.

Biota Tissue Sampling for Mussels

Mussels are non-mobile, therefore good indicators for bioaccumulation of metals and/or persistent organics in the water column or sediment. Mussels will be collected if they are encountered in the intertidal zones (e.g., attached to *Spartina* stubs), from the floating docks and piers at the marinas in Paerdegat Basin, and on hard surfaces at the head or mouth of the basin. Priority will be given to attempt and locate up to four target stations in intertidal regions upstream and/or downstream of the outfall. The initial shoreline survey will provide a better understanding of the feasibility of these sample locations; however, it is important to note the exact locations of biota sampling stations will be highly dependent on field conditions and the availability of mussels. At least one target station will be located at one of the marinas on the east side of the basin near the outfall. If mussels are not available in intertidal regions, another potential target station will be located at marina on the west side of the basin.

The total weights of mussels collected per station will be recorded. All biota samples will be wrapped in aluminum foil, packed in plastic bags and immediately placed on ice in coolers for same day shipment to the analytical laboratory. All samples will be processed in the analytical laboratory. The mussels will be shucked and the soft tissue retained for analysis. The sampling locations will be recorded with a GPS and photographically recorded. Complete chain of custody records will be maintained. If sufficient weight of samples are collected (e.g., >20 grams for each sample type) for all 20 samples prior to the last day, the sampling for that effort may be discontinued. If sample mass from each station is insufficient for laboratory requirements, samples will be composited and submitted on an area basis as opposed to individual stations.

Structure Sampling

Sample Locations and Rationale

The objective of this element is to determine if PCBs are present at or above the USEPA clean up criteria of 1 mg/kg in structures that could have been impacted by the spill. Proposed sample locations are indicatedon Figure 4 in the Work Plan. Floating docks, piers and bulkheads in the area of investigation in the Basin will be inspected for oil-like staining where access is permitted by the owner. Proposed sample locations will be adjusted to to locations where oil-staining is observed with suspected impact from the gas condensate release. If no oil-staining is observed or not suspected to be related to the recent release of gas condensate, samples of the outer surface of the structures in contact with the surface water will be collected at the scum line as as indicated in Figure 3 in the Work Plan. It may not be possible to collect samples from some locations for safety reasons or due to accessibility.

A summary of the proposed structure sample locations is presented in the following table.

Location	Number of Samples	Sample Media
Docks and Piers		
Hudson River Yacht Club	10	Wood and Styrofoam
Midget Squadron Marina	18	Wood and Styrofoam
Kayak Club	3	Wood and Styrofoam
Diamond Point Club	6	Wood and Styrofoam
Paerdegat Squadron	4	Wood and Styrofoam
Canarsie Athletic Club	4	Wood and Styrofoam
Bulkheads		
CSO Outfall Structure Bulkhead	2	Concrete

Sampling Methods

Samples of wood and Styrofoam materials will be collected from the surface using USEPA sampling protocols for collection of porous materials for PCB analysis (USEPA, May 2011). Wood chisels or handheld rotary drill with a corer attachment will be used to collect surface samples from the porous material. The depth of sampling will not exceed 1/2 inch into the surface sampled. Samples of wooden and Styrofoam dock materials will be collected and analyzed as discrete samples. Samples collected from wooden piers which are exposed to daily tidal fluctuations, will be comprised of a composite of three subsamples collected during low tide from three locations representative of depth approximate heights of the low tide, mid tide and high tide. Samples of dock material will be collected from the side wall of the dock while kneeling on the dock surface.

The concrete bulkhead at the CSO outfall is considered porous materials and samples of it will be collected using a

vibratory hammer drill and core as described in USEPA sampling protocols (May, 2011).

Some samples may require the use of a boat for added safety during sample collection. All samples will be placed in laboratory provided sampling jars for PCB analysis.

Proposed Sample Analysis

Based upon analytical results of the condensate oil collected from the standpipe , the condensate included PCBs, volatile organic compounds (VOCs) and semivolatile organic compounds. The highest concentration contaminant is PCB Aroclor 1242. Given that this compound is present at the highest concentration, partitions strongly to sediments, is persistent in the environment and can bioaccumulate in biota, PCBs are the primary contaminants of concern for this Work Plan. All samples will be analyzed for PCB Aroclors and 20% of the sediment samples will also be analyzed for VOCs. The analyses will be done on an expedited turn-around-time basis.

Sediment samples will be analyzed for the following parameters:

- PCBs according to USEPA Method 8082A
- Target compound list VOCs according to USEPA Method 8260B at seven of the sample locations
- Total organic carbon (TOC) according to USEPA Method Lloyd Kahn
- Grain Size according to ASTM D422
- Samples will be archived with the potential for future analysis of PCB congeners according to USEPA Method 1668A for forensic evaluations
- Percent Moisture

Surface water, biological tissue and porous structural material samples will be analyzed for the following parameter:

- PCBs according to USEPA Method 8082A
- Samples will be archived with potential future analysis of PCB congeners according to USEPA Method 1668A for forensic evaluations
- Percent moisture
- Percent lipid (for biological tissue samples only).

Soils and semi-porous materials will be reported on a dry-weight basis, while surface water and mussel tissue will be reported on a

wet-weight basis.

QAPP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table

Locations	Sample ID	Matrix	Depth (inch)	Analytical Group	Concentration Level	Number of Samples[1]	Sampling SOP Reference[2]	Rationale for Sampling Location
Stations SD-001 through SD-042	NG-PB-SD-001 through NG-PB-042	Sediment	0-1	PCBs Aroclors*, TOC,Grain size, percent moisture, VOCs (subset of 7 samples)	Unknown	42	PB-04	See Work Plan
Stations T-001 through T-005	NG-PB-T-001 through PB-T-020	Tissue (mussels)	NA	Tissue PCB Aroclors*, percent moisture, percent lipid	Unknown	20	PB-10	See Work Plan
Stations SW-001 through SW-005	NG-PB-SW-001 through PB-SW-005	Surface Water	0-5	PCBs Aroclors*	Unknown	5	PB-08	See Work Plan
Stations PS-001 through PS-047	NG-PB-PS-001 through PB-PS-047	Porous solid	0-0.5	PCBs Aroclors*	Unknown	47	PB-09	See Work Plan
Stations PS-048 through PS-075	NG-PB-PS-048 through PB-PS-075	Porous solid	0-0.5	PCBs Aroclors*, VOCs, SVOCs, TPH (subset of 3 samples)	Unknown	28	PB-09	See Work Plan Addendum 1
Stations MH-001 through MH-004	NG-PB-MH-001 through PB-MH-004	Man hole wipe test	Surface	PCBs Aroclors*	Unknown	4	PB-12	See Work Plan Addendum 1

Notes:

[1]Does not include quality assurance/ quality control samples. Quality assurance/ quality control samples will include blind duplicate samples, matrix spike/ matrix spike duplicate samples, and equipment rinsate blank samples. These samples will be completed on a frequency of 1 per 20 samples per matrix or once per week of sampling per matrix.

[2]Appropriate letter or number from the Project Sampling SOP References table (Worksheet #21).

* Samples will be archived with potential future analysis of PCB congeners according to USEPA Method 1668A

QAPP Worksheet #19 – Analytical SOP Requirements Table

For each matrix, analytical group and concentration level, list the analytical and preparation method/SOP and associated sample volume, container specifications, preservation requirements, and maximum holding time.

Matrix	**Analytical Group**	**Concentration Level**	**Analytical and Preparation Method / SOP Reference**[1]	**Sample Volume**	**Containers** (number, size, and type)	**Preservation Requirements** (chemical, temperature, light protected)	**Maximum Holding Time** (preparation / analysis)
Solid/Sediment	PCBs Aroclors	unknown	Method SW846 8082A BR-GC-005(A), BR-EX-027(P)	50 g	4 oz. glass jar	Cool to 4 ± 2 C	14 days for extraction and 40 days for analysis.
Sediment	TOC Lloyd Kahn	unknown	Lloyd Khan / 006	10 g	Taken from 32 oz glass jar	4 C	14 days
Sediment	Grain Size	unknown	ASTM D422 / 007	500 g	Taken 32 oz glass jar	4 C	NA
Sediment	PCB Congeners	Low	1668A / 002	4 oz	8 oz. amber glass jar	<6 °C in transit to lab. Lab storage at < -10 °C	Up to 1 year when stored frozen and in the dark
Sediment/Solids	VOCs	Low	Method 8260B BR-MV-006(A), BR-MV-007(P)	(2) 40 mL glass vials	2 x 5 grams	To Lab: Cool 4±2°C At lab: Freeze -20 ± 10°C 5 mL of VOA Free water	48Hours to Freeze 14 days to analysis
				(1) 40 mL glass vial	1 x 5 grams	Cool 4±2°C 10 mL of Methanol	14 days to analysis
Tissue	PCBs Aroclors	unknown	Method SW846 8082A BR-GC-005(A), BR-EX-009(P)	20 g	Foil and ziplock bag	Cool to 4 ± 2 C	14 days for extraction and 40 days for analysis.

Matrix	**Analytical Group**	**Concentration Level**	**Analytical and Preparation Method / SOP Reference[1]**	**Sample Volume**	**Containers** (number, size, and type)	**Preservation Requirements** (chemical, temperature, light protected)	**Maximum Holding Time** (preparation / analysis)
Tissue	PCB Congeners	Low	1668A / 002	20 g	Foil and ziplock bag/ transfer in lab	<6 °C in transit to lab. Lab storage at < -10 °C	Up to 1 year when stored frozen and in the dark
Water	PCBs Aroclors	Low	Method SW846 8082A BR-GC-005(A), BR-EX-005(P)	1 L	(1) 1 L amber glass bottle	Cool to 4 ± 2 C	7 days for extraction and 40 days for analysis.
Solids	SVOCs	Low	Method 8270D BR-MS-001(A), BR-EX-008(P)	(1) 4 oz Amber Glass	50 grams	Cool 4±2°C	14 days to extraction 40 days to analysis
Solids	TPH GRO	Low	Laboratory Defined BR-GC-004(A), BR-EX-008(P)	(1) 4 oz glass jar	50 g	Cool to 4 ± 2 C	14 days for extraction and 40 days for analysis.
Solids	TPH DRO	Low	Method 8015D BF-GV-005, BF-MV-012	(1) Soil 4oz wide-mouth jar	3 grams	Cool 4±2°C	14 days to analysis

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

QAPP Worksheet #20 -- Field Quality Control Sample Summary Table

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference	No. of Sampling Locations	No. of Field Duplicate Pairs[1]	No. of MS/MSD[1]	No. of Rinsate Blanks[1]	No. of Trip Blanks[2]	Total No. of Samples to Lab
Sediment	PCBs	Unknown	EPA Method 8082A	42	1 (1 per 20)	1 (1 per 20)	1 (1 per 20)	0	48
Sediment	PCB Congeners	Unknown	EPA Method 1668A	42	1 (1 per 20)	1 (1 per 20)	1 (1 per 20)	0	48
Sediment	TOC	Unknown	EPA Method Lloyd Kahn	42	1 (1 per 20)	1 (1 per 20)	1 (1 per 20)	0	48
Sediment	Grain Size	Unknown	ASTM D422	42	1 (1 per 20)	1 (1 per 20)	0	0	46
Surface Water	PCBs	Unknown	EPA Method 8082A	5	1 (1 per 20)	1 (1 per 20)	1 (1 per 20)	0	8
Tissue	PCBs	Unknown	EPA Method 8082A	20	0 (1 per 20)	1 (1 per 20)	0 (1 per 20)	0	21
Solids	SVOCs	Unknown	EPA Method 8270	8	1 (1 per 20)	1 (1 per 20)	0 (1 per 20)	0	10
Solids	TPH	Unknown	EPA Method 8015	8	1 (1 per 20)	1 (1 per 20)	0 (1 per 20)	0	10

Notes

[1] Quality assurance/ quality control samples will include field duplicate samples, matrix spike/ matrix spike duplicate samples, and equipment rinsate blank samples. These samples will be completed on a frequency of 1 per 20 samples or once per week of sampling.

[2] Trip blanks are only required for volatile organic carbon samples. These samples will be completed on a frequency of 1 per 20 samples or once per week of sampling.

QAPP Worksheet #21 – Project Sampling SOP Reference Table

The following is a list of all SOPs associated with project sampling including, but not limited to sample collection, sample preservation, equipment cleaning and decontamination, equipment testing, inspection and maintenance, supply inspection and acceptance, and sample handling and custody.

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
PB-01	Field Notebook	GEI Consultants	NA	No	Attachment B
PB-02	Equipment Decontamination	GEI Consultants	Various – see SOP	No	Attachment B
PB-03	Water Saftey	GEI Consultants	Various – see SOP	No	Attachment B
PB-04	Sediment Sampling- Ponar or Shipex Grab Sampler	GEI Consultants	Ponar	No	Attachment B
PB-05	Sample Handling and Chain of Custody	GEI Consultants	NA	No	Attachment B
PB-06	Sample Handling	GEI Consultants	NA	No	Attachment B
PB-07	YSI Quick Card	GEI Consultants	YSI	No	Attachment B
PB-08	Biological Tissue Sampling	GEI Consultants	Various – see SOP	Yes	Attachment B
PB-09	Semi-porous Surface Sampling	GEI Consultants	Various – see SOP	Yes	Attachment B
PB-10	Surface Water Sampling	GEI Consultants	Various – see SOP	No	Attachment B
PB-11	Mussel Tissue Extraction	GEI Consultants	Various – see SOP	Yes	Attachment B
PB-12	Sediment Sampling Using Vibracore Equipment	GEI Consultants	Various – see SOP	No	Attachment B
PB-13	Wipe Sampling	GEI Consultants	Various – see SOP	No	Attachment B

Procedural modifications to these documents may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification will be approved in advance by the GEI Project QA Coordinator and GEI Manager and communicated to the Respondents and to the USEPA. Deviations will be documented in the field records.

QAPP Worksheet #22 – Field Equipment Calibration, Maintenance, Testing and Inspection Table

Identify all field equipment and instruments (other than analytical instrumentation) that require calibration, maintenance, testing, or inspection and provide the SOP reference number for each type of equipment. In addition, document the frequency of activity, acceptance criteria, and corrective action requirements on the worksheet.

Field Equipment	Calibration Activity	Maint. Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference[1]
Multi-parameter water quality sonde	PB-07 provided in Attachment B	PB-07 provided in Attachment B	PB-07 provided in Attachment B	PB-07 provided in Attachment B	PB-07 provided in Attachment B	PB-07 provided in Attachment B	PB-07 provided in Attachment B	Field Team Leader*	PB-07 provided in Attachment B

Notes:

[1]Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet #21).

*Calibration will be performed by field team leader and field personell trained through GEI SOPs for Multi-parameter water quality sonde calibration.

Field Instrumentation: The Field Team Leader will be responsible for insuring that these instruments are calibrated before each field sampling event. Field equipment must be inspected and calibrated before use according to the criteria given in the Field Sampling Plan. If problems occur with field instruments or equipment which cannot be resolved by the field team personnel they should contact the Field Team Leader. If field equipment fails inspection it is the Field Team Leader's responsibility to investigate and resolve the problem. The GEI Field Team Leader can coordinate with equipment vendors assist in resolution of problems with field equipment and supply or obtain any spare or replacement parts or equipment.

QAPP Worksheet #23 -- Analytical SOP References Table

All referenced analytical SOP's can be found in Attachment C.

Analytical SOP References Table						
Lab SOP Number	**Title, Revision Date, and/or Number**	**Definitive or Screening Data**	**Matrix and Analytical Group**	**Instrument**	**Organization Performing Analysis**	**Modified for Project Work (Y/N)**
BR-EX-002r11_EXcleanup	Extract Clean Up Procedures	Defninitive	Solid/Sediment PCBs	GC	TestAmerica-Burlington	N
BR-GC-005	PCBs by Gas Chromatography (GC) SOP No. BR-GC-005, Rev 11, 04/01/11	Definitive	Solid/Sediment PCBs	GC	TestAmerica-Burlington	N
BR-GC-005	PCBs by Gas Chromatography (GC) SOP No. BR-GC-005, Rev 11, 04/01/11	Definitive	Aqueous PCBs	GC	TestAmerica-Burlington	N
BR-GC-005	PCBs by Gas Chromatography (GC) SOP No. BR-GC-005, Rev 11, 04/01/11	Definitive	Tissue PCBs	GC	TestAmerica-Burlington	N
BR-EX-027	Automated Soxhlet Extraction (SW846 3541) SOP No. BR-EX-027, Rev 0, 11/10/10	Definitive	Solid/Sediment PCBs	NA Preparation	TestAmerica-Burlington	N
BR-EX-005	Separatory Funnel Extraction (SW-846 3510C) SOP No. BR-EX-005, Rev 9, 12/08/11	Definitive	Aqueous PCBs	NA Preparation	TestAmerica-Burlington	N
BR-EX-009	Homogenization of Biota & Tissue SOP No. BR-EX-009, Rev 7, 08/01/12	Definitive	Tissue PCBs	NA Preparation	TestAmerica-Burlington	N
BR-WC-008 Current Version	TOC Lloyd Kahn/	Definitive	Solid/Sediment	Carlo Erba Elemental	TestAmerica-Burlington	N

				Analyzer		
BR-GT-006 BR-GT-018	Grain Size	Definitive	Solid/Sediment	NA	TestAmerica-Burlington	N
BR-MV-006	Volatile Organic Compounds by GC/MS (SW-846 8260B) SOP No. BR-MV-006, Rev 8, 05/28/10	Definitive	Solid/Sediment Volatiles	GC/MS	TestAmerica-Burlington	N
BR-MV-007	VOA Sample Preservation & Screen Analysis Procedure (SW-846 5030A, 5035 and 5035A) SOP No. BR-MV- 007, Rev 5, 12/05/08	Definitive	Solid/Sediment Volatiles	NA Preparation	TestAmerica-Burlington	N
BR-GC-004	Diesel Range Organics by GC/FID SOP No. BR-GC-004, Rev 14, 03/18/10	Definitive	Solid/Sediment Organotins	GC	TestAmerica-Burlington	N
BR-EX-008	Ultrasonic Extraction (SW846 3550C) SOP No. BR-EX-008, Rev 10.1, 12/31/13	Definitive	Solid/Sediment Organotins	NA Preparation	TestAmerica-Burlington	N
BF-GV-005	Volatile Organic Compounds by GC (SW-846 8015D) SOP No. BF-GV-005, Rev 2, 8/30/2013	Definitive	Solid/Sediment Volatiles	GC	TestAmerica-Buffalo	N
BF-MV-012	Purge and Trap (SW-846 5030C) SOP No. BF-MV- 012, Rev 0, 07/30/13	Definitive	Solid/Sediment Volatiles	NA Preparation	TestAmerica-Buffalo	N
BR-MS-001	Semivolatile Organic Compounds by GC/MS (SW-846 8270D) SOP No. BR-MS-001, Rev 7, 04/01/11	Definitive	Solid/Sediment Semivolatiles	GC/MS	TestAmerica-Burlington	N

QAPP Worksheet #24 -- Analytical Instrument Calibration Table

Analytical Instrument Calibration Table						
Instrument	**Calibration Procedure**	**Frequency of Calibration**	**Acceptance Criteria**	**Corrective Action (CA)**	**Person Responsible for CA**	**SOP Reference**
GC	Five-point calibration	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	Option 1: Mean relative standard deviation (RSD) for all analytes ≤ 20% Option2: Linear Regression: r > 0.995	Correct problem, reanalyze, and repeat calibration.	Laboratory Analyst	SOP BR-GC-005
GC	Initial Calibration Verification	Immediately after each initial calibration	%R ± 20% of true value	Correct problem and verify second source standard. If that fails repeat calibration.	Laboratory Analyst	SOP BR-GC-005
GC	Continuing Calibration Verification	Daily before sample analysis, every 10 samples and at the end of the analytical sequence	% Difference or Drift ±20%	See Laboratory SOP	Laboratory Analyst	SOP BR-GC-005

QAPP Worksheet #24 - Analytical Instrument Calibration Table (cont.)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
HRMS	Mass Resolution Check	Before ICAL	- Document acceptable mass resolution check prior to analysis of ICAL per method.	Inspect system; adjust source tune parameters to achieve required mass resolution.	Analyst	002-KNOX-ID-0013
HRMS	Initial Calibration (ICAL)	Prior to sample analysis; after major instrument changes/maintenance or when continuing calibration criteria are no longer met.	- Analyte peaks and labeled IS peaks must have a S/N ratio of 10 or more in the CS0.5 standard - Percent valley between PCB 34 and 23 must be <= 40% - Percent valley between PCB 187 and 182 must be <= 40% - %RSD for analytes calculated using isotope dilution <= 20% - % RSD for all other analytes and internal standards <= 35%	Inspect analytical system; correct problem; repeat ICAL	Analyst	002-KNOX-ID-0013
HRMS	Initial Calibration Verification (ICV)	After ICAL and prior to sample analysis	%D can not exceed 35% for more than 4 analytes or labeled standards. %D must not exceed 50% any one analyte or labeled standard. Note: the RFs from the CS3 level of the ICAL are used to quantitated the ICV.	Inspect system; correct problem. Reanalyze ICV. Repeat ICAL if necessary.	Analyst	002-KNOX-ID-0013
HRMS	Continuing Calibration Verification (CCV)	At the beginning of each 12 hour analytical shift	- Document acceptable mass resolution checks at the start and end of each 12 hour analytical shift - The CS3 solution is used to verify RFs. The CS3 CCAL	Inspect system; correct problem; reanalyze CCV. Repeat ICAL if necessary.	Analyst	002-KNOX-ID-0013

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
			check is analyzed at the start of each 12 hour analytical shift. - S/N ratio for each analyte and labeled Is must be 10 or greater - CCV %D for Toxics/LOCs <= 30% - CCV %D for non-toxic/LOC analyte must be within 40-160% - Absolute RTs for all labeled IS must be within 15 seconds of the RTs established during the ICAL.			
LRMS	Initial Calibration (ICAL)	Prior to sample analysis; after major instrument changes/maintenance or when continuing calibration criteria are no longer met.	- %RSD for all parent PAHs must be <=30% - For each parent PAH linearity must be documented by obtaining a r^2 of 0.990 or greater	Inspect analytical system; correct problem; repeat ICAL.	Analyst	003-KNOX-ID-0018
LRMS	Initial Calibration Verification (ICV)	After ICAL and prior to sample analysis	%D can not exceed 35%	Inspect system; correct problem. Reanalyze ICV. Repeat ICAL if necessary.	Analyst	KNOX-ID-0018
LRMS	Continuing Calibration Verification (CCV)	At the beginning of each 24 hour analytical shift	- Two CCVs are analyzed at the start of each 24 hour analytical sequence - The %D for each parent PAH	Inspect system; correct problem; reanalyze CCV. Repeat ICAL if	Analyst	003-KNOX-ID-0018

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
			should be <=30% - In the event a parent PAH CCV %D is greater than 30%, calculate the mean RF CCV %D using both CCV runs. If this mean is <=30%D an acceptable CCAL has been obtained	necessary.		
5310C - TOC	Initial Calibration	The instrument is calibrated at the beginning of each day or if the CCV/CCB fails to meet acceptance criteria.	The calibration correlation coefficient is $\geq$0.995.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/Supervisor	029-PT-WC-017
	Initial Calibration Verification (ICV)	Analyze a standard at the beginning after calibration	The acceptance criterion for the initial calibration verification standard is 90 to 110% recovery of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/Supervisor	
	Initial Calibration Blank (ICB)	Before beginning a sample sequence, following ICV.	The result must be < RL.	Terminate analysis; Correct the problem; Recalibrate.	Analyst/Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
	CCV	Analyze a standard at the beginning and end of the sequence and after every 10 environmental samples.	The acceptance criterion for the continuing calibration standard ± 10% of the initial curve.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standard. Reanalyze the affected data.	Analyst/Supervisor	
	CCB	Immediately following CCV and at the end of the sequence.	The result must be < RL.	Correct the problem, then reprepare and reanalyze calibration blank and previous 10 samples.	Analyst/Supervisor	
GC/MS SW-846 8260B	Tune Standard	Prior to initial calibration and every 12 hours	See Laboratory SOP	Reanalyze, retune mass spectrometer; no samples may be analyzed without a valid tune.	Laboratory Analyst	SOP BR-MV-006
GC/MS SW-846 8260B	Five-point calibration	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	Option 1: Mean relative standard deviation (RSD) for all analytes ≤ 20% Option2: Linear Regression: r > 0.995	Instrument maintenance, standard, inspection, recalibration	Laboratory Analyst	SOP BR-MV-006
GC/MS SW-846 8260B	Initial Calibration Verification	Immediately after each initial calibration	%R ± 25% of true value	Correct problem and verify second source standard. If that fails repeat calibration.	Laboratory Analyst	SOP BR-MV-006
GC/MS SW-846 8260B	Continuing Calibration Verification	Beginning of each 12-hour window after the tune standard	See Laboratory SOP	See Laboratory SOP	Laboratory Analyst	SOP BR-MV-006

QAPP Worksheet #24 -- Analytical Instrument Calibration Table (cont.)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Incubators	Temperatures checked daily during weekdays	Thermometers calibrated biannually	Must match temperature range in log book	Report/discontinue use/Repair	QA officer	034-039
Scales	Checked monthly	Vendor calibrator annually	Must match range in QC packet	Report/discontinue use/Repair	QA officer	034-039
Microscopes	Micrometer checked	Vendor calibration and maintenance annually	Must be uniform with analyst verification	Report/discontinue use/Repair	QA officer	034
Pipettors	Checked quarterly	Vendor calibrator annually	Must match range in QC packet	Report/clean/Repair	QA officer	034-039

QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Identify all analytical instrumentation that requires maintenance, testing, or inspection and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
TOC – 5310C	Check Oxygen supply Persulfate supply Acid supply Carrier gas flow rate (~ 150 cc/min) IR millivolts for stability (after 30 min. warm-up) Reagent reservoirs	TOC	Check injection port septum after 50-200 runs. Tube end-fitting connections after 100 hours or use. Indicating drying tube. NDIR zero, after 100 hours of use. Sample pump, after 2000 hours for use. Digestion vessel/condensation chamber, after 2000 hours of	As needed	CCV +/- 10%	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data	Analyst/ Supervisor	029- PT-WC-017

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
			use. Permeation tube, after 2000 hours of use. NDIR cell, after 2000 hours of use					
GC	Replace Septa, Clean and replace Injection Port Liner, Replace or clip Guard Column Replace or clip Analytical Column Bake, Re-foil, Refurbish Detector		Check Septa, Injection Port Liner, Guard Column and Analytical Column	As required	Passing calibration	Perform maintenance, check standards, recalibrate	Laboratory Analyst	SOP BR-QAM
GC/MS (VOA)	Clean Injection Port and Liner, Change Septa, Cut 2-3 inches from GC Column, Fill Autosampler rinse vials, Clean Purge and Trap mount and purge vessel		Check Injection Port and GC columns, Check autosampler rinse vials, check purge and trap mount and purge vessel, check Purge Flow	As required	Passing calibration	Perform maintenance, check standards, recalibrate	Laboratory Analyst	SOP BR-QAM

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Carlo Erba Elemental Analyzer	Oven maintenance	Lloyd Kahn	Pass calibration and blank checks	Daily	Linear Regression r $\geq$ 0.995 ICB < RL CCV 85-115 %	Perform Maintenance, Check Standards, Recalibrate, Reanalyze	Assigned Lab personnel	006
Costech Elemental Combustion System	Oven maintenance	Lloyd Kahn	Pass calibration and blank checks	Daily	Linear Regression r $\geq$ 0.995 ICB < RL CCV 85-115 %	Perform Maintenance, Check Standards, Recalibrate, Reanalyze	Assigned Lab personnel	006
HRMS	Injection port maintenance, clean ion volume, clean source, replace filament	Refer to Worksheet #24	Refer to Worksheet #24	As needed	Refer to Worksheet #24	Refer to Worksheet #24	Analyst	002-KNOX-ID-0013
HRMS	Tune instrument to maximize sensitivity and mass resolution	Refer to Worksheet #24	Refer to Worksheet #24	Daily	Refer to Worksheet #24	Refer to Worksheet #24	Analyst	002-KNOX-ID-0013
HRMS	Change carrier gas filters	Refer to Worksheet #24	Refer to Worksheet #24	Yearly	Refer to Worksheet #24	Refer to Worksheet #24	Analyst	002-KNOX-ID-0013
HRMS	Change mechanical pump fluid	Refer to Worksheet #24	Refer to Worksheet #24	Yearly	Refer to Worksheet #24	Refer to Worksheet #24	Analyst	002-KNOX-ID-0013

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
LRMS	Injection port maintenance, clean source, replace filament	Refer to Worksheet #24	Refer to Worksheet #24	As needed	Refer to Worksheet #24	Refer to Worksheet #24	Analyst	003-KNOX-ID-0018
LRMS	Tune to maximize sensitivity and mass resolution	Refer to Worksheet #24	Refer to Worksheet #24	As needed	Refer to Worksheet #24	Refer to Worksheet #24	Analyst	003-KNOX-ID-0018
Scales	Clean, calibrate	Vendor tests yearly	As needed and yearly	Monthly	As documented in log books	Report/repair	QA Officer	036, 037,038,039, 040
Microscopes	Clean	Vendor tests yearly	As needed and yearly	As needed and yearly	As documented in log books	Report/repair	QA Officer	036
Pipettors	Clean, change o-ring	Vendor tests yearly	As needed and yearly	As needed and quarterly	As documented in log books	Report/repair	QA Officer	036, 037,038,039, 040

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

QAPP Worksheet #26 – Sample Handling System

Use this worksheet to identify components of the project-specific sample handling system. Record personnel, and their organizational affiliations, who are primarily responsible for ensuring proper handling, custody, and storage of field samples from the time of collection, to laboratory delivery, to final sample disposal. Indicate the number of days field samples and their extracts/ digestates will be archived prior to disposal.

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): GEI Consultants Field Team supervised by the Field Team Leader will collect samples.
Sample Packaging (Personnel/Organization): GEI Consultants Field Team
Coordination of Shipment (Personnel/Organization): GEI Consultants Field Team
Type of Shipment/Carrier: Federal Express for Overnight Delivery or courier to the laboratory
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Assigned laboratory personnel
Sample Custody and Storage (Personnel/Organization): Assigned laboratory personnel
Sample Preparation (Personnel/Organization): Assigned laboratory personnel
Sample Determinative Analysis (Personnel/Organization): Assigned laboratory personnel
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): Samples will not be stored in the field, but will be kept in cooler at 4 degree C and shipped within 24 hours of collection. If due to an emergency they are stored in the field, they will be kept in a cooler or transferred to a refrigerator kept at 4 degrees C.
Sample Extract/Digestate Storage (No. of days from extraction/digestion): Sample extraction and digestion will be conducted according to the SOPs and the requirements given in Worksheet 19.
Biological Sample Storage (No. of days from sample collection): NA
SAMPLE DISPOSAL
Personnel/Organization: Test America Laboratories Sample Custodians
Number of Days from Analysis: At least 60 days

QAPP Worksheet #26 – Sample Handling System (cont.)

Sample Handling System

Sample handling and custody procedures ensure the timely, correct, and complete analysis of each sample for all parameters requested. A sample is considered to be in a person's custody if it is in:

- his/her possession;
- his/her view, after being in his/her possession;
- his/her possession and has been placed in a secure location; or
- a designated secure area.

Sample custody documentation provides a written record of sample collection and analysis. The sample custody procedures provide for specific identification of samples associated with an exact location, the recording of pertinent information associated with the sample, including time of sample collection and any preservation techniques, and a Chain of Custody (COC) record which serves as physical evidence of sample custody. Custody procedures will be similar to the procedures outlined in the USEPA's Contract Laboratory Program Guidance for Field Samplers (USEPA, 2007). The COC documentation system provides the means to individually identify, track, and monitor each sample from the time of collection through final data reporting. Sample custody procedures are developed in three areas: sample collection, laboratory analysis, and final evidence files, which are described below.

Field Sample Handling and Custody

Field records provide a means of recording information for each field activity performed at the Site. COC procedures document pertinent sampling data and all transfers of custody until the samples reach the analytical laboratory. The sample packaging and shipment procedures summarized below will ensure that the samples arrive at the laboratory with the COC intact. Worksheet 19 lists the specific sample preservation requirements for each test method.

QAPP Worksheet #26 – Sample Handling System Field Procedures (cont.)

The general responsibilities of the field team are listed below:

- The field sampler is personally responsible for the care and custody of the samples until they are transferred to the Sample Management Officer (SMO) or until they are properly dispatched. As few people as possible should handle the samples.
- The Field Team Leader, or designee, is responsible for entering the proper information in the field logbook, including all pertinent information such as sample identification number, date and time of sample collection, type of analysis, and description of sample location. The information entered into the field log book will be used to generate a COC.
- All sample containers will be labeled with the project identification, sample identification, matrix, type of analysis required, and preservation requirements.
- The samples will be properly preserved, bagged, and packed into coolers. The original COC form will be placed into the lead cooler and will be shipped to the laboratory.
- The SMO or designee will review all field activities to determine whether proper custody procedures were followed during the field work and if additional samples are required.

Field Records

The field log book will provide the means of recording data collection activities. Entries will be described in as much detail as possible, so that persons going to the Project Properties can reconstruct a particular situation without reliance on memory. At the beginning of each field day, the date, start time, weather, and names of all sampling team members present will be entered. The names of visitors to the Project Properties and the purpose of their visit will also be recorded. All field measurements, as well as the instrument(s), will be noted.

Samples will be collected following the sampling procedures documented in the workplan. Observations such as sampling conditions or any problems will also be recorded. Sample identification numbers will be assigned at the time the data are entered in the logbook. Field duplicate samples, which will receive a unique sample identification number, are “blind” to the laboratory and will be identified under the sample description so that they can be associated with their respective samples by project staff.

QAPP Worksheet #26 – Sample Handling System (cont.)

Sample Identification System

All samples collected from the Project Properties must be identified with a sample label in addition to an entry on a COC record. Indelible ink will be used to complete sample labels and handwritten COC records. Each sample will be identified by a unique sample number assigned by the field team as described in the workplan. The unique sample identification will include a sequential sample number, the well location identification (ID), and the type of sample and the depth of the sample, if applicable.

Sample Labels/Tags

Sample labels will require the field team to complete the following information for each sample container:

1. Sample Number;
2. Sample Matrix;
3. Parameters to be analyzed;
4. Date of Collection;
5. Time of Collection;
6. Preservation Method(s); and
7. Sampler's Name.

QAPP Worksheet #27 -- Sample Custody Requirements

Describe the procedures that will be used to maintain sample custody and integrity. Include examples of chain-of-custody forms, traffic reports, sample identification, custody seals, laboratory sample receipt forms, and laboratory sample transfer forms.

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory):
Samples will be collected per the procedures described in the workplan. The field sample custody procedures including sample packing, shipment, and delivery requirements are discussed in the text in Worksheets 17 and 26.

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal):
Each laboratory will have a sample custodian who accepts custody of the samples and verifies that the information on the sample labels matches the information on the COC. The sample custodian will document any discrepancies and will sign and date all appropriate receiving documents. The sample custodian will also document the condition of the samples upon receipt at the laboratory. The laboratory sample custody procedures ware discussed further in the following text.

Sample Identification Procedures:
The sample identification scheme that will be employed is described in the Surface Sediment Investigation Work Plan. Sample labeling procedures are discussed in the text in Worksheet 26.

Chain-of-Custody Procedures:
A COC record will accompany the samples from the time of sampling through all transfers of custody. The COC procedures are detailed in the following text.

QAPP Worksheet #27 -- Sample Custody Requirements (cont.)

Sample Custody Requirements:

Chain of Custody Procedure

The following information should be recorded on COC forms. All COC forms must be signed in ink:

- Project name and/or project number;
- Signature of SMO or designee;
- Sampling station number;
- Date and time of collection;
- Grab or composite sample designation;
- Sample matrix;
- Sampling location description;
- Field identification number;
- Analyses required;
- Preservation technique;
- Signatures and dates for transfers of custody; and
- (if applicable) Air express/shipper's bill of lading identification numbers.

The COC form serves as an official communication to the laboratory detailing the particular analyses required for each sample. The COC record will accompany the samples from the time of sampling through all transfers of custody. It will be kept on file at the laboratory where samples are analyzed and archived. Three copies of the COC form are created; one copy is retained by the Field Team Leader and two are sent to the laboratory. An electronic copy of each COC should be also made and kept in the project directory. The SMO or designee completes a COC record to accompany each shipment from the field to the laboratory.

The completed COC is put in a zip-lock bag and taped to the inside cover of the sample shipping container. If there is more than one container in a shipment, copies of the COC form will be placed in each container. Each container is then sealed with custody seals and custody is transferred to the laboratory.

QAPP Worksheet #27 -- Sample Custody Requirements (cont.)

Transfer of Custody and Shipment

The custody of samples must be maintained from the time of sampling through shipment and relinquishment to the laboratory. Instructions for transferring custody are given below:

- All samples are accompanied by a COC. When transferring custody of samples, the individuals relinquishing and receiving will sign, date, and note the time on the COC. This form documents sample custody transfer from the SMO or designee, through the shipper, to the analytical laboratory. Since a common carrier will usually not accept responsibility for handling COC forms, the name of the carrier is entered under "Received by," the bill-of-lading number is recorded in the comments section, and the COC form is placed in a zip-lock plastic bag and taped to the inside lid of the lead shipping cooler. Copies of the COC form will be placed in each additional cooler in a shipment.

- Samples will be packaged for shipment and either picked up at a pre-arranged location by the laboratory or dispatched to the appropriate laboratory via overnight delivery service. A separate COC record must accompany each shipment. Shipping containers will be sealed for shipment to the laboratory. Two custody seals will be applied to each cooler to document that the container was properly sealed and to determine if the container was tampered with during shipment. The custody seals will be placed on the coolers in such a manner that the custody seal would be broken if the cooler were opened (*i.e.*, diagonally opposite corners of the cooler lid).

- The original COC will accompany the shipment. A copy will be retained by the Field Team Leader.

- If the samples are sent by common carrier or air freight, proper documentation must be maintained. For example, the bill of lading must be retained by the Field Team Leader.

Laboratory Custody Procedures

The laboratory custody procedures will be equivalent to those described in the latest edition of the SOW. The following will be addressed in the laboratory custody SOPs:

- A designated sample custodian accepts custody of the samples and verifies that the information on the sample labels matches the information on the COC. The sample custodian will document any discrepancies and will sign and date all appropriate receiving documents. The sample custodian will also document the condition of the samples upon receipt at the laboratory.

QAPP Worksheet #27 -- Sample Custody Requirements (cont.)

- Once the samples have been accepted by the laboratory, checked and logged in, they must be maintained in accordance with laboratory custody and security requirements.
- To ensure traceability of samples while in the possession of the laboratory, a method for sample identification that has been documented in a laboratory SOP will be used to assign sample numbers.
- The following stages of analysis must be documented by the laboratory:
  - Sample Extraction/Preparation.
  - Sample Analysis.
  - Data Reduction.
  - Data Reporting.
- Laboratory personnel are responsible for the custody of samples until they are returned to the sample custodian.
- When sample analyses and QA checks have been completed in the laboratory, the used portion of the sample must be stored or disposed of in accordance with the protocols specified in the SOW or the subcontract agreement. Identifying labels, data sheets, COCs, and laboratory records will be retained until analyses and QA checks are completed in accordance with the protocols specified in the subcontract agreement.

Final Evidence Files

This is the final phase of sample custody. The COC records and sample analysis request form copies are archived in their respective project files. Laboratory custody forms, sample preparation and analysis logbooks, and data packages will become part of the laboratory final evidence file. Other relevant documentation including records, reports, and correspondence, logs, pictures, and data review reports will be archived by GEI Consultants.

Sample Holding Times

Information on sample holding times and required preservation for each test method are provided in Worksheet 19.

Sample Packaging and Shipping Requirements

Custody of samples must be maintained through the shipment of samples to the selected laboratory. All samples will be packaged and shipped at the end of each day unless other arrangements are made with the laboratory.

QAPP Worksheet #28 (UFP-QAPP Manual Section 3.4) -- QC Samples Table (cont.)

Matrix	Aqueous / Solid / Sediment / Tissue
Analytical Group	PCBs
Analytical Method/ SOP Reference	BR-GC-005

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per extraction batch of 20 or fewer samples	< LOQ	See laboratory SOP	TestAmerica Laboratory	Contamination	See worksheet 15 for lab CRQL
Surrogates	Each sample, standard, blank	See worksheet 15	See laboratory SOP	TestAmerica Laboratory	Accuracy/Bias	Per Laboratory SOP
Laboratory Control Sample	One per extraction batch of 20 or fewer samples	See worksheet 15	See laboratory SOP	TestAmerica Laboratory	Accuracy	Per Laboratory SOP
Matrix Spike/Matrix Spike Duplicates	Per client Request	See worksheet 15	See laboratory SOP	TestAmerica Laboratory	Accuracy/Bias and Precision	Per laboratory SOP
Method Detection Limits	Annual	Per Laboratory SOP	Reanalyze MDL	TestAmerica Laboratory	Sensitivity	Low enough to support CRQLs

QAPP Worksheet #28 (UFP-QAPP Manual Section 3.4) -- QC Samples Table (cont.)

Matrix	Sediment
Analytical Group	Total Organic Carbon
Concentration Level	Low
Sampling SOP	See Worksheet 20
Analytical Method / SOP Reference	Lloyd Kahn, 006
Analytical Organization	Test America, Inc Burlington VT
Number of Sample Locations	See Worksheet 18

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equip blank	One per day per type of sampling equipment	Per laboratory SOP	Investigate source of contamination	Assigned Lab personnel	Accuracy/Bias	Per laboratory SOP
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	Per client submission	Per laboratory SOP	None if blank spike passes	Assigned Lab personnel	Accuracy/Bias	Per laboratory SOP
Field Duplicate	1 per 20 field samples	QAPP	If the limits exceed limits for the field duplicate, this will be addressed by the GEI Data Reviewer	GEI Field Team Leader	Precision	RPD < 30% for duplicate for values greater than or equal to five times the CRQL
LFB Method Blank (MB)	Once per batch	QAPP	Re-prep Batch	Assigned Lab personnel	Sensitivity	Per laboratory SOP

QAPP Worksheet #28 (UFP-QAPP Manual Section 3.4) -- QC Samples Table (cont.)

Matrix	Aqueous / Solid / Sediment
Analytical Group	Volatile Organics
Analytical Method/ SOP Reference	BR-MV-006

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Once every 12 hours	< LOQ	Reanalyze Batch	TestAmerica Laboratory	Contamination	See worksheet 15 for lab CRQL
Surrogates	Each sample, standard, blank	See worksheet 15	Reanalyze sample	TestAmerica Laboratory	Accuracy/Bias	Per Laboratory SOP
Laboratory Control Sample	Once every 12 hours	Per Worksheet 15	Reanalyze Batch	TestAmerica Laboratory	Accuracy	Per Laboratory SOP
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	Each group of field samples in an SDG or each SDG, whichever is most frequent	See worksheet 15	None if laboratory control sample passes	TestAmerica Laboratory	Accuracy/Bias and Precision	Per laboratory SOP
Internal Standard	Each sample, standard, Blank	Area between 50-100% of area of daily calibration internal standard area	Reanalyze Sample	TestAmerica Laboratory	Instrument Performance	Per Laboratory SOP
Method Detection Limits	Annual	Per Laboratory SOP	Reanalyze MDL	TestAmerica Laboratory	Sensitivity	Low enough to support CRQLs

QAPP Worksheet #28 (UFP-QAPP Manual Section 3.4) -- QC Samples Table (cont.)

Matrix	Sediment
Analytical Group	PCB Congeners
Concentration Level	Low
Sampling SOP	See Worksheet 20
Analytical Method / SOP Reference	002
Analytical Organization	TestAmerica Knoxville, TN
Number of Sample Locations	See Worksheet 18

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per batch (20 or fewer client samples)	No Target Analytes > EML	Reanalyze Method Blank (MB) and samples if instrument performance is believed to have contributed to MB failure. If sample results >20x blank or ND, report results. If analyte result in MB > RL and sufficient sample is available, re-prepare and reanalyze batch if project data quality objectives are not met or flag data in consultation with client.	Analyst/Section Supervisor/PM	Accuracy/Bias-Contamination	No Target Analytes > EML
Laboratory Control Sample	1 per batch (20 or fewer client samples)	Toxic/LOC Analytes percent recovery between 50% and 150%	Verify calculations. Reanalyze extract if instrument performance is believed to have contributed to LCS failure. If LCS recovery is high and the analyte is not detected, document excursion in narrative. If sufficient sample is available, re-prepare and reanalyze batch or flag data in consultation with client.	Analyst/Section Supervisor/PM	Accuracy/Bias	Laboratory SOP % Recovery Control Limits
Internal Standards	Every client sample field QC and lab QC sample	Percent recovery between 30% and 140%	Verify calculations. For low recovery calculate S/N ratio. If S/N is >= 10 and EDL <= EML report data as is and narrate.	Analyst/Section Supervisor/PM	Accuracy/Bias	Laboratory SOP % Recovery Control Limits

QAPP Worksheet #28 (UFP-QAPP Manual Section 3.4) -- QC Samples Table (cont.)

Matrix	Aqueous / Solid / Sediment
Analytical Group	Semivolatile Organics
Analytical Method/ SOP Reference	BR-MS-001

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Once every 12 hours	< LOQ	Reanalyze Batch	TestAmerica Laboratory	Contamination	See worksheet 15 for lab CRQL
Surrogates	Each sample, standard, blank	See worksheet 15	Reanalyze sample	TestAmerica Laboratory	Accuracy/Bias	Per Laboratory SOP
Laboratory Control Sample	Once every 12 hours	Per Worksheet 15	Reanalyze Batch	TestAmerica Laboratory	Accuracy	Per Laboratory SOP
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	Each group of field samples in an SDG or each SDG, whichever is most frequent	See worksheet 15	None if laboratory control sample passes	TestAmerica Laboratory	Accuracy/Bias and Precision	Per laboratory SOP
Internal Standard	Each sample, standard, Blank	Area between 50-100% of area of daily calibration internal standard area	Reanalyze Sample	TestAmerica Laboratory	Instrument Performance	Per Laboratory SOP
Method Detection Limits	Annual	Per Laboratory SOP	Reanalyze MDL	TestAmerica Laboratory	Sensitivity	Low enough to support CRQLs

QAPP Worksheet #28 (UFP-QAPP Manual Section 3.4) -- QC Samples Table (cont.)

Matrix	Solid
Analytical Group	Total Petroleum Hydrocarbons
Analytical Method/ SOP Reference	BF-MS-004/BF-MV-005

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Once every 12 hours	< LOQ	Reanalyze Batch	TestAmerica Laboratory	Contamination	See worksheet 15 for lab CRQL
Surrogates	Each sample, standard, blank	See worksheet 15	Reanalyze sample	TestAmerica Laboratory	Accuracy/Bias	Per Laboratory SOP
Laboratory Control Sample	Once every 12 hours	Per Worksheet 15	Reanalyze Batch	TestAmerica Laboratory	Accuracy	Per Laboratory SOP
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	Each group of field samples in an SDG or each SDG, whichever is most frequent	See worksheet 15	None if laboratory control sample passes	TestAmerica Laboratory	Accuracy/Bias and Precision	Per laboratory SOP
Internal Standard	Each sample, standard, Blank	Area between 50-100% of area of daily calibration internal standard area	Reanalyze Sample	TestAmerica Laboratory	Instrument Performance	Per Laboratory SOP
Method Detection Limits	Annual	Per Laboratory SOP	Reanalyze MDL	TestAmerica Laboratory	Sensitivity	Low enough to support CRQLs

QAPP Worksheet #29 -- Project Documents and Records Table

This section identifies the documents and records that will be generated for all aspects of the project including, but not limited to, sample collection and field measurement, on- and off-site analysis, and data assessment.

Project Documents and Records Table

Sample Collection Documents and Records (as applicable):

- Field Notes and or data sheets
- Chain of Custody Forms
- Air bills
- Analytical and Testing Sample Data Packages
- Data Validation Reports

On-Site Activities Documents and Records:

- Sample collection and processing record and custody records.
- Sample custody records
- Air bills (if applicable)
- Custody records
- Copies of field notes

Off-Site Analysis Documents and Records

- Chain of Custody (COC) records will be made and stored in the project files
- Copies of air bills (if applicable) will be kept in project files

• Copies of all Analytical Data Deliverables stored in Lab and transferred to Project files, instrument calibration records, lab, raw data stored in electronically or in hardcopy. Laboratory electronic data deliverables (EDD) will be obtained in a NYS DEC EDD ("EqUIS") format.**Data Assessment Documents and Records**

- Project Records: Copies of all field notes must be sent to GEI Consultants, Inc.
- Project Records: Copies of COC must be kept by GEI Consultants, Inc.
- Field and/or lab inspection reports/checklists
- Corrective action documentation
- Data validation narratives
- QA Review sheet
- Copies of Form 1
- Final Report

This section describes the project data management process, tracing the path of the data from their generation to their final use or storage. All project data and information must be documented in a format useable to the project personnel.

Project Document Control System

Project documents will be controlled by the GEI Consultants, Inc. Project Manager who will maintain and distribute the hardcopies and electronic copies of the project documents, including any amendments. Electronic copies of project information will be maintained in the project directory on the server at GEI Consultants, Glastonbury, CT office, which is backed up at least once per day.

Data Recording

Data for this project will be collected by handwritten entries and will be recorded into field logbooks or on forms. Software may be the used to generate COC records and sample labels, or COCs and labels may be created manually. Computer-generated data associated with laboratory analyses will be managed under the control of the laboratory's laboratory information management system (LIMS).

Laboratory Data Transmittal

Laboratory data are managed by the laboratory's LIMS system, beginning with sample check-in on the sample receiving data terminal. Full laboratory data reports will be delivered to GEI Consultants, Inc. and will include electronic data deliverables (EDDs).

Data Storage and Retrieval

Paper copies of the forms, electronic copies of files, and the photographic log will be transmitted regularly to the GEI PM or designee. The completed forms and notebooks will be stored in the custody of the PM for the duration of the project. The full laboratory data reports submitted to GEI Consultants, Inc. will be stored in the custody of the Project Quality Officer or designee.

The Laboratory will maintain copies of documents and backups of all data associated with the analyses of samples. Raw data and electronic media of all field samples, including QC samples and blanks, will be archived from the date of generation and will be kept by the laboratory. Hard copies of project files will be archived at a secure facility and retained until the end of the contract. Data will be transferred to National Grid upon completion of the project. Retrieval of data by others will be at the discretion of National Grid and the NYSDEC. The length of time that records will be archived will be at the discretion of the National Grid and NYSDEC.

Each laboratory will archive, electronically, the sample analyses and submit the electronic data files along with the data deliverable package. Laboratory electronic data deliverables (EDD) will be obtained in a NYS DEC EDD ("EqUIS") format.

QAPP Worksheet #30 – Analytical Services Table

Identify all laboratories or organizations that will provide analytical services for the project, including on-site screening, on-site definitive, and off-site laboratory analytical work. Group by matrix, analytical group, concentration, and sample location or ID number. If applicable, identify the subcontractor laboratories and backup laboratory or organization that will be used if the primary laboratory or organization cannot be used.

Matrix	Analytical Group	Concentration Level	Sample Locations/ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/ Organization	Backup Laboratory/Organization
Sediment Samples							
Sediment/Solids	PCBs USEPA Method 8082A	Low	See Worksheet 18	001	Validation Level Data Package: Fifteen Business Days	TestAmerica-Burlington, VT	A backup lab has not been assigned at this time
Sediment/Solids	PCB Congeners USEPA Method 1668A	Low	See Worksheet 18	002	Validation Level Data Package: Fifteen Business Days	TestAmerica-Knoxville, TN	A backup lab has not been assigned at this time
Sediment	Total Organic Carbon USEPA Method Lloyd Kahn	Low	See Worksheet 18	003	Validation Level Data Package: Fifteen Business Days	TestAmerica-Burlington, VT	A backup lab has not been assigned at this time
Sediment	Grain Size ASTM D422	Standard	See Worksheet 18	004	Validation Level Data Package: Ten Business Days	TestAmerica-Burlington, VT	A backup lab has not been assigned at this time
Solids	SVOCs	Low	See Worksheet 18	004	Validation Level Data Package: Ten Business Days	TestAmerica-Burlington, VT	A backup lab has not been assigned at this time
Solids	TPH	Low	See Worksheet 18	004	Validation Level Data Package: Ten Business Days	TestAmerica-Buffalo, NY	A backup lab has not been assigned at this time

Note: Environmental data suppliers (labs) will provide accredited information by the NYSDOH ELAP in appropriate analytical categories.

QAPP Worksheet #31 – Planned Project Assessment Table

Identify the type, frequency, and responsible parties of planned assessment activities that will be performed for the project.

Assessment Type	**Frequency**	**Internal or External**	**Organization Performing Assessment**	**Person(s) Responsible for Performing Assessment** (title and organizational affiliation)	**Person(s) Responsible for Responding to Assessment Findings** (title and organizational affiliation)	**Person(s) Responsible for Identifying and Implementing Corrective Actions (CA)** (title and organizational affiliation)	**Person(s) Responsible for Monitoring Effectiveness of CA** (title and organizational affiliation)
Field Safety Audit	Discretionary	Internal	GEI	GEI Corporate Health and Safety Officer	GEI PMs	GEI PMs	GEI PMs
Contractor Performance Evaluation	Monthly or as warranted	External	National Grid	National Grid delegate	GEI PMs	GEI PMs	GEI PMs

QAPP Worksheet #32 – Assessment Findings and Response Actions

For each type of assessment describe procedures for handling QAPP and project deviations encountered during the planned project assessments.

PROJECT ASSESSMENT TABLE

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Party responsible for performing assessment	Person(s) responsible for responding to assessment findings	Person(s) responsible for identifying and implementing corrective actions
Field Sampling Assessment	Project commencement and as needed.	Internal	GEI	Field Team Leader/Project Safety Officer, GEI, Inc.	Project Manager, GEI	Project Manager, GEI
Fixed Laboratory Technical Systems Audit	As needed.	External	GEI	Project QA/QC Officer, GEI, Inc.	Project Manager, Project Laboratory	Project Manager, Project Laboratory

Field Oversight

Field oversight of the project will be conducted by the Task Manager/Field Leader on a daily basis. The Task Manager/Field Leader will oversee the field samplers and subcontractors to see that the work goes smoothly and according to the site-specific plans. Corrective actions will be addressed immediately in the field and any issues that might possibly impact the data will be documented in the field notes.

Field Sampling Assessment

An assessment of field sampling procedures would take place on-Site early in the field program so that necessary corrective action measures can be implemented, if required. The assessment would consist of an evaluation by the Field Team Leader and Project Safety Officer of sampling techniques, field parameter measurements, record keeping including log books and COCs, sample collection and handling sample design, subcontractor oversight and health and safety.

QAPP Worksheet #32 – Assessment Findings and Response Actions (cont.)

Fixed Laboratory Technical Systems Audit

A laboratory technical systems audit would consist of a review of any, but not necessarily all, of the following: sample handling procedures, equipment condition and operation, analytical methods and procedures and overall conformance with SOPs provided in this QAPP. The audit may span a period of one or more days, so that the audit team can view various types of analytical procedures that will be used on the project.

Assessment Findings and Corrective Action Responses

Deficiencies that are found as a result of the audits will be communicated both verbally to the responsible party upon discovery and will also be documented in a written audit report. A formal corrective action response in writing will be requested from the responsible party. The response will document the reason for the deficiency and the actions that will be put in place to correct the deficiency. Corrective action responses will be filed in the project files.

Additional QAPP Non-Conformances

The corrective action procedures discussed in this section will also be applied to significant deviations from procedures outlined in this QAPP. Project personnel who determine that a deviation has occurred will document the deviation and notify the GEI project manager. The project manager will evaluate the severity of the deviation, document deviations, and implement corrective action procedures as appropriate.

QAPP Worksheet #33 – Planned Project Assessment Table

Identify the frequency and type of planned QA Management Reports, the project delivery dates, the personnel responsible for report preparation, and the report recipients.

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Data Validation Report	After laboratory data are received	Within 30 days after receiving the full deliverable	Data Validator	Project QC Officer and Project Manager

The National Grid PM will receive various types of management reports, such as the results of the data validation reports. In addition, monthly progress report, provided to National Grid and USEPA, may contain a section on quality control issues. Problems or issues that arise between regular reporting periods may be identified to program management. The progress report will include an assessment of problems with the measurement data, including accuracy, precision, completeness, representativeness, and comparability.

QAPP Worksheet #34 – Sampling and Analysis Verification (Step 1) Process Table

Describe the processes that will be followed to verify project data. Describe how each item will be verified, when the activity will occur, and what documentation is necessary, and identify the person responsible. *Internal* or *external* is in relation to the data generator.

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Chain of custody (COC)	Form will be internally reviewed upon completion and verified against field logs and laboratory reports. Review will occur with the completion of each report.	I	GEI Consultants, Inc.
Field report	Field reports will be verified with the field logbooks.	I	GEI Consultants, Inc.
Laboratory data packages	Laboratory data packages will be used to verify the reported results in the project report and against QAPP criteria.	I	GEI Consultants, Inc.

Data Verification

- The Field Team Leader or designee is required to review the logbook entries for errors or omissions. This information is transmitted to the Project QC Officer or designee for correction.

- In addition, the Project QC Officer or designee is responsible for reviewing field data for completeness and to verify that the field crew followed the QC requirements detailed in this QAPP (*e.g.*, the collection of QC samples at the required frequency, response checking the field instruments). If any problems with the information are found, the Project QC Officer or designee will document the problems.

The Project QC Officer or designee reviews the field data.

QAPP Worksheet #35 – Sampling and Analysis Validation (Steps IIa and IIb) Process Table

Describe the processes that will be followed to validate project data. Validation inputs include items such as those listed in Table of the UFP-QAPP Manual (Section 5.1). Describe how each item will be validated, when the activity will occur, and what documentation is necessary and identify the person responsible. Differentiate between steps IIa and IIb of validation.

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa	Methods	Records support implementation of SOP in QAPP.	GEI Consultants, Inc
IIa	Chain of Custody	Examine traceability of data from sample collection to generation of project report	GEI Consultants, Inc
IIb	Deviations from SOP and project documents.	Determine impacts of any deviation from methods and the project plan.	GEI Consultants, Inc

QAPP Worksheet #36 – Sampling and Analysis Validation (Steps IIa and IIb) SummaryTable

Identify the matrices, analytical groups, and concentration levels that each entity performing validation will be responsible for, as well as criteria that will be used to validate those data.

Step IIa/ IIb	Matrix	Analytical Group1	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa/ IIb	Sediment/solid and Tissue	Chemical Parameters	Low to High	NYSDEC Validation Criteria*	Lorie MacKinnon, GEI Consultants, Data Validator
IIa/ IIb	Sediment/solid and tissue	Chemical/Geotechnical Parameters	Low to High	NYSDEC Validation Guidance* and Laboratory SOP Criteria	Lorie MacKinnon, GEI Consultants, Data Validator

[1]. Analytical data on chemical parameters produced by subcontract laboratories will be reviewed by a qualified data validator assigned by GEI.

*Validation will be performed in accordance with the NYSDEC DER-10 based on a Category B NYSDEC ASP Category B Data Deliverable, followed by validation per *USEPA Region II Functional Guidelines for Evaluating Organic Analyses* (September 2006b).

QAPP Worksheet #37—Data Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:
The GEI Consultants' data validator will validate chemical data in accordance with the protocols outlined on Worksheet 35. Data validation alone does not insure usability of the data. Other factors will be considered, including comparison of actual reporting limits achieved by the lab on the samples collected to the project action levels and data needs.

Describe the evaluative procedures used to assess overall measurement error associated with the project:
As part of the data validation process, the validator identifies any qualifications, the bias (if known) of the data, applies qualifiers and comments on the usability of the data. Once the validation package is received from the validator it is reviewed by the Project Quality Officer or a designee. Any QA/QC problems with the validation will be discussed with the validator and laboratories. Data will be compared to appropriate reference limits provided in Worksheet No. 15.

Identify the personnel responsible for performing the usability assessment:
The usability of the data is the responsibility of the project team. The PMs will reconvene the project team after all data has been validated and reviewed. The data users performing the remediation design will participate in a usability assessment to determine if the data is sufficient to meet the data needs and the project DQOs, and will recommend if additional data is required. A data assessment report will be issued by the PM or his designee documenting the results of the usability assessment review performed by the project team. The report will be submitted to the USEPA and National Grid for their approval and regulatory review.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:
The Data Validation Report will present the findings of the data evaluation processes. Resulting data quality and conformance with evaluation guidelines will be presented.

References

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NYSDEC. 2005. Analytical Services Protocol (ASP)

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USEPA, 2006b. SOP NO. HW-37/Aroclor Validation of Data USEPA Contract Laboratory Program for Organic Analysis of Low/Medium Concentration of Aroclor Organic Compounds SOM01.1, Revision 0, April 2006.

USEPA, 2007a. Contract Laboratory Program Guidance for Field Samplers, OSWER 9240.0-44, EPA 540-R-07-06, July 2007.

USEPA, 2007b. Introduction to the Analytical Services Branch (ASB) Contract Laboratory Program, EPA 540-R-07-05, OWER 9240.0-42, January 2007.

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USEPA, 2007d. Modifications Updating SOM01.1 To SOM01.2 October 5, 2005, (Updated 02-12-2007) Amended 04-11-2007).

USEPA, 2007e. Basic Manual for Electronic Data EPA Region 2, Version 1.1, November 2007.

Attachment A

Regulations and Guidance Documents (Electronic)

New York State
Department of Environmental Conservation

Division of Fish, Wildlife and Marine Resources

Technical Guidance for Screening Contaminated Sediments

GEORGE E. PATAKI, *Governor*

JOHN P. CAHILL, *Commissioner*

New York State Department of Environmental Conservation
Division of Fish, Wildlife and Marine Resources

Technical Guidance for Screening Contaminated Sediments

Change Sheet for January 25, 1999

This document is a reprint of the original "Technical Guidance for Screening Contaminated Sediments" that was first printed in November 1993, and subsequently reprinted in July 1994 and March 1998, with the following changes noted:

- Additional sediment screening values have been added to Table 1 for benzene, toluene, ethylbenzene, xylene, and nine polycyclic aromatic hydrcarbon compounds. The 13 new substances have not been integrated alphabetically into table 1. They are listed separately as an aditional page (page 25).

In all other respects, this edition is an exact reprint of the editions dated November 1993, July 1994, and March 1998 w/changes

New York State Department of Environmental Conservation
Division of Fish, Wildlife and Marine Resources

Technical Guidance for Screening Contaminated Sediments

Change Sheet for March 2, 1998

This document is a reprint of the original "Technical Guidance for Screening Contaminated Sediments" that was first printed in November 1993, and reprinted in July 1994, with the following changes noted:

- The Division of Fish and Wildlife and the Division of Marine Resources were merged into a single entity, the Division of Fish, Wildlife and Marine Resources

- New tables have been added for screening marine and estuarine sediments only. The new tables have been taken from Long et al (1995), and are included as appendix 4. These tables have been distributed with earlier editions of this document as an addendum since April 25, 1996. Wherever the current text makes reference to Table 2 for screening sediments for metals contamination, Table 3 in Appendix 4 should be used instead if the sediments are in marine or estuarine water bodies.

In all other respects, this edition is an exact reprint of the November 1993 and July 1994 document.

New York State Department of Environmental Conservation
Division of Fish and Wildlife
Division of Marine Resources

Technical Guidance for Screening Contaminated Sediment

22 November 1993

(reprinted July 1994, March 1998, January 1999)

This document describes the methodology used by the Division of Fish and Wildlife and the Division of Marine Resources for establishing sediment criteria for the purposes of identifying contaminated sediments. Sediments with contaminant concentrations that exceed the criteria listed in this document are considered to be contaminated, and potentially causing harmful impacts to marine and aquatic ecosystems. These criteria do not necessarily represent the final concentrations that must be achieved through sediment remediation. Comprehensive sediment testing and risk management are necessary to establish when remediation is appropriate and what final contaminant concentrations the sediment remediation efforts should achieve.

- ORIGINAL SIGNED -
Kenneth F. Wich
Director
Division of Fish and Wildlife

- ORIGINAL SIGNED -
Gordon Colvin
Director
Division of Marine Resources

Table of Contents

1. Executive Summary

The Department of Environmental Conservation originally proposed sediment criteria in 1989, as an appendix of a Cleanup Standards Task Force Report. These criteria were controversial because the proposed methodology, equilibrium partitioning, had not yet been endorsed by the U.S. Environmental Protection Agency (EPA) Science Advisory Board, and because the criteria themselves were perceived as remediation target concentrations. This revised sediment criteria document was prepared to incorporate scientific literature published since 1989, and to establish the purpose of sediment criteria for screening; that is, to identify areas of sediment contamination and to make a preliminary assessment of the risk posed by the contamination to human health and the environment. Criteria are developed for two classes of contaminants - non-polar organic contaminants and metals. Non-polar organic contaminant criteria are derived using the equilibrium partitioning approach, which has now been endorsed by the EPA Science Advisory Board. This approach estimates the biological impacts that a contaminant may cause based on it's affinity to sorb to organic carbon in the sediment. The concentration of biologically available contaminant is predicted and related to potential toxicity and bioaccumulation by using existing criteria established for the water column. New York State water quality standards and guidance values are used to derive sediment criteria. EPA water quality criteria are used only when New York State has not published a standard or guidance value for a particular compound. Water quality criteria for bioaccumulation proposed by the Divisions of Fish and Wildlife and Marine Resources are used when no New York State water quality standard or guidance value for bioaccumulation has been developed.

Metals criteria are derived from Ministry of Ontario guidelines and NOAA data that make use of the screening level approach. This methodology measures the concentration of contaminants present in areas where ecological impacts have been noted, and correlates the contaminant concentration with the severity of the impact. Toxicity mitigating conditions such as acid volatile sulfides are not considered because with the screening level approach, the metal concentrations present are correlated directly to a measurable ecological impact. Finally, this document discusses risk management for contaminated sediment, and makes recommendations for implementing sediment criteria. Table 1 lists sediment criteria for 64 non-polar organic compounds or classes of compounds, and Table 2 lists sediment criteria for 12 metals.

II. Background and Objectives

The Department of Environmental Conservation originally proposed draft sediment criteria in December 1989 as Appendix D to the Draft Clean Up Standards Task Force Report (DEC 1991). These criteria were based on the EPA equilibrium partitioning (EP) model, which had at that time just been submitted to the EPA Science Advisory Board for review. Two problems developed relative to these criteria. The first was that the equilibrium partitioning model did not receive a complete endorsement by the EPA Science Advisory Board (EPA SAB 1990). The SAB raised questions about the degree of uncertainty, sources of variability, and applicability of EP-based sediment criteria. Secondly, the New York State sediment criteria were published in the context of a clean-up standards report for contaminated sediment remediation. The perception of the reviewers and potential users was that the criteria represented mandatory clean-up levels that must be achieved by remediation methodologies. Appendix D of the Draft Clean-up Standards Task Force Report did state that risk management decisions were necessary and appropriate in the application of the sediment criteria, but the perception remained that the low concentrations described therein were in fact the primary target levels for sediment remediation. This issue was further clouded by real-world environmental problems such as dioxin in the New York-New Jersey Harbor area. Dredging and dredge spoil disposal is necessary for continued harbor operation, but attainment of the dioxin sediment criterion described in Appendix D could be economically unachievable.

There were three objectives for revising the sediment criteria document. The first objective was simply to clarify the document, make it easier to read, and provide greater scientific documentation to support the information presented.

The second objective was to incorporate scientific literature that has been published since 1989. This revision will be based primarily upon an EPA Proposed Technical Support Document (TSD) for the Development of Sediment Quality Criteria (EPA 1991). The EPA TSD was also published verbatim in peer-reviewed scientific literature (DiToro et al., 1991). The revised sediment criteria document will also incorporate a new EPA Science Advisory Board Report that endorses the equilibrium partitioning methodology and commends the EPA for satisfactorily addressing many of the concerns noted in the original SAB review (EPA SAB 1992). Also, this revision incorporates the 1992 Ministry of Ontario Guidelines for the Protection and Management of Aquatic Sediment Quality in Ontario, for metals concentrations in sediment (Persaud et al., 1992). These guidelines were only draft in 1989, when the first sediment criteria document was produced.

The final objective of the revised document was to establish the role of EP-based sediment criteria as screening criteria; that is, for identifying areas of sediment contamination, and providing an initial assessment of potential adverse

impacts. While attainment of the EP-based sediment criteria will provide the maximum assurance of environmental protection, it is not necessary in all cases and at all times to achieve these criteria through remediation efforts. Risk assessment, risk management, and the results of further biological and chemical tests and analyses are vital tools for managing sediment contamination. To view sediment criteria in a one-dimensional, go/no go context is to miss potential opportunities for resource utilization through appropriately identified and managed risk.

Ill. Need, Basis, and Concept of Sediment Criteria

Sediments can be loosely defined as a collection of fine-, medium-, and course- grain minerals and organic particles that are found at the bottom of lakes [and ponds], rivers [and streams], bays, estuaries, and oceans (Adams et al., 1992). Sediments are essential components of aquatic [and marine] ecosystems. They provide habitat for a wide variety of benthic organisms as well as juvenile forms of pelagic organisms. The organisms in sediments are in constant contact with the sediments, and therefore, constant contact with any contaminants that may be adsorbed to the sediment particles. Potential impacts to benthic organisms include both acute and chronic toxicity with individual-, population-, and community- level affects, bioaccumulation of contaminants, and the potential to pass contaminants along to predators of benthic species (Adams, et al, 1992; Marcus, 1991; Milleman and Kinney, 1992).

Potential to harm benthic organisms is not the only adverse impact of contaminated sediments. They serve as diffuse sources of contamination to the overlying water body; slowly releasing the contaminant back into the water column (Marcus, 1991; DEC, 1989).

Contamination is a concept that is not always clearly defined relative to sediments. The mere presence of a foreign substance in a sediment could be construed as contamination. However, the presence of a foreign substance does not necessarily mean it is harmful. Metals can be present in naturally occurring concentrations (background levels) in species, or forms, that are not harmful to aquatic life. While there are no naturally occurring background concentrations for synthetic organic compounds, the presence of a synthetic organic compound does not necessarily imply harm. Some evaluation must be made to estimate the potential risk to aquatic life or human health that the compound will have.

The EPA has defined a contaminant as: "Any solid, liquid, semisolid, dissolved solid, gaseous material, or disease-causing agent which upon exposure, ingestion, inhalation, or assimilation into any organism, either directly from the environment or indirectly by ingestion through food chains, may . . . pose a risk of or cause death, disease, behavioral abnormalities, cancer, genetic mutations,

physiological malfunctions ... or physical deformations, in the organism or their offspring" (EPA, 1992). This definition clearly explains that a contaminant is not simply the presence of a foreign substance, but an element of harm to some organism, species, population, or community must be involved.

The EPA defines sediment criteria in the following manner: A sediment criterion is a specific level of protection from the adverse effects of sediment associated pollutants, for beneficial uses of the environment, for biota, or for human health ... (EPA, 1992). A sediment criterion, then, must relate to the element of harm that the contaminant possesses by specifying an appropriate level of protection. To develop sediment criteria, it is necessary to identify the potential elements of harm to the various organisms, populations, and communities that could be affected. The criterion must then specify the level of protection necessary to balance each identified element of harm.

A corollary of the EPA definition is that if the specified level of protection is not attained, then a certain level of risk exists. The concentration of a contaminant in sediment can be compared to a number of criteria and their associated levels of protection, to determine the overall potential risk posed by that particular contaminant concentration to various exposed organisms. Only if the contaminant concentration is less than all of the available criteria can exposure to the sediment, or to organisms that inhabit the sediment, be considered to be without significant risk from those contaminants (risk could still result from other sources, such as contaminants for which criteria have not yet been derived). This is the concept of screening criteria. By comparing the contaminant concentration to various criteria and their associated levels of protection, the resource manager can begin to identify the appropriate tests, studies, and procedures to quantify and refine the level of risk; set remediation goals; prioritize remediation actions; and select risk management and communications options.

EP-based sediment criteria are tied to water quality standards, guidance values, (DEC, 1991) and criteria (EPA, 1991)[1]. Within the framework of New York State water quality regulations, five primary levels of protection are identified (6NYCRR, 1991) from which sediment criteria can be derived. These are:

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[1]Water quality standards and guidance values are New York State regulatory terms that are essentially synonymous with the EPA term criterion. A standard is a water quality criterion that has been adopted into regulation. A guidance value is a water quality criterion that has been derived in the same manner as a standard, but has not yet been adopted into regulation, or subjected to public review and comment. When referring to water quality in this document, the use of the general term criteria will mean either a New York standard or guidance value.

A. Protection of human health from acute or chronic toxicity;

B. Protection of human health from toxic effects of bioaccumulation;

C. Protection of aquatic life from acute toxicity;

D. Protection of aquatic life from chronic toxicity;

E. Protection of wildlife from toxic effects of bioaccumulation.

Other levels of protection include fish flesh tainting, and aesthetics (taste, odor, or appearance). Human health-based criteria can be further subdivided into oncogenic (cancer causing) effects and non-oncogenic effects (6NYCRR, 1991). Unfortunately, water quality standards or guidance values do not usually exist for all five levels of protection simultaneously.

This document will identify a series of screening criteria concentrations for a number of contaminants that can be used to identify areas of sediment contamination, and evaluate the potential risk that the contaminated sediment may pose to human health or the environment. A contaminated sediment can be identified as one in which the concentration of a contaminant in the sediment exceeds any of the sediment criteria for that contaminant. Once a sediment has been identified as contaminated, a site-specific evaluation procedure must be employed to quantify the level of risk, establish remediation goals, and determine the appropriate risk management actions. The site-specific evaluation might include for example: additional chemical testing; sediment toxicity testing; or sediment bioaccumulation tests.

Sediment contaminants. primarily consist of heavy metals and persistent organic compounds (EPA, 1990). Sediment criteria for non-polar organic compounds are derived using equilibrium partitioning methodology (EPA, 1991, DiToro, et al., 1991). This document will derive sediment criteria for non-polar organic contaminants listed in the TOGS 1.1.1. (DoW, 1991), using the water quality standards and guidance values listed there. If a water quality criterion for a particular contaminant is not identified in TOGS 1.1.1., an EPA water quality criterion is used. These criteria are annotated with the suffix (E). Proposed water quality criteria for the protection of human health and piscivorous wildlife from bioaccumulative affects are derived using procedures identified in Appendix 1; Newell et a[. (1987); and 6NYCRR Parts 702.8 and 702.13. These criteria are annotated with the suffix (P). With the exception of PCBs, these water quality guidance values are not yet listed in TOGS 1.1.1.

Sediment criteria for metals are based upon procedures and data developed by the Ministry of Ontario (Persaud et al., 1992), and the National Oceanic and

Atmospheric Agency (NOAA) (Long and Morgan, 1990). Sediment criteria for polar organic compounds are not derived. Instead, contaminant concentrations in pore water should be compared directly to surface water quality criteria; see section V. Some polar organics such as phenolic compounds behave as non-polar compounds under conditions of neutral pH. For these compounds, EP-based sediment criteria can be derived. Both the equilibrium partitioning methodology and the Ministry of Ontario procedures are discussed below.

IV. Derivation of Sediment Quality Criteria for Non-polar Organic Compounds using Equilibrium Partitioning.

A. Characteristics of Non-polar Organics

Non-polar organic compounds are substances that contain carbon, and do not exhibit a net electrical (ionic) charge (Nebergall, et al. 1968). Non-polar organic contaminants tend to be of low solubility in water. Otherwise they would dissolve and not accumulate in sediments (Manahan, 1991). Many non-polar contaminants are highly soluble in lipids, and thus can be bioaccumulated. They are persistent, meaning they do not break down or degrade rapidly, and can remain in sediments for long periods of time. The International Joint Commission defines persistent compounds as compounds with a half life greater than 56 days (IJC, 1978). Some contaminants such as pesticides can cause direct, acute toxicity to exposed benthic organisms in low concentrations. Others such as DDT, PCB, and dioxin are more insidious and bioaccumulate over time to cause chronic toxicity effects such as reproductive failure, either in populations exposed directly to the contaminated sediment or to organisms further up the food chain (Rand and Petrocelli, 1985).

B. Fundamentals of Equilibrium Partitioning (EP)

The basis for the EP methodology for deriving sediment criteria is that the toxicity of a contaminant in a sediment is attributable to the fraction of the contaminant that dissolves in the interstitial pore water, and is considered to be freely biologically available. The EP methodology predicts the concentration of contaminant that will dissolve in the interstitial pore water from three factors: 1) the concentration of contaminant in the sediment; 2) the concentration of organic carbon in the sediment; and 3) the affinity of the contaminant for organic carbon in the sediment.

The affinity of a contaminant for sediment organic carbon can be directly measured. The sediment/water partition coefficient or K_P is a measure of the concentration of a contaminant sorbed to the sediment divided by the concentration dissolved in water (measured in l/kg), after mixing. The K_P is only useful as a site specific measure because the K_P will vary with different sediment

samples. The EPA (1991) reported that the organic carbon content of a sediment accounts for most of the variation in the uptake of the contaminant by the sediment. The K_{OC}, or sediment organic carbon/water partition coefficient is a measure of the concentration of contaminant that adsorbs to the organic carbon content of the sediment divided by the concentration dissolved in water, after mixing (measured in l/kg). When normalized for organic carbon, concentrations of a contaminant in different sediment samples are comparable. Another partition coefficient that is closely correlated with K_{OC} and is useful for predicting soil adsorption is the octanol/water partition coefficient, or K_{OW} (Kenaga, 1980). Voice, et al. (1983) citing Karickhoff (1979), reports that the relationship between the three coefficients can be described in two equations:

$$K_{OC} = K_P/f_{OC}$$

and

$$\log_{10}K_{OC} = \log_{10}K_{OW} - 0.21 \quad \text{(also in Kenaga, 1980)}$$

where f_{OC} is the fraction of solids by weight that is comprised of organic carbon.

The EPA (1991) refers to DiToro (1985) to define the relationship between K_{OC} and K_{OW} as:

$$\text{Log}_{10}K_{OC} = 0.00028 + 0.983\log_{10} K_{OW}$$

Using the DiToro (1 985) relationship, the Koc very nearly equals the K_{OW}. Using either relationship, it can be readily seen that the Koc and Kow for a given non-polar organic compound are very similar, and vary in direct proportion. In their initial review of the equilibrium partitioning methodology, the EPA SAB considered the equating of K_{OC} and K_{OW} to be a source of uncertainty (EPA SAB 1990). In their 1992 review, the EPA SAB states that uncertainties have diminished largely as a result of more accurate determination's of K_{OW}s, and that occasionally the Kow may not be a good predictor of the Koc (EPA SAB 1992).

When a non-polar organic contaminant enters the sediment, it will partition between the sediment and pore water in three compartments: a fraction will adsorb to the organic carbon in the sediment; another fraction will adsorb to dissolved organic carbon in the interstitial pore water; and a third fraction will dissolve in the pore water. An equilibrium will be established so that any change in the contaminant concentration in one compartment will result in a corresponding change in the contaminant concentration in other compartments. For example, if some of the contaminant dissolved in the pore water is removed, some of the contaminant adsorbed to the sediments will desorb to balance the loss from the pore water. If dissolved contaminant is added to the pore water, it will not all

remain in the pore water, but some will adsorb to dissolved organic carbon and sediment organic carbon, re-establishing the equilibrium. Interestingly, the EPA (1991) noted that an increase in the volume of dissolved organic carbon in the pore water causes contaminant sorbed to the sediment to desorb and in turn sorb to the dissolved organic carbon. The freely dissolved fraction of the contaminant remains practically unchanged.

Equilibrium partitioning methodology contends that sediment toxicity is attributable to the concentration of contaminant dissolved in the interstitial pore water and considered to be biologically available (EPA 1989, EPA 1991). It can be inferred, then, that a water quality criterion developed to protect aquatic life from contaminants dissolved in the water column should also protect benthic aquatic life from contaminant concentrations dissolved in pore water. The EPA (1991) compared the sensitivity of benthic organisms to the sensitivity of water column organisms to toxicity from the same chemicals, and found that they were very similar. Therefore the prediction that exceeding a water column-based criterion in sediment pore water would harm benthic organisms was considered valid.

C. Derivation of Sediment Criteria using Equilibrium Partitioning

To derive an organic carbon normalized sediment criterion, two items of information are required:

A. An ambient water quality criterion for a particular contaminant;

B. the K_{OW} partition coefficient for the contaminant;

For example, the PCB water quality criterion (see footnote 1 on page 4) for the protection of piscivorous wildlife from bioaccumulation is 0.001 ug/l. The K_{OW} for PCB is $10^{6.14}$, or 1,380,384.3 l/kg. The organic carbon normalized PCB sediment criterion (SCoc) would be:

$$SC_{OC} = WQC * K_{OW}$$

$$PCB\ SC_{OC} = 0.001\ /ug/l * 1{,}380{,}384.3\ l/kg * 1\ kg/1{,}000\ gOC$$

$$1.38\ (\approx 1.4)\ \mu g/gOC$$

1 kg/1,000 gOC is a conversion factor.

The meaning of the criterion is: based on the equilibrium partitioning characteristic of PCBs, in order not to exceed the water quality criterion of 0.001 ug/l in the pore water, the concentration of PCB in the sediment must not exceed 1.4 μg for each gram of organic carbon in the sediment.

To apply this SC_{OC} on a site specific basis, the concentration of organic carbon in the sediment at the site must be known. If a sediment sample was known to contain 3% organic carbon, the site specific sediment criterion (SC) for PCB could be derived:

$$SC = SC_{OC} * f_{OC}$$

f_{OC} = 3% OC/kg sediment = 30 gOC/kg

PCB SC = 1.4 μg/gOC * 30 gOC/kg = 42 μg PCB/kg sediment

This criterion states that: if there are less than 42 ug PCB/kg of sediment in a sediment containing > 3% organic carbon, there is no appreciable risk to piscivorous wildlife from consuming fish or other aquatic life from the water body over the contaminated sediment.

D. Limitations of Equilibrium Partitioning Derived Sediment Criteria

There are several limitations to the application of EP-based criteria:

1. EP-based criteria are only applicable to non-polar organic compounds, or other substances that behave as non-polar organic compounds in the sediment and prevailing environmental conditions, such as pH.

2. EP-based criteria apply only to the specific level of protection identified in the criterion. In the example above, the 42 μg/kg PCB concentration in the 3% sediment sample does not pose appreciable risk to wildlife, however, it may or may not pose a risk to human beings. A sediment criterion derived from a human health-based water quality criterion must be compared to make that determination.

3. EP-based criteria should only be derived for sediments with organic carbon fractions between approximately 0.2 - 12% (EPA SAB, 1992). Outside of this range, other factors that the EP methodology does not account for may influence contaminant partitioning.

4. The equilibrium partitioning method should not be applied to broad classes of compounds or mixtures if one K_{OW} value is used to represent the entire class or the mixture (EPA SAB, 1992). In this respect, PCB congeners would not be considered a broad class of compounds; they are a narrow class of quite similar compounds.

5. For compounds with a K_{OW} less than 100 ($\log_{10}K_{OW} \leq 2$), the water quality criterion can be greater than the site specific sediment quality

criterion. This implies that virtually all of the contaminant is biologically available. Since the water quality criterion delineates the concentration that is harmful to aquatic life, it is not reasonable that a smaller concentration in the sediments would be harmful to benthic organisms, especially considering that some fraction of the contaminant will be sorbed to the sediment and not biologically available. For these compounds, the organic carbon normalized sediment criterion should be derived in the manner described above. However, when determining the site specific criterion, compare the product of the SC_{OC} * f_{OC} with the water quality criterion, converted from a volumetric to mass units (μg/l * 1/kg = μg/kg). If the water criterion is greater than the site specific sediment quality criterion, use the water quality criterion as the sediment criterion. For example, the $\log_{10}K_{OW}$ of benzidine is 1.4. The SC_{OC} for the protection of benthic life (chronic toxicity), based on a TOGS 1.1.1. water quality criterion of 0.1 μg/l is 0.003 μg/gOC. If the sediment contained 3% organic carbon, the site specific SC would be 0.09 μg/kg. The water quality criterion (converted from a volumetric measure to a mass measure) of 0.1 μg/kg is greater, so the site specific sediment criterion should be 0.1 μg/kg. If the site contained 5% organic carbon the site specific sediment criterion would be 0.15 μg/kg, which is greater than the water quality criterion of 0.1 μg/I. In this instance, the 0.15 μg/kg would be the appropriate criterion to use.

6. Derivation of EP-based criteria assumes that an equilibrium between the sediment/pore water compartments has been achieved. Rand and Petrocelli (1985) indicate that the sorption-desorption equilibria are achieved rapidly, usually in a few minutes to several hours. Voice et al. (1983) found that in laboratory studies, equilibria were generally achieved in about 4 hours. In investigating contamination of stable sediments with long term exposure to a contaminant, it is likely that equilibrium has been achieved. However for spill sites, and areas with unstable sediments, attainment of the equilibrium condition may be questionable. The EPA SAB (1992) recommends that EP-based criteria not be used in areas of rapid deposition or erosion (e.g.>10 cm/yr), such as active dredge disposal areas, areas of heavy boat and barge traffic, and some river channels.

7. The EP methodology is not a highly accurate procedure in and of itself. Several related sampling and analysis procedures could introduce additional variation and uncertainty into the results. Some of these factors include: the value of the K_{OW} used and how it was derived; how the sediment sample was taken and analyzed for contaminant content; and how the organic content of the sediment sample (f_{OC}) was determined. For consistent application of sediment criteria, these factors must be considered systematically and consistently. ASTM (1993) recommendations should be followed for the proper collection, storage, and analysis techniques when

applying EP-based sediment criteria. The analysis method is particularly important for determination of sediment total organic carbon, because there are several methods available that may give variable results. The authors and EPA (1992b) recommend the use of catalytic combustion with nondispersive infrared carbon dioxide detection (Leonard, 1991) when developing total organic carbon-normalized criteria for non-polar organic compounds. However, unless the "true" K_{OW} differs by a factor of 10, or the 'true' f_{OC} differs by 50 - 100% from the K_{OW} and f_{OC} values used to derive the sediment criteria, the level of imprecision introduced into the criteria calculation will be minor. An EP-based criterion applies to a single sediment sample. Results obtained from composite samples may be misleading in that the contaminant concentration at a single point or depth might be diluted with uncontaminated samples. Conversely, a contaminated sample mixed with uncontaminated samples from other points or depths might cause a greater area appear to be contaminated than actually is.

8. There are still a number of uncertainties related to equilibrium partitioning-derived sediment criteria. These include such factors as particle size, particle density, organic carbon content, K_{OW}/K_{OC} relationship, route of exposure, the impact of dissolved organic carbon, and the uncertainty of extrapolating laboratory data to field conditions (EPA, 1991; EPA SAB, 1992). Despite these uncertainties, the EPA has found that sediment toxicity from laboratory experiments generally falls within a factor of 5 of the toxicity predicted by equilibrium partitioning. EP-based criteria are considered to be valid for screening and assessment. These preliminary assessments can be followed up with further testing if necessary to more accurately quantify risk.

Table 1 lists 52 non-polar organic compounds or classes of compounds for which sediment criteria have been derived using the equilibrium partitioning methodology. The derivation procedure is the same as that recommended by the EPA (1991). The only difference is that New York State water quality standards and guidance values are used instead of EPA ambient water quality criteria. EPA criteria have been used to derive a sediment quality criterion only when a New York standard or guidance value is not available. Four criteria, corresponding to four of the five levels of protection, are listed for each contaminant whenever possible. Sediment criteria are not derived for the protection of human health from toxicity, because that type of exposure would constitute human consumption of the interstitial pore water within the contaminated area, which is an unreasonable assumption. A sediment is considered to be contaminated if the contaminant concentration exceeds any of the criteria listed. The table also identifies the K_{OW} and the water quality criterion used to derive the sediment criterion. Water quality criteria are from DoW TOGS 1.1.1., unless suffixed with an (E), which indicates an EPA water quality criterion. Proposed water quality criteria for the protection of human health and piscivorous wildlife from bioaccumulative effects are used when

no TOGS 1.1.1. criterion for bioaccumulation has been developed. These criterion are annotated with the suffix (P), and are derived according to the method described in Appendix 1 and Newell et al. (1987).

V. Polar Organics - Application of Water Quality Criteria to Pore Water via Direct Measurement of Pore Water

For polar organics (except for phenols) no algorithms have been developed yet for sediment criteria that account for sediment characteristics which may affect substance toxicity. However, in order to screen sediments for potential impacts from polar organic compounds, interstitial (pore) water from sediment samples should not exceed existing water quality standards and guidance values for polar organics in TOGS 1. 1. 1.

The application of these criteria to pore water is complicated by dissolved organic carbon (DOC) in pore water that is generally much higher than DOC in the water column. DOC tends to reduce toxicity and bioaccumulation of chemicals by reducing their availability for uptake by the organism. However, even though water column DOC is usually low, water quality criteria are not modified to account for the effects of DOC. If the partitioning coefficient between DOC and water for a contaminant is known, that coefficient could be used to account for the effect of DOC on toxicity or bioaccumulation in the application of water quality criteria to pore water. The bioaccumulation of contaminants with low K_{OW} is generally not suppressed by water column DOC, indicating that the effects of DOC can probably be ignored. In any case, a conservative risk assessment is assured if the effects of DOC in pore water are ignored during a preliminary screening. In follow-on assessments, DOC affects should be evaluated. As a consequence, the water quality criteria becomes the pore water criteria, and sediment criteria per se are not derived for these compounds.

VI. Derivation of Sediment Quality Criteria for Metals

A. Characteristics of Metals as Sediment Contaminants

A wide variety of metals in a wide variety of forms can be found in marine and aquatic sediments. Some concentrations occur naturally, while others have been introduced through man's activities. Very low concentrations of most metals are required nutrients for living organisms, but in excess concentrations, metals can be harmful (Rand and Petrocelli, 1985). The properties that metals exhibit in water depend largely on the form in which the metal occurs (Manahan, 1991). In waterbodies, metals are typically found (Demayo et. a[, 1978):

1. Dissolved as free ions and complexes;

2. As particulates:

a. inorganic precipitates such as hydroxides, sulfide, carbonates, and sulfates;

b. sorbed onto or complexed with high molecular weight organic compounds or clay particles;

3. Mixed or sorbed to bottom sediments;

4. Incorporated into the tissues of biota.

The toxicity and bioavailability of metals in water [and sediment] vary with the form of the metals (EPA 1992a). The form of the metal, and thereby the toxicity of a metal, are highly influenced by environmental conditions such as pH, alkalinity, REDOX potential, and the availability of complexing ions or ligands. Very generally, it can be said that the dissolved fraction of metals seems to account for most toxicity, however, some particulate forms of some metals also exhibit toxicity (EPA 1992a).

Metals in water can generally be measured as total (total recoverable) dissolved metal. Currently, the EPA recommends using water effects ratios for evaluating the impact of metals on surface water quality (EPA 1993). Conduct toxicity tests using water from a specified site, and compare the toxicity with reference toxicity tests in relatively pure water. The resulting "water effects ratio" can then be used to adjust either a total recoverable metal criterion or effluent limitation, or dissolved metals water quality criterion (preferred in areas of highly variable suspended solids concentrations) to account for local conditions.

In sediments, metals exhibit the same variety of forms as in water; they can dissolve as ions or soluble complexes in the interstitial pore water, precipitate as organic or inorganic compounds, or sorb to binding sites in the sediment. The complexity of metals behavior in water and sediments makes it impossible to accurately predict the levels at which toxic effects will occur. For metals, the primary concern in sediments is toxicity to benthic organisms. Metals can bioaccumulate in organisms. Bioaccumulation of metals is highly variable and dependent on the form of the metal and how it enters the organism (Doull et al., 1980). Different organs and tissues will have different affinities for different metals and species of metals. Metals can be absorbed by an organism but be bound by proteins known as metallothioneins into relatively harmless forms. Toxicity of metals are dependent on many environmental conditions and are difficult at best to predict consistently.

B. Establishing Screening Level Concentrations

Because of the inability to predict biological affects from metals concentrations in sediment, the best alternative is to identify adverse ecological effects that are attributable to sediment-borne metals concentrations, and measure what concentration caused the adverse effect. The Ontario Ministry of the Environment issued metals guidelines derived by the "Screening Level Concentration" approach. This is an effects-based approach which uses field data on co-occurrence of benthic animals and contaminants (Persaud et al., 1992). The Ontario guidelines span background, lowest effect levels and severe effect levels. The methods used to derive these guidelines do not account for the effects of organic content, acid volatile sulfide concentration, particle size distribution or iron and manganese oxide content, or other toxicity-mitigating factors on the bioavailability of metals within the sediments, because the total metals concentration is related directly to an observed, measurable ecological effect. It is possible that this methodology might not discern toxicity from other compounds besides metals.

Long and Morgan (1990) reviewed and categorized chemical effects data in sediments according to low and median toxic effects ["Effects Range-Low (ER-L)" and "Effects Range-Median (ER-M)" concentrations] and "Overall Apparent Effects Thresholds" for benthic organisms observed in field studies across the nation. Effects levels reported were associated with bulk sediment concentrations without normalizing for any toxicity mitigating factors. For metals, effects levels in Long and Morgan (1990) may be compared with effects levels taken from Persaud et al. (1992). Both are based on a selection of observed effects from field studies, although Persaud et al. (1 992) is restricted to Great Lakes data while Long and Morgan (1 990) used both fresh and salt water data. For six metals (arsenic, cadmium, chromium, copper, lead and nickel), the lowest effects levels described by Persaud et al. (1992) are lower than the ER-L (effects range-low) from Long and Morgan (1990). This could be because in the relatively pure waters of Lake Ontario, fewer ligands were available to complex metal ions, so biological affects were noted at lower metals concentrations. The Long and Morgan (1990) study included more eutrophic waters, wherein, metals could be complexed to a greater extent into biologically unavailable forms. Exposed organisms were able to tolerate higher total metals concentrations because the greater fraction of metal present was biologically unavailable.

To establish screening criteria for sediments in New York State, two levels of protection as a basis sediment quality screening criteria were established, following the Ministry of Ontario Guidelines definitions. These are the Lowest Effect Level and the Severe Effect Level. The Lowest Effect Level indicates a level of sediment contamination that can be tolerated by the majority of benthic organisms, but still causes toxicity to a few species. The Severe Effect Level indicates the concentration at which pronounced disturbance of the sediment

dwelling community can be expected (Persaud et al. 1992). The ER-L and ER-M

from Long and Morgan (1990) were compared with the Lowest Effect Level and Severe Effect Level from Persaud et al. (1 990). The lowest concentration in each of the two effect levels was selected as the New York sediment screening criteria. These sediment criteria for metals are listed in Table 2. If a total metals concentration in a sediment sample is less than the Lowest Effect Level listed in Table 2, the effects of the metal in the sediment are considered to be acceptable. If the concentration is greater than the lowest effect level but less than the severe effect level concentration, the sediment is considered to be contaminated, with moderate impacts to benthic life. If the concentration is greater than the severe effect level, the sediment is contaminated and significant harm to benthic aquatic life is anticipated.

Background concentrations described in Persaud et al. (1992) were not used to establish criteria. For some metals, cadmium and copper for example, Persaud lists a Lowest Effect Level that exceeds the typical background concentration. Because a metal concentration in sediment is considered to be naturally occurring, or background, does not mean that the concentration is not causing an adverse ecological effect.

As noted above, metals guidelines from Persaud et al. (1992) are based on freshwater sediments only, and effects levels in Long and Morgan (1990) reflect data from both fresh and salt water. Although differences in the bioavailability of metals in fresh and salt water sediments may be elucidated in the future, at this time, the sediment criteria identified in Table 2 are considered suitable for identifying areas of metal contaminated sediment, assessing potential risk, and identifying suitable follow-up tests, studies, and risk management options in both fresh and salt water sediments.

C. Limitations to Sediment Criteria for Metals

There are limitations to the application of the metals sediment quality criteria listed in Table 2:

1. Persaud et al. (1 992) values are based on oligotrophic waters with low concentrations of metals-complexing ligands. These criteria are possibly over-protective when applied to more eutrophic waters. However, many streams and ponds in New York are oligotrophic, and the low effects concentrations are justified. These criteria are intended to be used for screening; that is, to identify potentially contaminated sites and provide a qualitative estimate of risk. Once a site is found to be contaminated with metals, further studies are necessary to quantify risk and determine if remediation actions are necessary. Remediation should not be based solely on exceedances of these criteria.
2. These criteria have limited applicability to mixtures of metals. Metals

criteria are most clearly applicable to sediments with high concentrations of a single metal, or situations where one metal has a disproportionately greater abundance in a sediment sample than any other metal. The presence of one metal can significantly affect the impact that another metal has on an organism. The effect can be synergistic, additive, or antagonistic (Eisler, 1993). A reasonable level of protection can be expected if none of the criteria are exceeded for metals that are present, however, effects may be present if the sum of the fractions of criteria over sediment concentrations exceed one, for all of the metals present. For example, in a sediment sample, four metals are detected. The concentration of each metal in the sediment sample is 0.3 of its corresponding sediment criterion. The sum of the fractions would be 1.2. In this case, further testing is warranted.

3. Total metals, or the bulk metals concentration should be measured in sediment samples.

VII. Use of Sediment Criteria in Risk Management Decisions

Once it has been determined that a sediment criterion is exceeded, more information is required to determine if remediation is necessary and what actual risks to the environment are present. The volume and location of sediment exceeding a criterion, which levels of protection are exceeded, the persistence of the contaminant, the uncertainty about the criteria, and the results of more detailed, site specific sediment tests all play a role in making decisions about how, and how much sediment to clean up in order to eliminate or minimize adverse effects. If the volume of sediment that exceeds sediment criteria is small and the sediment is fairly accessible, the remediation of all contaminated sediment may be the most expedient action. If volumes of sediment are large and/or difficult to remediate either because of accessibility, sensitivity of the impaired habitat, or lack of efficacious technology, further risk management evaluations are warranted. In general the areal extent of the contaminated sediments should be a factor in considering the need for, and method of remediation.

Once the source of contaminants to sediments is terminated, the length of time a particular area of sediments remain contaminated will depend on the persistence of the chemicals, and the site-specific characteristics of the sediment such as: rate of sedimentation; resuspension; and biological and chemical degradation. If a contaminant is not persistent (e.g. contaminant concentrations would be expected to fall to acceptable levels within six months to a year), and the effect of the contaminant is not severe, then sediment remediation may not be necessary. Even for a persistent contaminant, it may not be necessary to re-mediate the sediments if the contaminated area is a deposition zone, and the natural burying of the contaminated sediments beneath the zone of biological

activity and availability would be expected to occur within a short time, and resuspension of the contaminants was unlikely.

EPA SAB (1 992) examined a number of factors relating to the uncertainty of EP based sediment criteria, including sediment composition variability, measurement variation and Kow - Koc correlations and measurements. They report that all these variabilities amount to an estimated uncertainty factor of five. This suggests with good confidence that sediment criteria exceeded by a factor of five will result in the onset of toxicity. Toxicity could also result from sediment contaminant concentrations just below the sediment criterion. The EPA SAB (1992) identifies the range of concentrations from 1/5 - 5 times an EP-derived sediment criterion as a "grey" area, where observable impacts may or may not occur. Based on the statistical analysis of EP-derived sediment criteria, there is a high degree of confidence that contaminant concentrations -< 1/5 of a sediment criterion pose little or no risk. Similarly, if a contaminant concentration in sediment exceeds an EP-derived sediment criterion by a factor of 5, there is little or no doubt that adverse ecological impacts are occurring. Within the range in-between, the actual occurrence of effects is unknown. However, to avoid making the criteria excessively overprotective or under protective, the best use of the factor of 5 is in interpreting the results of sediment screening, not to modify the criteria.

The onset of chronic toxicity may be difficult to detect in natural systems. Water quality criteria designed to prevent acute toxicity are generally about ten times greater than comparable chronic criteria. Therefore, in general, sediments with contaminants at 50 times chronic toxicity sediment criteria concentrations (a factor of five for uncertainty and a factor of ten based on acute to chronic toxicity ratios), will result in the onset of acute toxicity to benthic animals with a high degree of confidence.

It must also be noted that with this uncertainty the possibility exists that the sediment criteria may be somewhat underprotective as well as than overprotective.

Sediment criteria for metals are based on empirical evidence from both lab and field studies without an attempt to normalize for any toxicity mitigating factors in the sediment. Variability of toxicity from metals in any given sediment is evident (Appendix 2). Many of the Lowest Effect Levels from Persaud et al. (1 992) are lower than the mean background concentrations in Great Lake sediments. This suggests that in some sediments relatively low levels of metals, even below mean background, are toxic, whereas in other sediments fairly high levels, up to and possibly even above background, may not be toxic. For all metals, the Severe Effect Level criteria exceeds mean background considerably; consequently, significant and noticeable toxicity is expected in all sediments that exceed that level of protection.

VIll. Implementation of Sediment Criteria for Screening

Implementation guidance can be outlined in a strategy to apply sediment criteria for screening areas suspected of sediment contamination and recommending actions to take if they are exceeded.

1. Compare sediment contaminant concentrations with sediment criteria

    a. Quantify the area and volume of sediment wherein the criteria is exceeded; determine whether biota are exposed to contaminated sediment, e.g. deeply buried sediments may be below active biological zones.

    b. Describe the significance of exceedances in terms of the predicted effects. For example, would bioaccumulation or toxicity be the predominant impact. Based on the levels of protection exceeded, evaluate whether impacts are expected to be isolated or widespread through the ecosystem of concern. Consider the potential for transport of contaminants by natural processes to other areas.

2. For naturally occurring substances such as metals, compare sediment concentrations in the area of interest with local background concentrations in areas known to be unaffected by anthropogenic sources of contamination. Evaluate sediments relative to sediment criteria to identify contaminated sites. Compare suspected contaminated sites with uncontaminated sites, looking for adverse ecological impacts.

3. If sediment concentrations of a compound are less than all of the sediment criteria for that substance, aquatic resources can be considered to be not at risk (from that compound). However, additional testing would be warranted if the concentration of numerous contaminants were just below the criteria thresholds.

4. If sediment contaminant concentrations exceed criteria, and especially if widespread in the area of interest, steps may be taken to verify the need for remediation:

    a. For sediments with non-persistent, non-polar organic contaminants that are not causing observable acute or significant chronic toxicity, further remedial investigation or sediment remediation is not necessary if the source of contamination will be eliminated and the sediment will cleanse itself. Many chemicals with $\log K_{OW} < 3$ can be expected to be non-persistent in sediments. If it is decided not to remediate sediments contaminated with non-persistent chemicals, then, assurance

must be made that water quality standards in offsite waters will not be contravened, and the public is informed of risks related to the contamination.

b. For sediments exceeding criteria based on aquatic life toxicity, including metals Lowest Effect Levels:

1. Assess the degree of impairment to the benthic community; compare site specific impairment with sediment contaminant concentrations; correlate site specific level of impairment with other known level of impairments and contaminant concentrations.

2. Collect sediment samples and conduct acute and chronic toxicity tests with fish and benthic invertebrates; correlate toxicity test results with sediment contaminant concentrations. It is important to follow established toxicity identification evaluation (TIE) techniques to ensure correct identification of the cause of toxicity, e.g. ammonia is a common cause of toxicity to benthic animals that can be mistakenly attributed to other toxics. Similarly, dissolved oxygen depletion in organically enriched sites such as wetlands could be confused with acute toxicity from contaminants.

3. For non-polar organic contaminants, exceedance of sediment criteria based on aquatic life chronic toxicity by a factor of 50 in a significantly large area indicates that biota are probably impaired and to achieve restoration of the ecosystem will require remediation of organic contaminants present.

4. For metals, if Severe Effect Levels are exceeded in significant portions of the ecosystem of concern, biota are most likely impaired and to achieve restoration of the ecosystem would likely require remediation of metals present.

C. For sediments exceeding criteria based on human health concerns:

1. Collect data on residues in edible, resident biota from the areas of concern and compare with tolerances, action levels, guidance values, or 1×10^{-6} cancer risk levels, or

2. Collect sediment samples, expose representative edible biota to sediments, measure residue in biota.

d. For sediment contaminant concentrations exceeding sediment criteria for the protection of piscivorous wildlife:

1. Collect data on residues in resident prey of piscivorous wildlife and compare with fish flesh criteria for protection of wildlife.

2. Expose wildlife food supply to contaminated sediment and measure residues in the food supply; compare with food supply residue levels known to be toxic to wildlife.

If sediment concentrations and criteria are less than analytical detection limits, ecological assessments are necessary to measure toxicity of sediments or residues in organisms exposed to sediments suspected of contamination. Generally, it is reasonable to predict that some, possibly high, levels of toxicity or bioaccumulation may associated with contaminants in sediments below analytical detection.

Table 1. Sediment criteria for non-polar organic contaminants. Water quality criteria used are taken from Togs 1.1.1. If a water quality criterion was not listed in TOGS 1.1.1., then an EPA criterion was used. These are annotated with the suffix (E). EPA criteria were extracted from the "Water Quality Criteria Summary" chart (EPA, 1991). EPA water quality criteria for the protection of human health (bioaccumulation) were taken from the "Recalculated Values - Organisms Only" column. Wildlife (bioaccumulation) and Human Health (bioaccumulation) protection criteria were derived in Appendix 1, unless TOGS 1.1.1. (bioaccumulation) criteria already existed. Although these criteria are only proposed, they are useful as guidance for estimating potential human health risks. These criteria are annotated with a suffix (P), for "Proposed criteria values".

			Levels of Protection							
			Human Health Bioaccumulation		**Benthic Aquatic Life Acute Toxicity**		**Benthic Aquatic Life Chronic Toxicity**		**Wildlife Bioaccumulation**	
Contaminant	**LogK_{ow}**	**Fresh-FW Salt -SW Both -FS**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**
Acenapthene	4.33	FW SW						140(E)[2] 240(E)[1]		
Aldrin & Dieldrin	5.0	FS	0.001	0.1					0.0077 (P)	0.77
Azinphosmethyl	2.4	FW SW					0.005 0.01	0.001 0.003		
Azobenzene	3.82	FS	0.16 (P)	1.0						
Benzene	2.0	FS	6.0	0.6						
Benzo(a)pyrene[3]	6.04	FW SW	0.0012 0.0006	1.3 0.7						

[2]EPA proposed sediment quality criterion for the protection of benthic organisms.

[3]These values also apply to benz(a)anthracene, benzo(b)fluoranthene, benzo(k)fluoranthene, chrysene, indeno(1,2,3-cd)pyrene, and methylbenz(a)anthracene.

Contaminant	$LogK_{ow}$	Fresh-FW Salt -SW Both -FS	Levels of Protection: Human Health Bioaccumulation Water Qual Criteria µg/l	Human Health Bioaccumulation Sediment Criteria µg/gOC	Benthic Aquatic Life Acute Toxicity Water Qual Criteria µg/l	Benthic Aquatic Life Acute Toxicity Sediment Criteria µg/gOC	Benthic Aquatic Life Chronic Toxicity Water Qual Criteria µg/l	Benthic Aquatic Life Chronic Toxicity Sediment Criteria µg/gOC	Wildlife Bioaccumulation Water Qual Criteria µg/l	Wildlife Bioaccumulation Sediment Criteria µg/gOC
Benzidine	1.4	FW	0.1	0.003						
Bis(2-chloroethyl) ether	1.73	FS	0.5 (P)	0.03						
Bis(2-ethylhexyl) phthalate	5.3	FW					0.6	199.5		
Carbofuran	2.26	FW			10.0	1.82	1.0	0.2		
Carbon tetrachloride	2.64	FS	1.3 (P)	0.6						
Chlordane	2.78	FW SW	0.002 0.002	0.001 0.001	2.4 (E) 0.09 (E)	1.4 0.05	0.043 (E) 0.004 (E)	0.03 0.002	0.01 (P) 0.01 (P)	0.006 0.006
Chlorobenzene	2.84	FS			50.0	34.6	5.0	3.5		
Chloro-o-toluidine	≈2.0	FS	6.5 (P)	0.65						
Chlorpyrifos	5.11	FW SW			0.083 (E) 0.011 (E)	10.7 1.4	0.041 (E) 0.0056 (E)	5.3 0.72		
DDT, DDD, & DDE[4]	6.0	FW SW	0.00001 (P) 0.00001 (P)	0.01 0.01	1.1 (E) 0.13 (E)	1100 130	0.001 (E) 0.001 (E)	1.0 1.0	0.001 0.001	1.0 1.0
Diazinon	1.92	FW					0.08	0.007		
Dichlorobenzenes	3.38	FS			50.0	120.0	5.0	12.0		
1,2 Dichloroethane	1.48	FS	24.0 (P)	0.7						
1,1 Dichloroethylene	1.48	FS	0.8 (P)	0.02						

[4]Criteria for acute and chronic benthic toxicity apply to DDT only.

			Levels of Protection							
			Human Health Bioaccumulation		Benthic Aquatic Life Acute Toxicity		Benthic Aquatic Life Chronic Toxicity		Wildlife Bioaccumulation	
Contaminant	**LogK_{ow}**	**Fresh-FW Salt -SW Both -FS**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**
Dieldrin	5.0	FW SW	0.001 0.001	0.1 0.1				9.0 (E)[5] 17.0 (E)		
Diphenylhydrazine	3.03	FS	0.54 (E)	0.58						
Endosulfan	3.55	FW SW			0.22 0.034	0.78 0.12	0.009 0.001	0.03 0.004		
Endrin	5.6	FW SW	0.002	0.8				4.0 (E)[1] 0.73 (E)[1]	0.0019 (P)	0.8
Fluoranthene	5.19	FW SW						1020 (E)[1] 1340 (E)[1]		
Heptachlor & Heptachlor Epoxide	4.4	FW SW	0.00003 (P) 0.00003 (P)	0.0008 0.0008	0.52 (E) 0.053 (E)	13.1 1.3	0.0038(E) 0.0036(E)	0.1 0.09	0.001	0.03
Hexachlorobenzene	6.18	FW	0.0001 (P)	0.15	6.0 (E)	9081	3.68 (E)	5570	0.008 (P)	12
Hexachlorobutadiene	3.74	FW SW	0.06 (P) 0.06 (P)	0.3 0.3	10.0 3.0	55.0 16.4	1.0 0.3	5.5 1.6	0.7 (P) 0.7 (P)	4 4

[5]EPA proposed sediment quality criteria for the protection of benthic organisms.

			Levels of Protection							
			Human Health Bioaccumulation		Benthic Aquatic Life Acute Toxicity		Benthic Aquatic Life Chronic Toxicity		Wildlife Bioaccumulation	
Contaminant	**LogK_{ow}**	**Fresh-FW Salt -SW Both -FS**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**
Hexachlorocyclohexanes	3.8	FW SW	0.009 (P) 0.009 (P)	0.06 0.06	2.0 0.16	12.6 1.0	0.01 0.004	0.06 0.03	0.23 (P) 0.23 (P)	1.5 1.5
Hexachlorocyclopentadiene	3.99	FW SW			4.5 0.7	44.0 6.8	0.45 0.07	4.4 0.7		
Isodecyldiphenyl phosphate	5.4	FW			22	5526	1.7	427		
Linear Alkyl Benzene Sulfonates	3.97	FW					40	373		
Malathion	2.2	FS					0.1	0.02		
Methoxychlor	4.3	FS					0.03	0.6		
Mirex	5.83	FS	0.0001 (P)	0.07			0.001	0.7	0.0055 (P)	3.7
Octachlorostyrene	≈6.0	FS							0.0005 (P)	0.5
Parathion and Methyl Parathion	2.5	FW			0.065 (E)	0.02	0.008	0.003		
Pentachlorophenol	5.0	FW			1.0	100	0.4	40		
Phenanthrene	4.45	FW SW						120 (E)[6] 160 (E)[1]		
Phenols, total chlorinated	2.75	FW					1.0	0.6		

[6]EPA proposed sediment quality criteria for the protection of benthic organisms.

			Levels of Protection							
			Human Health Bioaccumulation		Benthic Aquatic Life Acute Toxicity		Benthic Aquatic Life Chronic Toxicity		Wildlife Bioaccumulation	
Contaminant	**LogK_{ow}**	**Fresh-FW Salt -SW Both -FS**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**
Phenols, total unchlorinated	2.0	FW					5.0	0.5		
PCB	6.14	FW SW	0.0000006 0.0000006	0.0008 0.0008	2.0 (E) 10.0 (E)	2760.8 13803.8	0.014 (E) 0.03 (E)	19.3 41.4	0.001 0.001	1.4 1.4
2,3,7,8-TCDD	7.0	FS	0.000001	0.01					$2x10^{-8}$ (P)	0.0002
1,1,2,2-Tetrachloroethane	2.56	FS	0.7 (P)	0.3						
Tetrachloroethylene	2.88	FS	1.0	0.8						
o-Toluidine	1.4	FS	18.0 (P)	0.5						
Toxaphene	3.3	FW SW	0.009 (P) 0.009 (P)	0.02 0.02	1.6 0.07	3.2 0.14	0.005 0.005	0.01 0.01		
Trichlorobenzenes	4.26	FS			50	910	5	91		
1,1,2-Trichloroethane	2.17	FS	4.0 (P)	0.6						
Trichloroethylene	2.29	FS	11.0	2.0						
Triphenyl phosphate	4.59	FW			40	1556	4	156		
Vinyl Chloride	0.6	FS	18.0 (P)	0.07						

			Levels of Protection							
			Human Health Bioaccumulation		Benthic Aquatic Life Acute Toxicity		Benthic Aquatic Life Chronic Toxicity		Wildlife Bioaccumulation	
Contaminant	**LogK_{ow}**	**Fresh-FW Salt -SW Both -FS**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**	**Water Qual Criteria µg/l**	**Sediment Criteria µg/gOC**
Anthracene	4.45	FW			35	986	3.8	107		
Benz(a)anthracene	5.61	FW			0.23	94	0.03	12		
Benzene	2.13	FW SW			760 670	103 90	210 190	28 26		
Ethylbenzene	3.15	FW SW			150 41	212 58	17 4.5	24 6.4		
Fluorene	4.18	FW SW			4.8 23	73 348	0.54 2.5	8 38		
Isopropylbenzene (cumeme)	3.66	FW			23	105	2.6	12		
2-methylnaphthalene	3.86	FW SW			42 48	304 348	4.7 4.2	34 30		
Naphthalene	3.37	FW SW			110 140	258 328	13 16	30 38		
Pyrene	5.32	FW			42	8775	4.6	961		
Toluene	2.69	FW SW			480 430	235 211	100 92	49 45		
1,2,4-trimethylbenzene	3.75	FW SW			290 170	1631 956	33 19	186 107		
Xylene	3.15	FW SW			590 170	833 240	65 19	92 27		

Table 2. Sediment Criteria for Metals. Two levels of risk have been established for metals contamination in sediments. These are the Lowest Effect Level and the Severe Effect Level. The Lowest Effect Level for each metal is the lowest of either the Persaud et al. (1992) Lowest Effect Level or the Long and Morgan (1990) Effect Range-Low. Similarly, the Severe Effect Level for each metal is the lowest of either the Persaud et al. (1992) Severe Effect Level or the Long and Morgan (1990) Effect Range-Moderate. A sediment is considered contaminated if either criterion is exceeded. If both criteria are exceeded, the sediment is considered to be severely impacted. If only the Lowest Effect Level criterion is exceeded, the impact is considered moderate. The units are µg/g, or ppm, except for iron, which is listed as a percentage. An "L" following a criterion means that it was taken from Long and Morgan (1990); a "P" following a criterion indicates that it is from Persaud et al. (1992). Complete tables from both sources can be found in appendix 2.

Metal	Lowest Effect Level µg/g (ppm)	Severe Effect Level µg/g (ppm)
Antimony	2.0 (L)	25.0 (L)
Arsenic	6.0 (P)	33.0 (P)
Cadmium	0.6 (P)	9.0 (L)
Chromium	26.0 (P)	110.0 (P)
Copper	16.0 (P)	110.0 (P)
Iron (%)	2.0% (P)	4.0% (P)
Lead	31.0 (P)	110.0 (L)
Manganese	460.0 (P)	1100.0 (L)
Mercury	0.15 (L)	1.3 (L)
Nickel	16.0 (P)	50.0 (L)
Silver	1.0 (L)	2.2 (L)
Zinc	120.0 (P/L)	270.0 (L)

Appendix 1. Basis for the Water Quality Criteria Used for Deriving Sediment Criteria for the Protection of Human and Health and Piscivorous Wildlife from Bioaccumulation Effects.

This appendix provides the basis and calculations for ambient water quality criteria in Table 1 with the suffix (P), which were developed by the Divisions of Fish and Wildlife and Marine Resources for use in calculation of sediment criteria.

Human health (bioaccumulation) based criteria in Table 1 with the (P) suffix are derived according to the method in 6NYCRR 702.8.

$$\text{Water Quality Criterion, ug/l} = \frac{\text{ADI, ug/d}}{0.033 \text{ kg/d} \times \text{BF}}$$

where

ADI, ug/d = acceptable daily intake for humans taken from fact sheets supporting drinking water standards and guidance values in TOGS 1. 1. 1

0.033 kg/d = the human daily intake from fish consumption cited in Part 702.8, and

BF = bioaccumulation factor

Wildlife residue based criteria in Table 1 with the (P) suffix are derived according to the method in 6NYCRR 702.13.

$$\text{Water Quality Criterion, ug/l} = \frac{\text{A, mg/kg}}{\text{BF}}$$

where

A = a fish flesh criterion for protection of piscivorous wildlife taken from Newell et a[(1987), and

BF = Bioaccumulation Factor

Bfs for human health based criteria are about 3% lipid based, whereas the BCF's for wildlife based criteria are about 10% lipid based. BFs were determined as a best judgement from review of available information in EPA water quality criteria documents, EPA (1 979), and other scientific literature.

Aldrin and Dieldrin

Wildlife Residue Based Criterion

$$0.0077 \text{ mg/l} = \frac{0.12 \text{ mg/kg}}{15570}$$

Azobenzene

Human Health Residue Based Criterion

$$0.16 \text{ ug/l} = \frac{1 \text{ ug/d}}{0.033 \text{ kg/d} \times 179}$$

Bis (2-chloro-ethyl) ether

Human Health Residue Based Criterion

$$0.5 \text{ ug/l} = \frac{0.06 \text{ ug/d}}{0.033 \text{ kg/d} \times 4}$$

Carbon tetrachloride

Human Health Residue Based Criterion

$$1.3 \text{ ug/l} = \frac{0.8 \text{ ug/d}}{0.033 \text{ kg/d} \times 19}$$

Chlordane

Wildlife Residue Based Criterion

$$0.01 \text{ ug/l} = \frac{0.5 \text{ mg/kg}}{47020}$$

Chloro-o-toluidine

Human Health Residue Based Criterion

$$6.5 \text{ ug/l} = \frac{1.4 \text{ ug/d}}{0.033 \text{ kg/d} \times 15}$$

DDT, DDD & DDE

Human Health Residue Based Criterion

$$0.00001 \text{ ug/l} = \frac{0.02 \text{ ug/d}}{0.033 \text{ kg/d} \times 53610}$$

1.2-Dichloroethane

Human Health Residue Based Criterion

$$24 \text{ ug/l} = \frac{1.6 \text{ ug/d}}{0.033 \text{ kg/d} \times 2}$$

1,1-Dichlorethylene

Human Health Residue Based Criterion

$$0.8 \text{ ug/l} = \frac{0.14 \text{ ug/d}}{0.033 \text{ kg/d} \times 2}$$

Endrin

Wildlife Residue Based Criterion

$$0.0019 \text{ ug/l} = \frac{0.025 \text{ mg/kg}}{13240}$$

Heptachlor & Heptachlor Epoxide

Human Health Residue Based Criterion

$$0.00003 \text{ ug/l} = \frac{0.018 \text{ ug/d}}{0.33 \text{ kg/d} \times 15666}$$

Hexachlorobenzene

Human Health Residue Based Criterion

$$0.0001 \text{ ug/l} = \frac{0.04 \text{ ug/d}}{0.033 \text{ kg/d} \times 12000}$$

Wildlife Residue Based Criterion

$$0.008 \text{ ug/l} = \frac{0.33 \text{ mg/kg}}{40000}$$

Hexachlorobutadiene

Human Health Residue Based Criterion

$$0.06 \text{ ug/l} = \frac{1 \text{ ug/d}}{0.033 \text{ kg/d} \times 545}$$

Wildlife Residue Based Criterion

$$0.7 \text{ ug/l} = \frac{1.3 \text{ ma/kg}}{1818}$$

Hexachlorocyclohexanes

Human Health Residue Based Criterion

$$0.009 \text{ ug/l} = \frac{0.04 \text{ ug/d}}{0.033 \text{ kg/d} \times 130}$$

Wildlife Residue Based Criterion

$$0.23 \text{ ug/l} = \frac{0.1 \text{ mg/kg}}{433}$$

Mirex

Human Health Residue Based Criterion

$$0.0001 \text{ ug/l} = \frac{0.08 \text{ ug/d}}{0.033 \text{ kg/d} \times 18100}$$

Wildlife Residue Based Criterion

$$0.0055 \text{ ug/l} = \frac{0.33 \text{ mg/kg}}{60333}$$

Octachlorostyrene

Wildlife Residue Based Criterion

$$0.0005 \text{ ug/l} = \frac{0.02 \text{ mg/kg}}{40000}$$

2,3,7,8-Tetrachlorodibenzodioxin

Wildlife Residue Based Criterion

$$2 \times 10\text{-}8 \text{ ug/l} = \frac{0.000003 \text{ mg/kg}}{150{,}000}$$

1,1,2,2-Tetrachloroethane

Human Health Residue Based Criterion

$$0.7 \text{ ug/l} = \frac{0.4 \text{ ug/d}}{0.033 \text{ kg/d} \times 17}$$

0-Toluidine

Human Health Residue Based Criterion

$$18 \text{ ug/l} = \frac{1.2 \text{ ug/d}}{0.033 \text{ kg/d} \times 2}$$

Toxaphene

Human Health Residue Based Criterion

$$0.009 \text{ ug/l} = \frac{0.02 \text{ ug/d}}{0.033 \text{ kg/d} \times 67}$$

1,1,2-Trichloroethane

Human Health Residue Based Criterion

$$4 \text{ ug/l} = \frac{1.2 \text{ ug/d}}{0.033 \text{ kg/d} \times 9}$$

Vinyl Chloride

Human Health Residue Based Criterion

$$18 \text{ ug/l} = \frac{0.6 \text{ ug/d}}{0.033 \text{ kg/d} \times 1}$$

Appendix 2. The following tables are photocopied directly from Long and Morgan (1990) and Persaud et. al. (1992). They are presented here to provide further information about the metals criteria developed in Table 2., and the text above.

Copied directly from Persuad et. al. (1992)

Table 1: Provincial Sediment Quality Guidelines for Metals and Nutrients.
(values[a] in ug/g (ppm) dry weight unless otherwise noted)

METALS	No Effect Level	Lowest Effect Level	Severe Effect Level
Arsenic	-	6	33
Cadmium	-	0.6	10
Chromium	-	26	110
Copper	-	16	110
Iron (%)	-	2	4
Lead	-	31	250
Manganese	-	460	1100
Mercury	-	0.2	2
Nickel	-	16	75
Zinc	-	120	820
NUTRIENTS			
TOC (%)	-	1	10
TKN	-	550	4800
TP	-	600	2000

[a] - values less than 10 have been rounded to 1 significant digit. Values greater than 10 have been rounded to two significant digits except for round numbers which remain unchanged (e.g., 400).

"-" - denotes insufficient data/no suitable method.

TOC - Total Organic Carbon TKN - Total Kjeldahl Nitrogen TP - Total Phosphorus

(June 1992)

Copied Directly from Long and Morgan (1990)

Table 70. Summary of ER-L, ER-M, and overall apparent effects thresholds concentrations for selected chemicals in sediment (dry weight).

Chemical Analyte	ER-L Concentration	ER-M Concentration	ER-L:ER-M Ratio	Overall Apparent Effects Threshold	Subjective Degree of Confidence in ER-L/ER-M Values
Trace Elements (ppm)					
Antimony	2	25	12.5	25	Moderate/moderate
Arsenic	33	85	2.6	50	Low/moderate
Cadmium	5	9	1.8	5	High/high
Chromium	80	145	1.8	No	Moderate/moderate
Copper	70	390	5.6	300	High/high
Lead	35	110	3.1	300	Moderate/high
Mercury	0.15	1.3	8.7	1	Moderate/high
Nickel	30	50	1.7	NSD*	Moderate/moderate
Silver	1	2.2	2.2	1.7	Moderate/moderate
Tin	NA	NA	NA	NA	NA
Zinc	120	270	2.2	260	High/high
Polychlorinated Biphenyls (ppb)					
Total PCBs	50	400	7.6	370	Moderate/moderate
DDT and Metabolites (ppb)					
DDT	1	7	7	6	Low/low
DDD	2	20	10	NSD	Moderate/low
DDE	2	15	7.5	NSD	Low/low
Total DDT	3	350	117	No	Moderate/moderate
Other Pesticides (ppb)					
Lindane	NA	NA	NA	NSD	NA**
Chlordane	0.5	6	12	2	Low/low
Heptachlor	NA	NA	NA	NSD	NA
Dieldrin	0.02	8	400	No	Low/low
Aldrin	NA	NA	NA	NSD	NA
Endrin	0.02	45	2250	NSD	Low/low
Mirex	NA	NA	NA	NSD	NA
Polynuclear Aromatic Hydrocarbons (ppb)					
Acenaphthene	150	650	4.3	150	Low/low
Anthracene	85	960	11.3	300	Low/moderate
Benzo(a)anthracene	230	1600	7	550	Low/moderate
Benzo(a)pyrene	400	2500	6.2	700	Moderate/moderate
Benzo(e)pyrene	NA	NA	NA	NSD	NA
Biphenyl	NA	NA	NA	NSD	NA
Chrysene	400	2800	7	900	Moderate/moderate
Dibenz(a,h)anthracene	60	260	4.3	100	Moderate/moderate
2,6-dimethylnaphthylene	NA	NA	NA	NSD	NA
Fluoranthene	600	3600	6	1000	High/high
Fluorene	35	640	18.3	350	Low/low
1-methylnaphthalene	NA	NA	NA	NSD	NA
2-methylnaphthalene	65	670	10.3	300	Low/moderate
1-methylphenanthrene	NA	NA	NA	NSD	NA
Naphthalene	340	2100	6.2	500	Moderate/high
Perylene	NA	NA	NA	NSD	NA
Phenanthrene	225	1380	6.1	260	Moderate/moderate
Pyrene	350	2200	6.3	1000	Moderate/moderate
2,3,5-trimethylnaphthalene	NA	NA	NA	NSD	NA
Total PAH	4000	35000	8.8	22000	Low/low

* NSD = not sufficient data
** NA = not available

Appendix 3. Literature Cited

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Appendix 4. Change in the Guidance for Marine and Estuarine Sediments

The 22 November 1993, Technical Guidance for Screening Contaminated Sediments (reprinted July 1994) makes use of the sediment guidance values from a number of sources, including the ER-L and ER-M guidance values from Long and Morgan (1990). Long, MacDonald, Smith, and Calder (1995) further refined and enhanced the marine and estuarine data used by Long and Morgan (1990) and published new ERL and ERM specifically for marine and estuarine sediments. **For evaluation of risk from contaminants in marine and estuarine sediment,** the Division of Fish, Wildlife and Marine Resources will now use the Long et al (1995) guidance values rather than the Long and Morgan (1990) values. For non-polar organic compounds not listed in Long et al (1995) (Table 4, below), the equilibrium partitioning-derived values in Table 1. (pp 20-24 above) for saltwater should be used. The following Tables 3 and 4 are reproduced directly from:

Long, E.R., MacDonald, D.D., Smith, S.L., and F.D. Calder, 1995. "Incidence of Adverse Biological Effects Within Ranges of Chemical Concentrations in Marine and Estuarine Sediments". Environmental Management 19(1):81-97.

Table 3. ERL and ERM guideline values for trace metals (ppm, dry wt.) and percent incidence of biological effects in concentration ranges defined by the two values.

	Guidelines		Percent (ratios) incidence of effects[a]		
Chemical	ERL	ERM	<ERL	ERL-ERM	>ERM
Arsenic	8.2	70	5.0 (2/40)	11.1 (8/73)	63.0 (17/27)
Cadmium	1.2	9.6	6.6 (7/106)	36.6 (32/87)	65.7 (44/67)
Chromium	81	370	2.9 (3/102)	21.1 (15/71)	95.0 (19/20)
Copper	34	270	9.4 (6/64)	29.1 (32/110)	83.7 (36/43)
Lead	46.7	218	8.0 (7/87)	35.8 (29/81)	90.2 (37/41)
Mercury	0.15	0.71	8.3 (4/48)	23.5 (16/68)	42.3 (22/52)
Nickel	20.9	51.6	1.9 (1/54)	16.7 (8/48)	16.9 (10/59)
Silver	1.0	3.7	2.6 (1/39)	32.3 (11/34)	92.8 (13/14)
Zinc	150	410	6.1 (6/99)	47.0 (31/66)	69.8 (37/53)

[a]Number of data entries within each concentration range in which biological effects were observed divided by the total number of entries within each range.

Table 4. ERL and ERM guideline values for organic compounds (ppb, dry wt) and percent incidence of biological effects in concentration ranges defined by the two values.

	Guidelines		Percent (ratios) incidence of effects[a]		
Chemical	ERL	ERM	<ERL	ERL-ERM	>ERM
Acenaphthene	16	500	20.0 (3/15)	32.4 (11/34)	84.2 (16/19)
Acenaphthylene	44	640	14.3 (1/7)	17.9 (5/28)	100 (9/9)
Anthracene	85.3	1100	25.0 (4/16)	44.2 (19/43)	85.2 (23/27)
Fluorene	19	540	27.3 (3/11)	36.5 (19/52)	86.7 (26/30)
2-Methyl naphthalene	70	670	12.5 (2/16)	73.3 (11/15)	100 (15/15)
Naphthalene	160	2100	16.0 (4/25)	41.0 (16/39)	88.9 (24/27)
Phenanthrene	240	1500	18.5 (5/27)	46.2 (18/39)	90.3 (28/31)
Low-molecular weight PAH	552	3160	13.0 (3/23)	48.1 (13/27)	100 (16/16)
Benz(a)anthracene	261	1600	21.1 (4/19)	43.8 (14/32)	92.5 (25/27)
Benzo(a)pyrene	430	1600	10.3 (3/29)	63.0 (17/27)	80.0 (24/30)
Chrysene	384	2800	19.0 (4/21)	45.0 (18/40)	88.5 (23/26)
Dibenzo(a,h)anthracene	63.4	260	11.5 (3/26)	54.5 (12/22)	66.7 (16/24)
Fluoranthene	600	5100	20.6 (7/34)	63.6 (28/44)	92.3 (36/39)
Pyrene	665	2600	17.2 (5/29)	53.1 (17/32)	87.5 (28/32)
High molecular weight PAH	1700	9600	10.5 (2/19)	40.0 (10/25)	81.2 (13/16)
Total PAH	4022	44792	14.3 (3/21)	36.1 (13/36)	85.0 (17/20)
p,p'-DDE	2.2	27	5.0 (1/20)	50.0 (10/20)	50.0 (12/24)
Total DDT	1.58	46.1	20.0 (2/10)	75.0 (12/16)	53.6 (15/28)
Total PCBs	22.7	180	18.5 (5/27)	40.8 (20/49)	51.0 (25/49)

[a]Number of data entries within each concentration range in which biological effects were observed divided by the total number of entries within each range.

Attachment B

Field Standard Operating Procedures

STANDARD OPERATING PROCEDURE

FD-001 Field Notebook and Boring Log Forms

1. Objective

Proper documentation of all site activities is a crucial part of the field investigation process. Documentation, relative to sampling procedures, includes sample labels, sample seals, field logbooks, boring log forms, chain of custody records, sample analysis request forms, and laboratory sample logs. The field notebook serves as a record of significant field activities performed or observed during the project. The field notebook provides a factual basis for preparing field observation reports, if required, and reports to clients and regulatory agencies. Example field notes are provided in Appendix A. The field notebook can be used to record all soil boring information or, if desired, a separate boring log form can be used to document all soil boring information. The boring log form provides a factual basis for generating graphical boring logs for inclusion in reports to clients and regulatory agencies. An example boring log form is provided in Appendix A.

2. Execution

- Use a separate all-weather bound notebook for each site/location/project number. If separate boring log forms are used for soil borings, use a new form for each boring.
- Write neatly using black or blue waterproof pen (or note if field conditions [i.e., cold or wet weather] require use of pencil).
- Write the project name, project number, book number (i.e., 1 of 3), and date on the front cover. On the inside cover, identify the project name, project number, and “Return Book To:” the office address of the project manager.
- Number all of the pages of the field book starting with the first entry.
- Record activities as they occur. If separate boring log forms are being used, the field notebook should refer to the particular form.
- Neatly cross out mistakes using a single line and initial them. Erasures are not permitted. If an error is made on a document assigned to one individual, that individual will make all corrections. The person who made the entry will correct any subsequent error discovered on an accountable document. All subsequent corrections will be initialed and dated.
- Sign or initial and date the bottom of every page with an entry. Place a diagonal line through unused portions of a page.
- Record the following information upon each arrival at the site:
    1. Date/time/weather/project number
    2. GEI personnel
    3. Purpose of visit/daily objectives

4. Record conversations with: [Recommendation - If possible, record telephone numbers of individual contacts for the site in the field notebook.]
5. Contractors
6. Clients
7. Visitors (include complete names, titles, and affiliations whenever possible).
8. GEI office staff
9. Landowners (site or abutters)
10. Note time of arrival and departure of individuals visiting the site

- Additional observations to record:
  1. Type and quantity of monitoring well construction materials used
  2. Use of field data sheets or electronic logging equipment (e.g. boring logs, monitoring well sampling logs, etc.)
  3. Ambient air monitoring data
  4. Locations and descriptions of sampling points
  5. Sample media (soil, sediment, groundwater, etc.)
  6. Sample collection method
  7. Number and volume of sample(s) collected and sample bottle preservatives used
  8. Sample identification number (s) and date and time of sample collection
  9. Approximate volume of groundwater removed before sampling
  10. Field observations
  11. Any field observations made such as pH, temperature, turbidity, conductivity, water level, etc.
  12. References for all maps and photographs of the sampling site(s)
  13. Information pertaining to sample documentation: bottle lot numbers/ dates, method of sample shipments, chain-of custody record numbers, and overnight shipping numbers.
  14. Surveying data (including sketches with north arrows)
  15. Changes in weather
  16. Rationale for critical field decisions
  17. Recommendations made to the client representative and GEI Project Manager
  18. Include a site sketch or representative site photograph of conditions at the end of the day, if required
  19. Time
  20. Summarize work completed/work remaining

- Place a diagonal line though and sign portions of pages not used or skipped
- Bottom of each page signed and dated

3. Limitations

- Only record facts.
- Allow time at the end of the day to write your journal, and make it a priority, even at the expense of observing time.
- Record all observations regardless of relevancy.
- Identify conditions or events that could affect/impede your ability to observe conditions.
- Do not use spiral notebooks because pages can be easily removed.

4. Field Book and Boring Log Form Maintenance

Field notebooks and boring log forms are the primary sources of factual information that is the basis for technical reports. As such, their physical condition must be protected and maintained.

- When possible, digital scans of all field documents should be made at the end of each day and saved to the GEI network server in the appropriate project files.
- If digital scans cannot be generated and saved, hard copies should be made and saved separately from the field book and boring log forms.
- Regardless, when a field project is completed and field staff have returned to the office, a complete digital scan of all documents will be generated and saved to the network server in the appropriate project files.
- All original hard copies will be stored in file cabinets in the office, along with other relevant project files and managed per GEI's document retention policies.

5. References

New Jersey DEP Field Sampling Procedures Manual, August 2005.

Yerington Mine Site SOP-03 Standard Operating Procedure Field Notes and Documentation, Revision 0 Revision Date: June 6, 2006.

6. Attachments

Attachment A - Example Field Notes

7. Contact

Jerry Zak

STANDARD OPERATING PROCEDURE

QA-001 Equipment Decontamination

1. Objective

This SOP describes methods used for preventing or reducing cross-contamination, and provides general guidelines for sampling equipment decontamination procedures. Preventing or minimizing cross contamination in sampled media and in samples is important for preventing the introduction of error into sampling results and for protecting the health and safety of site personnel. Removing or neutralizing contaminants that have accumulated on sampling equipment ensures protection of personnel from permeating substances, reduces or eliminates transfer of contaminants to clean areas, prevents the mixing of incompatible substances, and minimizes the likelihood of sample cross-contamination.

2. Execution

- Inspect equipment for cleanliness prior to moving onto a site and prior to relocating to each new sampling location. All contractor-provided equipment (augers, rods, spoons, backhoe buckets) shall be decontaminated by steam cleaning **prior to coming on site.**
- Equipment decontamination is a sequential procedure consisting of the following general steps: Alconox-solution wash (or equivalent non-phosphate detergent); potable water rinse; methanol wash, and three distilled-water rinses.
- Alconox solution is a mixture of approximately 1 cup of Alconox per 1 gallon of potable water. Alconox solution wash requires scrubbing the equipment with a brush soaked in Alconox solution and removing any visible contamination or dirt from the equipment.
- Before advancing each boring, drilling equipment (including augers, casing, rods, and washtub) must be decontaminated by steam cleaning.
- Split-spoon samplers must be decontaminated prior to collecting each sample. The split-spoon decontamination procedure includes: a gross wash and scrub in a bucket of Alconox solution; potable water rinse; methanol wash, and three distilled-water rinses.
- Pumps and tubing used for sample collection and well development must be decontaminated by flushing with a minimum of one gallon of potable water; then flushing with a minimum of one pint of methanol and rinsing twice with distilled water.
- For pumps and tubing, perform a final rinse of the sampling equipment with the water being sampled.

3. Limitations

- Do not store the deionized/distilled water in polyethylene bottles, use Nalgene, glass, or Teflon. Polyethylene may leach phthalates.
- Do not attempt to decontaminate string or rope - replace it.
- Due to eye and skin absorption hazards, safety glasses and gloves must be worn when handling decontamination solvents.
- The decontamination procedure may require modification based on site specific conditions and methods used should not interfere with the site-specific chemical analyses. The procedure may also require modification based on state regulations.
- Steam cleaning with potable water is an acceptable decontamination method for drilling equipment (i.e., augers).
- If sampling for metals, the decontamination procedure requires modification to include rinsing with a 1:1 nitric acid and rinsing with deionized water in place of distilled water.
- Dedicated equipment need not be decontaminated beyond initial decontamination prior to field use.

4. References

Environmental Response Team (ERT), US EPA. Sampling Equipment Decontamination, SOP No. 2006, Revision 0.0. August 11, 1994.

US EPA Region 9. Sampling Equipment Decontamination, SOP No. 1230, Revision 1.September 1999.

5. Attachments

None

6. Contact

Brian Conte

SUMMARY GUIDANCE

SS-001 Water Safety

1. Objective

The safe deployment and return of personnel during field activities while aboard a boat.

2. Execution

Boat safety practices will be conducted in general accordance with guidance provided in the United States Army Corps of Engineers (USACE) Safety and Health Requirements Manual (EM) 385-1-1. Personnel will board the barge at specified locations to be determined and agreed upon prior to field deployment. The following safety practices shall be adhered to:

- Every employee shall wear a Personal Flotation Device (PFD) at all times when underway aboard any boat less than 25 feet except when that boat is equipped with a fully enclosed cabin and the employee is inside. Boats under 25 feet must also, at a minimum, have Coast Guard approved PFDs on board for each person and at least one throwable flotation device, such as a seat cushion.
- For every boating activity, a trip plan must be communicated to someone in a position to know when you are overdue and take appropriate action.
- For every trip requiring more than one day, daily voice-radio communications with an appropriate base must be maintained.
- The consumption of alcoholic beverages and the use of illegal drugs shall not be permitted at any time aboard boats owned and/or operated by the department.
- Firearms shall not be kept in a loaded condition aboard boats owned and/or operated by the department except when carried by law enforcement personnel or when actively being fired for work related purposes.
- Contractors working in an exposed marine location shall monitor the National Oceanic and Atmospheric Administration (NOAA) marine weather broadcasts and shall use other local commercial weather forecasting services as may be available.
- Type III Personal Floatation Devices (PFDs) will be worn by boat/barge occupants at all times when working over water.
- For retrieving a person overboard, the boat operator will throw a life ring and line, and use a ladder attached to the barge or the support

boat step transom to allow the person to climb out of the water. For retrieving a person overboard, the support boat will also be equipped with a life ring attached to approximately 90 feet of rope. The barge and the support boat will be equipped with an ABC rated fire extinguisher(s).

- Emergency procedures for fire and man overboard will be reviewed on the first day of operations and any time a change of personnel occurs.

3. Limitations

None

4. References

United States Army Corps of Engineers, Safety and Health Requirements Manual (EM), 385-1-1.November 3, 2003 – Section 19 Floating Plant and Marine Activities

5. Attachments

None

6. Contact

Robin DeHate, GEI Corporate Health & Safety Officer

STANDARD OPERATING PROCEDURE

SS-002 Ponar or Shipek Grab Sampler

1. Objective

Surficial sediment samples will be collected from the upper 6 inches (approximate) using a Ponar or Shipek type grab sampler. Both of these sampling devices have the advantages of being relatively easy to handle and operate, readily available, moderately priced, and versatile in terms of the range of substrate types they can effectively sample. In addition, both of these grab samplers provide sufficient sample volume (8.2 or 2.4 cubic liters, respectively) to allow sub sampling for multiple analytes.

2. Materials

Equipment needed for collection of sediment samples may include (depending on technique chosen):

- Ponar/Shipek Sampler
- Stainless steel sampling tools
- Laboratory provided sample bottles
- Resealable plastic bags
- Ice
- Coolers, packing material
- Chain of custody records, custody seals
- Decontamination equipment/supplies
- Maps/plot plan
- Safety equipment
- Tape measure
- Camera
- Field data sheets/field notebook/waterproof pen
- Permanent markers
- Sample bottle labels
- Paper towels
- Personal Protection Equipment (PPE)
- Global Positioning System (GPS)

3. Execution

- Prior to sample collection, the grab sampler will be decontaminated.
- When deploying the grab sampler, the speed of descent should be controlled, with no “free fall” allowed. In deep waters, use of a winching system is recommended to control both the rate of descent and ascent.
- The sampler will be carefully lowered the last few feet to minimize dispersal of fine material due to a sampler-induced shock wave.

- At the time of the sample collection, the sample location will be surveyed with GPS survey equipment.
- After the sample is collected, the sampling device should be lifted slowly off the bottom and raised to the surface at a slow and steady rate.
- Sediments in direct contact with sides or teeth of the grab sampler will be excluded from samples to prevent potential contamination from the grab sampling device when possible.
- Prior to sampling directly from the grab sampler, the overlying water will be removed by opening the jaws of the Ponar slightly and allowing the water to drain. If the overlying water is turbid, then the suspended solids will be allowed to settle, if possible, prior to draining.
- Where sampling directly from the sampler is not possible or feasible, the sampler will be slowly opened over a sample platform. The sampler will be placed such that the sample may be deposited with minimal disturbance.
- Photograph the sample in color with a camera. Place a small label with sample field ID number and approximate depth so that it appears in each frame. SOP FD-004 Photodocumentation provides further guidance on photodocumentation.
- Sediments will be described in accordance with the soil description procedure listed below in SOP SM-003 Soil Classification.
- Place sediment samples into pre-cleaned laboratory provided jars for the appropriate analyses as determined in the work plan. Label each jar with the unique grab sample identification number and depth of the sample.
- Place the sample containers into plastic sealable bags or bubble wrap and place them in an iced cooler until transfer shipment to the analytical laboratories. Add the sample to the chain of custody form.

4. Limitations

Careful use of grab samplers is required to avoid problems such as loss of fine-grained surface sediments from the bow wave during descent, mixing of sediment layers upon impact, lack of sediment penetration, and loss of sediment from tilting or washout upon ascent.

There are two primary interferences or potential problems associated with sediment sampling. These include cross contamination of samples and improper sample collection.

- Cross contamination problems can be eliminated or minimized through the use of dedicated or disposable sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary.

- Improper sample collection can involve using contaminated equipment, equipment that is potentially not compatible with the contaminants of concern, disturbance of the stream or impoundment substrate, and sampling in an obviously disturbed or non-representative area. Be sure to use sampling equipment of an appropriate composition based upon the suspected contaminants and analyses to be performed.

Following proper decontamination procedures, minimizing disturbance of the sample site, and careful selection of sampling locations will eliminate these problems.

If the above sampling protocols are followed, it will minimize the effects of typical disadvantages to Ponar or Shipek samplers such as possible shock wave and loss of very fine grained surface deposits, potential for water column contamination, and nearby down current sediment re-deposition. The potential does exist for larger materials such as twigs and stones to prevent jaw closure that will result in collection of unacceptable sample. In areas with significant debris in sediment, collection of a representative sediment surface sample may not be possible due to method and equipment limitations.

5. References

U.S. Environmental Protection Agency, Office of Water, Office of Science & Technology. 2001. Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual. EPA-823B-01-002, October 2001.

State of Ohio, Environmental Protection Agency, Division of Surface Water. 2001. Sediment Sampling Guide and Methodologies, Second Edition. November 2001.

ASTM, 2003. D4823-95 (2003) Standard Guide for Core Sampling Submerged, Unconsolidated Sediments. ASTM International, West Conshohocken, PA. August 2003.

Newfield's Environmental Forensics Laboratory, 2005. Geochronologic Sample Handling Procedure.

6. Attachments

None

7. Contact

Kim Bradley

STANDARD OPERATING PROCEDURE

FD-003 Sample Handling and Chain of Custody

1. Objective

To properly collect, label, document, preserve, package, transport environmental samples, and to provide a record of the custody of any environmental field sample from time of collection to delivery to the laboratory. The Chain-of-Custody (COC) can be used as a legal document to guarantee that samples were not mishandled and that they were delivered to the laboratory within the timeframe necessary to start analysis. A sample is under custody if:

a) it is in GEI's possession; or
b) it is in GEI's view after being in GEI's possession; or
c) it was in GEI's possession and then it was locked up to prevent tampering; or
d) it is in a designated secure area. GEI facilities are designated secure areas.

2. Execution

- Review the work plan prior to sampling to determine the following:
  i. The analysis required by the period and sample volumes required by the laboratory to perform those analysis. (Be explicit when requesting analysis on the COC (e.g. rather than "VOCs" (Volatile Organic Compounds) write "VOCs 8260".)
  ii. The turnaround time required by the project.
  iii. If the data will be sent directly from the laboratory to the data validator or Data Group.
  iv. Holding time restrictions for sampling media and analytical methods.
- Label the jar or bottle not on the cap.
- Following sample collection, the sample container is labeled using a waterproof marker with the sample ID, the date and time (military time) of sample collection, project number, sample preservatives, and the sampler's initials. Sample custody begins at this time.
- Record the above information in the field notebook.
- Individually wrap sample jars with packing material. Place samples in a chilled (4°C) cooler immediately after collection.
- Complete a chain of custody (COC) for the samples as described below, and sign off on the COC each time a new person takes possession of the samples. A COC form must accompany each shipment/delivery of samples to the laboratory. GEI or Laboratory COC forms may be used as long as the laboratory form contains the same required information as described below.

- An example COC is provided in Attachment A.
- Place a custody seal on the cooler if shipping.
- Transport samples to the laboratory as soon as possible. It is preferable the samples are sent from the field rather than brought back to the office for submission at a later date.

2.1. Chain-of-Custody (COC) Completion

- Record the project name and number, the sampler's name(s) and the state where the samples were collected.
- For each sample, enter the sample identification number, date and time (military time) collected, whether the sample is a grab or composite sample and the number of sample containers. Record the type of analysis (including laboratory method; e.g. EPA-SW846 Method XX) requested and the preservative (if appropriate) in the vertical boxes.
- When samples are ready to be relinquished, complete the bottom of the form with date and time (military time) and signatures of relinquisher and receiver of samples as indicated. The sample collector is always the first signature while the analytical laboratory is the final signature. Theoretically, all individuals handling the samples between collection and laboratory should sign the form; however, if a common carrier (i.e., Federal Express, UPS) is used for shipping, GEI must identify the carrier in the 'Received by' box on the COC. If the sampler hand delivers the samples to the laboratory, the received box must be signed by the laboratory.
- The forms are in triplicate (white, yellow, and pink copies). The pink copy should be retained by the sampling personnel and provided to the Data Group for proper filing. The white and yellow copies should accompany the samples to the laboratory.
- Prior to sample shipment, the COC must be placed inside the cooler (in a ziplock bag or other watertight package taped inside the lid of the cooler), and the cooler must be sealed with a signed COC seal.
- If a common carrier such as FedEx is used to transport the samples to the laboratory, include the carrier tracking number and identify the carrier in the "Received by" box on the COC.
- Any unused sampling containers/media that is sent back to the lab should be included on the COC. Return samples to the laboratory in a timely manner.
- Field duplicates should be anonymous to the laboratory, but must be recorded for use by the Data Group. To keep track of this information, link the field duplicate with the proper sample in the field copy of the COC and also the field book.

- After the samples are sent to the laboratory, the field copy must be sent to the Data Group. You can send the field copy with duplicate information in the mail to the Data Group.

3. Limitations

- The field notebook must document all GEI personnel who had custody of any samples prior to shipping the samples to the laboratory, the samples must be relinquished to the shipper and the COC signed and dated by the sampler and the shipper, even if both people are GEI personnel.
- Keep the number of people involved in collecting and handling samples and data to a minimum.
- Only allow people associated with the project to handle samples and data.
- Always document the transfer of samples and data from one person to another on chain-of-custody forms.
- Always accompany samples and data with their chain-of-custody forms.
- Give samples and data positive identification at all times that is legible and written with permanent ink.
- When sending samples via a common carrier, use one COC per package.
- Do not send samples from more than one site with separate COCs in a single package.

4. References

New Jersey Department of Environmental Protection, Field Sampling Procedures Manual, August 2005.

Connecticut Department of Environmental Protection, Guidance for Collecting and Preserving Soil and Sediment Samples for Laboratory

Determination of Volatile Organic Compounds, Version 2.0 February 28, 2006.

5. Attachments

Attachment A – Example Chain of Custody

6. Contact

Brian Skelly

STANDARD OPERATING PROCEDURE

SC-002 Sample Handling

1. Objective

Sample handling involves the collection and shipping of environmental samples to a laboratory for chemical analysis. The overall objective of sample handling is to ensure that samples are properly:

- labeled and documented;
- preserved;
- packaged; and
- transported to laboratories.

2. Execution

- Prior to mobilizing to the field, select a shipper or arrange for a courier for sample delivery to the laboratory. If using a shipper (i.e., Federal Express, or UPS) determine the time constraints for pickup requests, the location and hours of the nearest shipping office, and any size/weight restrictions.
- Label all laboratory glassware with waterproof ink prior to collecting samples. The label should have an adhesive and be placed on the jar or bottle, not on the cap. In addition, clear packing tape should be placed over the sample label to secure it to the bottle as moisture from the samples can loosen the label adhesive.
- Record the following information on the label and in the field notebook (See Field Notebook SOP FD-001): project number, sample identification (i.e. MW-201 or SS-2), date, and time (military time) of collection, sampler's initials, and preservative, if present.
- If sample jars are not pre-preserved, add preservative as appropriate.
- At each sampling location, samples must be collected in order of volatility, most volatile first. Samples collected for volatile analysis must be placed in sample containers immediately upon retrieval of the sample.
- Aqueous samples for volatile analysis must be collected without air bubbles. Soil samples for volatile analysis should be compacted to eliminate as much headspace as possible. Other laboratory glassware should also be filled when possible. Care must be taken to avoid getting soils on the threads of sample jars, which can cause a faulty seal.
- If compositing of samples is performed in the field, specify basis for composite (i.e. volume, weight, spoon recovery, etc.) and record procedure for compositing sample in the field book.

- The sample cooler should have any water drains securely sealed with duct tape, both on the inside and outside of the cooler. A layer of packing material should be placed on the bottom of the cooler as a cushion. A large plastic bag will then be placed inside the cooler to contain all sample containers and ice for chilling samples. Refer to TestAmerica Packing Samples for Shipment Back to the Laboratory Attachment for reference.
- Once samples have been collected, place samples in a cooler with ice or a blue pack (if allowed) to chill samples to 4°C and start the chain-of-custody form (SOP FD-003 Sample Handling and *Chain of Custody*). Only use double bagged ice and not loose ice when packing cooler.
- For shipping, individually wrap each sample bottle with bubble packing or suitable packing material and place the wrapped bottles upright in the cooler with sufficient packing material between samples to avoid breakage.
- Place a layer of packing material above and below the sample bottles. Place blue ice packs or ice bags on top of the packing material. Fill the remaining space in the cooler with packing material to eliminate the possibility of vertical movement of samples.
- Seal the large plastic bag with the samples and ice inside.
- Place the completed and signed chain-of-custody form in a plastic ziplock-type bag and place on top of the packing material in the cooler, or taped to the inside lid of the cooler.
- Fill out the appropriate shipping or courier forms and attach to the top or handle of the cooler. If necessary, place the proper shipping labels on the cooler. Have the courier sign the COC form (or write pickup by FEDEX, UPS, etc. with date and time). Place a signed and dated custody seal on the cooler.
- All samples should be submitted as soon as possible. It is preferable for samples to be mailed prior to returning to the office.
- A copy of the waybills must be kept by the field supervisor to track shipments if necessary.

3. Limitations

- At all times, follow safety procedures as defined in the site-specific Health and Safety Plan.
- Field personnel must be aware of analyses which have short holding times and schedule sampling events and shipping accordingly. Shipment of samples for analyses with short holding times must be planned in advance. Refer to the project work plan, quality assurance project plan, or state/federal regulations for holding time and preservative information. Contact the laboratory ahead of time when

shipping samples with short holding time to ensure the lab is prepared for these analyses.

- In general, glassware for aqueous samples contains preservatives, (i.e. HNO3, HCl, etc). When collecting the sample, take care not to overfill the container, thus flushing the preservative out of the bottle.
- Never composite samples for VOCs in the field. Collect individual aliquots and direct the laboratory to perform compositing.
- Collection of aqueous samples should not be performed over the opening of a monitoring well. Preservatives from overfilling, a marker pen or other objects could fall into the well.
- If the recharge volume for a monitoring well is low, completely fill all volatile vials and then collect the minimum sample volume required for each remaining analysis.
- During subsurface soil sampling, if the recovery from the split-spoon sample is inadequate, if appropriate, resample the bottom of the borehole to obtain proper sample volume.
- Laboratories will homogenize and test the contents of the sample container, unless directed otherwise. Samples should not contain rocks, twigs, leaves, etc. unless these materials are of interest.

4. References

New Jersey Department of Environmental Protection, Field Sampling Procedures Manual, August 2005.

Connecticut Department of Environmental Protection, Guidance for Collecting and Preserving Soil and Sediment Samples for Laboratory

Determination of Volatile Organic Compounds, Version 2.0 February 28, 2006.

5. Attachments

Attachment 1 - General Guidelines for selecting equipment on the basis of construction material and target analyte(s)

Attachment 2 – TestAmerica Packing Samples for Shipment Back to the Laboratory

6. Contact

Jennifer Sandorf

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To ensure accuracy, prevent lost time, and avoid additional fees:

- Do not store or use this equipment in a wet environment.
- Do not store this equipment in extremely high or low temperature environments.
- Use care to ensure that water and debris does not contact internal parts, connections, or accessories.
- Place re-chargeable batteries (if provided) on charge for at least 4 hours prior to use.
- Do not mark equipment or accessories with permanent marker or duct tape
- All contaminants must be removed from equipment and accessories prior to return.

The following items are included for the rental term. If they are not returned at the end of the rental or sustain damage, the replacement cost will be applied to your invoice.

Handheld Display

Cable

Flow Cell

Stand

Storage Cup

Comm. Cable

Sensor Guard

Manual

YSI FIELD CALIBRATION GUIDE

- Turn 650 MDS on.
- Press enter on sonde menu. (Sonde will connect with display.)
- Scroll down sonde menu and highlight Report. Press enter and scroll down to DO CHG and Ph mV and press enter. When there is a solid dot next to the parameter that means it is turned on. Also make sure the following parameters are turned on: Temp C; SpCond µS/cm; Cond µS/cm; DOsat%; DO mg/L; pH; and Orp mV. Press esc.
- Select Calibrate, and press enter.
- It is our recommendation that our calibration procedures and our calibration solutions are used. Failure to use our procedures with our solutions may compromise the accuracy of your results.
- **Start your calibration with conductivity first.**
  - Using our conductivity calibration solution, 1000 µS/cm, completely submerge probes in solution. Highlight Conductivity and press enter.
  - Highlight SpCond and press enter.
  - Now at "enter value" screen, enter the value, (If using our solutions enter 1 mS/cm, and it will calibrate to 1000 µS/cm.) Press enter once value is entered.
  - Here is where you need to check your DO CHG. **It should read 50 with an accuracy range of +/- 25.**
  - Check your SpCond, and Cond readings. If they display a drastic spike every few seconds try re-skinning the DO probe again. You may also need to change your KCL Solution. (6 month shelf life mixed)
  - If SpCond reading seems too high pre-cal, (1200+), you might want to change your cal solutions.
  - When the SpCond and Cond readings have become stable, count to ten and press enter.
  - The values for SpCond, and Cond will calibrate.
  - Press enter to return to calibration parameters screen.
  - Rinse and dry probes thoroughly.
- **Calibrate Ph Second**
  - Highlight Ph and press enter.
  - Highlight 3 point cal and press enter.
  - Always start your Ph calibration with 7. Press enter.
  - Check your Ph mV readings and make sure they are within specifications.

    Buffer 7 = 0 +/- 50 mv
    Buffer 4 = +180 +/- 50 mv
    Buffer 10 = -180 +/- 50 mv
  - When the Ph readings have become stable, count to ten and press enter.
  - Once calibrated press enter again to proceed to the next "enter value" screen. Rinse and dry probes thoroughly between each solution.
  - Enter 4, and press enter.
  - Repeat steps.
  - Enter 10 for last Ph value and press enter.
  - Repeat step.
  - Press enter again to return to the 1, 2, and 3 point cal screen. Press escape to return to calibration parameters screen. Rinse and dry probes thoroughly.
- **ORP Calibration**
  - Before you do your ORP calibration you need to be aware of the temperature so you can enter the correct value.
  - Place Orp container on Ph/Orp combo probe. Orp solution in calibration cup needs to be changed after 3 – 5 uses. Mixed Zobell solution has a shelf life of six months from the day it was mixed. Unmixed Zobell solution has an expiration date on the bottle.
  - Highlight Orp and press enter. "Enter value" screen will appear.
  - Enter correct value corresponding with temperature on chart and press enter.

°C	100mV ORP	Zobell
10	124.4	250.5
15	116.7	244
20	109.1	237.5
25	100	231
30	93.1	224.5
35	84.9	218
40	76.3	211.5

  - When the Orp value has become stable, count to ten and press enter.
  - Press enter to continue to calibration parameter screen.
  - Remove Orp container, being careful not to disturb the DO o-ring and membrane, rinse and dry probes thoroughly.
- **DO Calibration**
  - Put on storage/calibration container. (Make sure there is a wet sponge in the container, and container is on loosely.)
  - With the storage/calibration container on, highlight DO and press enter.
  - Highlight DO% and press enter. Press enter again for the barometric pressure displayed.
  - When the DO% value has become stable, count to ten and press enter.
  - Press enter to return to calibration parameter screen.
- **Exiting Calibration Mode**
  - Press escape from calibration parameter screen to return to sonde menu screen.
  - Scroll down and highlight Report and press enter.
  - Scroll down to Ph mV and DO CHG, and turn them off by highlighting them and pressing enter. No solid dot next to the parameter means that it is turned off. Ph mV, and DO CHG are only needed for calibration and checking the status of the probes, and are not needed for field reports.
  - Once parameters are selected for field reports and are turned on, press escape twice and return to the 650 menu.

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STANDARD OPERATING PROCEDURE

BS-008 Fish and Benthic Macroinvertebrate Tissue Sampling

1. Objective

This SOP details the methods for proper collection, documentation, and handling for whole body tissue analysis for fish, crab, and mussel.

2. Materials

Equipment needed for collection of tissue samples may include:

- Commercial minnow trap
- Commercial crab pot
- Canned cat food (bait)
- Hand scraper
- 5 gallon bucket
- Measuring board (1 millimeter increments, at least 500 mm long)
- Scale (at least 1 gram increments)
- Aluminum foil
- Labeling tape
- Plastic bags (small and 5 gallon sizes)
- Ice or dry ice
- Coolers, packing material
- Chain of custody records, custody seals
- Decontamination equipment/supplies
- Maps/plot plan
- Safety equipment
- Camera
- Field data sheets/field notebook/waterproof pen
- Permanent markers
- Sample bottle labels
- Paper towels
- Personal Protection Equipment (PPE)
- Global Positioning System (GPS)

3. Execution

- Bait minnow traps and crab pots and deploy at designated sample locations. Traps should be tied to a stake or other secure object along shore and an identification card should be secured to the line denoting location number and relevant contact information.
- Traps containing organisms should not be permitted to become exposed (and dry) at low tide.
- Check traps at least twice daily; once in the morning and once in the evening.
- Transfer any captured organisms into a 5 gallon bucket containing surface water.
- After traps have been emptied, bait and redeploy traps.
- Time deployed and each time checked, weather conditions, tide level, organisms captured, and time redeployed should be recorded within the field notebook.
- Mussels should be collected using a hand scraper to dislodge individuals exposed during low tide.

Sample Processing

- Photograph any organism captured and record length and weight. Measurements and photograph number should be recorded in the field book, along with date, species, and location. Any abnormalities in organisms should also be recorded.
- Non-targeted species should be returned to the water after measurement.
- Length of fish is defined as the maximum body length, measured from tip of the head to the end of the longest caudal fin ray. Crabs should be measured for the maximum lateral distance across the carapace. Mussel shell length should be measured from the hinge to the outer-most portion of the shell.
- Refer to the SAP for details on target species and, tissue biomass or number of individuals need for tissue analysis.
- Individual fish should be wrapped in aluminum foil and a sample label should be placed on the outside of the foil denoting the date, sample location, fish species, length, and weight.

- Place wrapped fish in individually labeled plastic bags, which should then be placed in a labeled 5 gallon bag; one 5 gallon bag per sample location.
- Crabs and mussels should similarly be wrapped in aluminum foil and placed in individually labeled plastic bags. Individual bags should be placed in the larger bag for that sample location.
- All bags should immediately be placed on ice and stored in a cooler and maintained at 3 °C.
- Large Location sample bags should be bubble wrapped and placed in an iced cooler until transfer shipment to the analytical laboratories. Samples should be added to chain of custody form.
- If shipping time is to exceed 24 hours, samples should be frozen on dry ice.

4. Limitations

There is potential that not enough organisms will be collected at each sample location for tissue analysis. If not enough biomass can be recovered, alternative species may have to be targeted, or composite samples from multiple locations may be required.

Careful handling of organisms is required to avoid damaging of tissues and to prevent unnecessary stress to targeted and non-targeted organisms.

5. References

U.S. Environmental Protection Agency (USEPA). 2000. Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories. Volume 1 – Fish Sampling and Analysis. Third Edition. Office of Water. EPA 823-B-00-007. November.

6. Attachments

None

7. Contact

Kim Bradley

STANDARD OPERATING PROCEDURE

SM-009 Porous Surface Sampling for Polychlorinated Biphenyls (PCBs)

1. Objective

Describe methods for collection of porous surface samples (0-0.5 inches) for polychlorinated biphenyls (PCBs) analysis.

Two techniques of sample collection for PCBs are described in this procedure: hard and soft porous sampling. Hard-porous surfaces include concrete, brick, and asphalt. Soft-porous surfaces include wood, wall plasterboard, rubber, caulking, and low density plastics.

The hard-porous sampling method produces a uniform, finely ground powder that is easily homogenized, extracted and analyzed. The soft porous sampling uses a chisel or sharp knife to generate a representative sample to be extracted for analysis.

2. Execution

2.1 Hard-Porous Sampling

- Hard-porous samples are collected using an impact hammer drill, which generates a dust or powder. Having several decontaminated impact drill bits on hand will help expedite sampling when numerous sample locations are to be drilled.
- Sample locations may be pre-marked using a crayon or a non-contaminating spray paint. Note, the actual sample point must not be marked.
- Depending on the appearance of the sample location, or the objectives of the sampling, it may be appropriate to wipe the surface with a clean dry cloth prior to sampling.
- All sampling decisions and activities should be noted in the field notebook (See Limitations Below).
- Suspected stained areas should be preferentially selected.
- A ½-inch deep hole (using a 1-inch diameter drill bit) generates about 10 grams of powder.
- At each sample location, collect at least three samples of each type of hard porous surface, regardless of the amount of each type of porous surface present.
- Drill bits and sample collection pans (if used), must be decontaminated between samples.
- Lock a clean 1-inch diameter carbide drill bit into the impact hammer drill and plug the drill into an appropriate power source. A gasoline generator will be needed if electricity is not available.

- Begin drilling in the designated location. Apply steady even pressure and let the drill do the work. Applying too much pressure will generate excessive heat and dull the drill bit prematurely. The drill will provide a finely ground powder that can be easily collected, homogenized, and analyzed.
- A decontaminated stainless steel scoop can be used to collect the sample. The powder can be collected directly from the surface.

2.2 Soft-Porous Sampling

- If possible, remove any non-porous inclusions from the sampling location by brushing or wiping, as appropriate.
- Samples should be collected at a depth of no more than 0.5 inches using a metal chisel of sharp cutting knife.
- It is important to collect at least 10 grams for analysis.

2.3 Sample Handling, Preservation, and Storage

- Samples must be collected in glass two-ounce containers with a Teflon-lined cap.
- Samples ate to be shipped refrigerated and maintained at 4°C until the time of extraction and analysis.
- The holding time for PCB samples is 14 days to extraction.
- SC-002 Sample Handling provides additional guidance.
- Complete Sample Collection Logs, if appropriate, and Chain of Custody Forms, label sample containers, and complete documentation.

2.4 Decontamination

- Assemble two decontamination buckets. The first bucket contains a detergent and potable water solution, and the second is for rinsate.
- Place all used drill bits and utensils in the detergent and water bucket.
- Scrub each piece thoroughly using a scrub brush.
- Rinse each piece with water and hexane.
- Place the rinsed pieces on clean paper towels and individually dry and inspect each piece. Note: all pieces should be dry prior to reuse.
- All investigational-derived waste must be handled and disposed of in accordance with federal, state, and local regulations. Further guidance on Investigational Derived Waste is provided in GEI SOP SC-003. The waste will be treated as PCB waste if the samples are positive for PCBs.

3. Limitations

- This SOP does not cover multiple depth interval sampling.
- Sampling of wood surfaces can employ the hard or soft porous methodology.
- Porous sampling may require removing tiles or laminate coverings with asbestos containing adhesives. These coverings should not be removed without an assessment of the presence of asbestos. If asbestos is present,

the asbestos-containing material will need to be removed prior to any concrete sampling.

- If collecting multiple samples using this method, avoid cross-contamination by decontaminating all sampling tools prior to collecting the next sample. If the sampler's gloves come in contact with the sampled material during sampling, gloves should also be changed prior to collecting the next sample.

4. References

Standard Operating Procedures for Sampling Porous Surfaces for Polychlorinated Biphenyls (PCBs), The United States Environmental Protection Agency Region 1, May 2011.

Environmental Restoration Project Standard Operating Procedure for Los Alamos National Laboratory, Los Alamos National Laboratory, December 2001.

5. Contact

Brian Conte

STANDARD OPERATING PROCEDURE

SW-001 Surface Water Sampling

1. Objective

This Standard Operating Procedure (SOP) is applicable to the collection of representative surface water samples from streams, rivers, lakes, ponds, lagoons, and surface impoundments. It includes samples collected from depth as well as samples collected from the surface. These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations, or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report. Location, equipment, and sampling situations will dictate the applicable method of sample collection for each point. Representative surface water samples will be collected from one of these four techniques.

- Kemmmer bottle
- Van Doren sampler
- Direct method
- Peristaltic pump

2. Materials

Equipment needed for collection of surface water samples may include (depending on technique chosen):

- Kemmerer bottles
- Van Doren sampler
- Line and messengers
- Peristaltic pump
- Teflon™/polyethylene tubing
- Laboratory provided sample bottles
- Resealable plastic bags Ice
- Coolers, packing material
- Chain of custody records, custody seals
- Decontamination equipment/supplies
- Maps/plot plan
- Safety equipment
- Tape measure
- Survey stakes, flags, or buoys and anchors
- Camera
- Field data sheets/field notebook/waterproof pen
- Permanent marker
- Sample bottle labels
- Paper towels

- Secchi Disk – Illustration provided as Figure 1
- Personal Protection Equipment (PPE)
- Global Positioning System (GPS) survey equipment

3. Execution

3.1. Pre-Sampling Procedures

3.1.1. Sample Location

A GPS navigation system will be used to identify and record sample location coordinates. If required, the proposed locations may be adjusted based on sample location access and obstructions.

3.1.2. Water Quality Data

Water quality data will be collected during sampling from the sample depth interval using an appropriate instrument to measure pH, specific conductance, temperature, turbidity, dissolved oxygen, and oxidation-reduction potential. In addition, water clarity will be measured at each sample location using a secchi disk. The water quality meter will be calibrated daily in accordance with manufacturer's specifications.

3.2. Sample Collection

3.2.1. Kemmerer Bottle

A Kemmerer bottle will be used in most situations to collect representative samples at the specific depths that are required. A picture of the Kemmerer bottle is provided as Figure 2. Sampling procedures are as follows:

- Prior to sample collection, the Kemmerer bottle will be properly decontaminated. The sampling device will be set so that the upper and lower stoppers are pulled away from the body of the sampler, allowing the surface water to enter tube.
- Lower the pre-set sampling device to the predetermined depth while avoiding disturbance of the bottom sediments.
- When the Kemmerer bottle is at the required depth, send the weighted messenger down the suspension line, closing the sampling device.
- Retrieve the sampler and discharge the first 10-20 milliliters (mL) from the drain to clear potential contamination from the valve.
- This procedure may be repeated if additional sample volume is needed to fulfill analytical requirements. Subsequent grabs may be composited or transferred directly to appropriate sample containers.

3.2.2. Van Doren Sampler

A Van Doren sampler will be used to collect surface water from a very specific sampling depth or from a shallow water body. A picture of the Van Doran sampler is provided as Figure 3. Since the sampler is suspended

horizontally, the depth interval sampled is the diameter of the sampling tube. The sampling procedure is as follows:

- Prior to sample collection, the Van Doren Sampler will be properly decontaminated. The sampling device will be set so that the end stoppers are pulled away from the body allowing surface water to enter the tube.
- Lower the pre-set sampling device to the predetermined depth. Avoid disturbance of the bottom.
- When the Van Doren is at the required depth, send the weighted messenger down the suspension line, closing the sampling device.
- Retrieve the sampler and discharge the first 10-20 mL from the drain to clear potential contamination from the valve.
- This procedure may be repeated if additional sample volume is needed to fulfill analytical requirements. Subsequent grabs may be composited or transferred directly to appropriate sample containers.

3.2.3. Direct Method

For surface water samples collected within the top 6-inches of the water column, the direct method will be utilized to collect water samples directly into unpreserved the sample container(s).

- Analytical samples that require field preservation will be transferred from the unpreserved container to a laboratory pre-preserved sampling container.
- Ensure that all samples are collected using adequate protective clothing in accordance with the site-specific Health and Safety Plan (HASP).
- Samples will be collected in a downstream to upstream direction. In shallow locations, collect the sample under the water surface while pointing the sample container upstream; the container must be upstream of the collector.
- Avoid disturbing the sediment surface during collection. The sample container will be held below the surface to avoid the collection of floating debris.

3.2.4. Peristaltic Pump

A peristaltic pump will be used to collect surface water from a very specific sampling depth or from a remote location that cannot be accessed with other sampling methods. Since the tubing can be weighted and suspended horizontally, the depth interval sampled is the opening of the sampling tubing. The sampling procedure is as follows:

- Prior to sample collection, the tubing weights will be thoroughly decontaminated. Clean, disposable Teflon™ or polyethylene tubing will be cut to the predetermined sampling depth. The outside of the tubing will be marked with appropriate gradations to determine actual

sample depth. The tubing will be affixed to an YSI, *In-situ* Troll 9000 or similar water quality meter to ensure water quality measurements are representative of the sample interval conditions.

- Lower the tubing and water quality meter to the predetermined sample depth. Avoid disturbance of the bottom.
- When the tubing is at the required depth, turn on the peristaltic pump.
- Discharge the two submerged tubing volumes from the pump to obtain a representative surface water sample.

3.2.5. Sample Interferences

Proper sampling procedures will be used to collect samples in accordance with this SOP to prevent cross contamination and improper sample collection. Common causes of sample interferences are listed below to ensure that the samplers can avoid potential sample collection problems.

- Cross Contamination: Eliminated or minimized through the use of dedicated or disposable sampling equipment where appropriate. Where the use of dedicated or disposable sampling equipment is not possible or practical, the equipment will be decontaminated in accordance with the SOP QA-001 Equipment Decontamination.

- Improper Sample Collection: Typical improper sample collection techniques include:
  i. Improper decontamination of sampling equipment
  ii. Use of sampling equipment or sample containers that are not compatible with the contaminants of concern or the laboratory analytical method
  iii. Excess sediment in the sample due to disturbance of the canal sediments by sampling equipment
  iv. Sample collection in an obviously disturbed or non-representative area
  v. Sample collection during a period of increased surface water velocity that causes significant re-suspension of canal sediments (i.e. tidal influences, storm surge)

3.2.6. Quality Assurance/Quality Control (QA/QC)

QA/QC procedures that apply to the these activities include QA/QC laboratory samples including blind duplicate, matrix spike and matrix spike duplicate (MS/MSD) samples, and field blank samples. QA/QC samples are detailed in the Work Plan and the Draft Quality Assurance Project Plan (QAPP). Prior to collection of the QA/QC samples, equipment will be decontaminated in accordance with procedures described in SOP QA-001 Equipment Decontamination.

The following general QA procedures apply:

- All data must be documented on field data sheets or within site field notebooks.
- All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation and they must be documented as indicated in the Draft QAPP.
- To avoid the incidental inclusion of disturbed sediment in the sample, surface water should be collected from a downstream to upstream direction and upstream of any activity that may disturb the sediment (i.e., wake from boat).

4. Limitations

There are two primary interferences or potential problems associated with surface water sampling. These include cross contamination of samples and improper sample collection.

- Cross contamination problems can be eliminated or minimized through the use of dedicated or disposable sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary.
- Improper sample collection can involve using contaminated equipment, equipment that is potentially not compatible with the contaminants of concern, disturbance of the stream or impoundment substrate, and sampling in an obviously disturbed or non-representative area. Be sure to use sampling equipment of an appropriate composition based upon the suspected contaminants and analyses to be performed.

Following proper decontamination procedures, minimizing disturbance of the sample site, and careful selection of sampling locations will eliminate these problems. Proper timing for the collection of samples must be taken into consideration due to tidal influences and low or fast-flowing streams or rivers.

5. References

Wilde, F.D., D.B. Radtke, J. Gibs and R.T. Iwatsubo. 1998. National Field Manual for the Collection of Water-Quality Data - Selection of Equipment for Water Sampling. U.S. Geological Survey Techniques of Water - Resources Investigations, Book 9, Chap. A2, variously paged. http://water.usgs.gov/owq/FieldManual/index.html and http://water.usgs.gov/owq/FieldManual/mastererrat.html

U.S. Environmental Protection Agency. 1984. Characterization of Hazardous Waste Sites - A Methods Manual: Volume II. Available Sampling Methods, Second Edition. EPA/600/4-84-076.

U.S. Environmental Protection Agency. 2002. U.S. EPA Environmental Response Team, Standard Operating Procedures #2013, Surface Water Sampling. EPA, 12/17/02.

6. Attachments

Figures 1, 2, and 3

7. Contact

Steven Canton

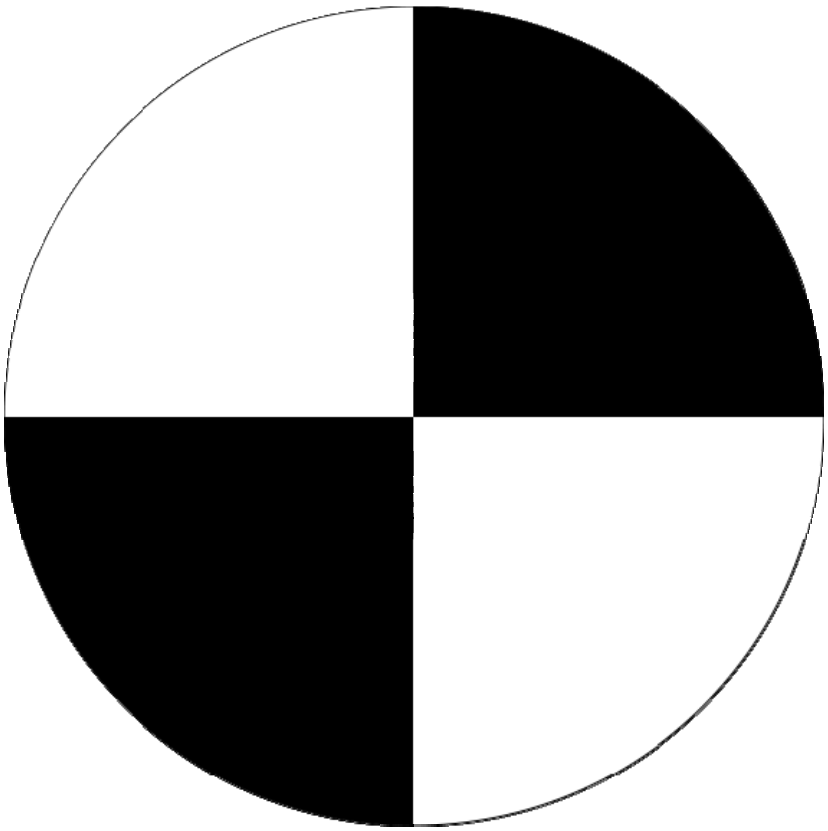

Figure 1 - Secchi Disk

Figure 2 – Kemmerer Bottle

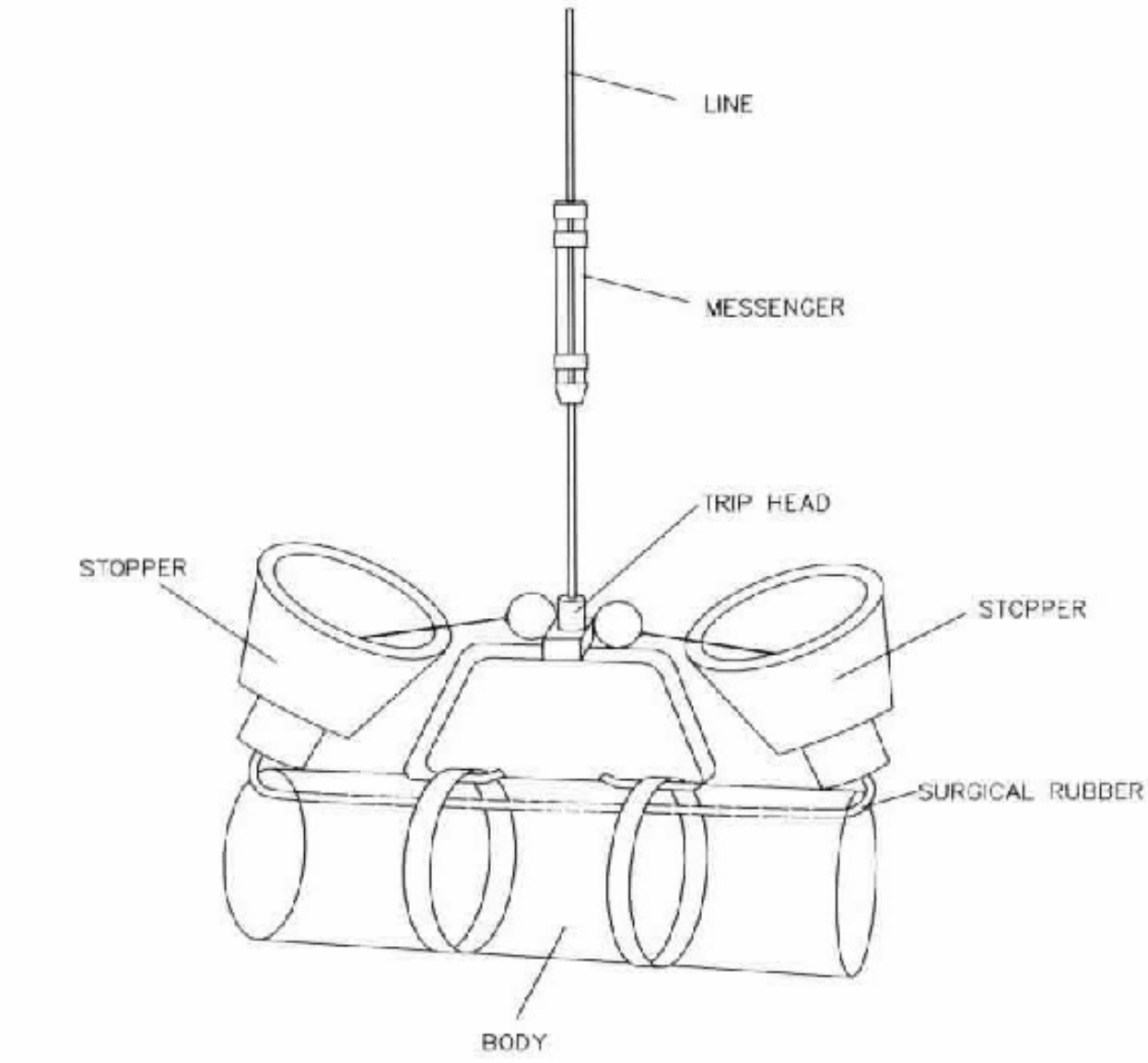

Figure 3 - Van Doran Sampler

STANDARD OPERATING PROCEDURE

BS-009 Mussel Handling and Tissue Extraction

1. Objective

This SOP details the methods for proper extraction of soft tissue from mussels for tissue analysis.

2. Materials

Equipment needed for collection of tissue may include:

- Commercial shucking knife
- Ice
- DI water

3. Execution

- Specimens should be unwrapped and inspected upon arrival to ensure samples have not been compromised. Any samples deemed unsuitable should be placed back in storage and the project manager should be informed of any issues. Compromised specimens should not be processed unless permission is given by the project manager or replacement samples are obtained.

Sample Processing

- If frozen, mussels should not be thawed to prevent the loss of liquids during tissue removal
- Organisms should be rinsed thoroughly with organic- and metal-free water prior to tissue removal to remove any external debris
- Using a shucking knife, the byssal threads (i.e. “beard”) from mussels should be removed and should not be included in the tissue analysis
- Insert the shucking knife near the hinge of the mussel
- Once the knife is able to penetrate into the shell, run the knife around the entire length of the shell in order to sever the adductor muscle
- Working the knife blade at the front of the shell, separate the two shell halves
- Scrape all soft tissue from the top and bottom shell halves. This includes viscera, meat, and body fluids
- Tissues should be placed on a clean surface or in a clean container until further processing or compositing of samples

4. Limitations

There is potential that not enough organisms will be collected at each sample location for tissue analysis. If not enough biomass can be recovered, alternative species may have to be targeted, or composite samples from multiple locations may be required.

5. References

U.S. Environmental Protection Agency (USEPA). 2000. Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories. Volume 1 – Fish Sampling and Analysis. Third Edition. Office of Water. EPA 823-B-00-007. November.

6. Attachments

None

7. Contact

Kim Bradley

STANDARD OPERATING PROCEDURE

SM-008 Wipe Sampling

1. Objective

Describe methods to standardize the collection of wipe samples.

2. Execution

- Determine the appropriate surface area (typically in cm^2) to be wiped.
- Choose appropriate sampling points and mark the surface area to be sampled. Photo documentation is recommended.
- Record surface area to be wiped.
- Don a new pair of disposable surgical gloves.
- Open new sterile package of gauze pad.
- Soak the pad with required solvent unless the laboratory has provided pre-soaked pads.
- Wipe the marked surface area using firm strokes. Wipe vertically, then horizontally to insure complete surface coverage.
- Place the gauze pad in an appropriately prepared sample container with a Teflon-lined cap.
- Cap the sample container, attach the label and custody seal, and place in a plastic bag. Record all pertinent data in the site fieldbook and/or on field data sheets.
- Store samples out of direct sunlight.
- Follow proper decontamination procedures, then deliver sample(s) to the laboratory for analysis.

3. Limitations

- Wipe sampling may require removing tiles or laminate coverings with asbestos containing adhesives. These coverings should not be removed without a determination of the presence of asbestos. If asbestos is present, the asbestos-containing material will need to be removed prior to any concrete sampling.
- If collecting multiple samples using this method, avoid cross-contamination by decontaminating all sampling tools prior to collecting the next sample. If the sampler's gloves come in contact with the sampled material during sampling, gloves should also be changed prior to collecting the next sample.

4. References

"Wipe Sampling and Double Wash/Rinse Cleanup as Recommended by the Environmental Protection Agency PCB Spill Cleanup Policy," dated June 23, 1987 and revised on April 18, 1991.

Environmental Restoration Project Standard Operating Procedure for Los Alamos National Laboratory, Los Alamos National Laboratory, December 2001.

5. Contacts

Brian Conte
Leslie Lombardo

Attachment C

Laboratory Standard Operating Procedures

SOP Change in Progress Attachment (CIPA)

SOP Number	SOP Title	SOP Revision	SOP Effective Date	CIPA Effective Date
BR-EX-005	Separatory Funnel Extraction (SW-846 3510C)	9	11/08/11	10/01/12

The following revisions were made to this standard operating procedure (SOP). These changes are effective as of the CIPA Effective Date. This change to this document is authorized by the laboratory's QA Department.

Page 7 of 12, Section 10.4 Extraction Concentration (KD-Technique):

Remove the phrase (KD-Technique) from header 10.4 so that the new header reads:

10.4 Extraction Concentration

Insert the following text between 10.4 and 10.4.1:

The following sections describe the procedures for the concentration of extracts. Any of the three techniques described may be used. However some techniques are more efficient than others to achieve the final extract volume. Use the following guidelines to select the concentration technique: For the concentration of volumes greater than 5 mL, use Macro Concentration. To concentrate extract volumes between 5 and 1 mL use Micro Concentration. To concentrate extract volumes below 1 mL, use Nitrogen Blowdown.

Title: SEPARATORY FUNNEL EXTRACTION (SW-846 3510C)

Approval Signatures:

William S. Cicero
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Dan Helfrich
Health & Safety Coordinator

Approval Date: November 8, 2011

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This SOP describes the laboratory procedure for the isolation of organic compounds from aqueous samples by separatory funnel liquid-liquid extraction.

1.1 Analytes, Matrix(s), and Reporting Limits

Refer to analytical methods for analyte lists and reporting limits.

2.0 Summary of Method

A measured volume of sample, usually 1 L, is serially extracted at a specified pH with methylene chloride using a separatory funnel rotator. The extract is dried with anhydrous sodium sulfate then concentrated, and if necessary, exchanged into a solvent compatible for extract cleanup or the determinative analysis method.

This SOP is based on the following reference method:

- SW-846 Method 3510C Separatory Funnel Liquid-Liquid Extraction, Revision 3, December, 1996.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment that can cause interference and/or elevated baselines in chromatography. All reagents and solvents used during this procedure should be reagent grade or high purity in order to minimize interference. All glassware must be cleaned in accordance with laboratory SOP BR-EX-017 *Glassware Cleaning Procedure* and rinsed with acetone and methylene chloride prior to use.

The decomposition of some analytes has been demonstrated under basic extraction conditions. Organochlorine pesticides may dechlorinate, phthalate esters may exchange, and phenols may react to form tannates. These reactions increase with increasing pH, and are decreased by the shorter reaction times available in Method 3510. Method 3510 is preferred over Method 3520 for the analysis of these classes of compounds. However, the recovery of phenols may be optimized by using Method 3520, and performing the initial extraction at the acid pH.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP and should not be used.

During Kuderna-Danish (KD) concentration do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield should be worn over safety glasses or goggle when it is performed.

The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds should be prepared in hood.

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The KD apparatus has ground glass joints which can become stuck. Technicians must use Kevlar or other cut/puncture resistant gloves when separating stuck joints.

5.2 Primary Materials Used

Table 1 lists those materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Extraction Equipment

- Separatory Funnel Shaker, Glas-Col or equivalent.
- Teflon Separatory funnel: 2 Liter, with stopcock and cap, Fisher Scientific or equivalent.
- Glass Funnel: 75mm, Fisher Scientific or equivalent.

6.2 Extract Concentration (KD Apparatus)

- Concentrator Tube: 10 mL graduated, ChemGlass Catalog Number CG-1316-11 or equivalent.
- Snyder Column: Three ball macro AMK Catalog Number SC2-01 or equivalent.
- Snyder Column: Two ball micro AMK Catalog Number SC3-01 or equivalent.
- Evaporation Flask: 500 mL attached to concentrator tube with clip, AMK Catalog Number KDF-500 or equivalent.
- Boiling Chips: silicon carbide, approximately 10/40 mesh, solvent extracted in methylene chloride, Troemner Catalog Number 133B or equivalent.
- Heating mantle rheostat controlled for water bath capable of temperature control (± 5°C). ChemGlass Catalog Number PL3122 or equivalent.
- Water Bath: capable of temperature control to ±5°C, Barnstead Corporation Catalog Number HM0500-HS1 or equivalent.
- Solvent Vapor Recovery System: Kontes K-54000-1006, K-547300-000, Ace Glass Catalog Number 6614-30 or equivalent.

6.3 Miscellaneous

- pH Paper/Strips: Range 1-14.
- pH Meter. Denver Instruments Model UB5 or equivalent.
- Pasteur Glass Pipettes: 1 mL, disposable. Fisher Scientific or equivalent.
- 0.5 mL – 2.0 mL Syringes: Hamilton Gastight® Syringes or equivalent.
- Vials and Caps: 1.8, 4, 8, 16, and 40 mL with Teflon lined septa and screw caps. Fisher Scientific.
- Graduated Cylinder: 1 Liter, Class A. Fisher Scientific or equivalent.
- Glass Wool

7.0 Reagents and Standards

7.1 Reagents

- Sodium Sulfate (granular, anhydrous), Na_2SO_4. J.T. Baker or equivalent. Purify by heating at 400°C for at least 4 hours.
- Methylene Chloride (CH_2C_{12}): Pesticide Quality, J.T. Baker or equivalent.
- Hexane, (C_6H_{14}), Pesticide Quality, J.T. Baker or equivalent.
- Acetone, ($(CH_3)_2CO$), Pesticide Quality, J.T. Baker or equivalent.
- Reagent Water: RO water filtered through a Nanopure System.
- Sodium Hydroxide (NaOH): Reagent Grade, J.T. Baker.
- Sulfuric Acid, (H_2SO_4): Reagent Grade, J.T. Baker.

7.1.1 Prepared Reagents

- NaOH Solution (6N): In a 2.5 L clear glass bottle dissolve 240 g NaOH in reagent water and dilute to 1 Liter. Store the solution in reagent bottle at room temperature. Assign an expiration date of 6 months from date of preparation unless the parent material expires earlier, in which case, use the earliest expiration date.

- H_2SO_4 Solution (1:1 v/v): Add 500 mL of reagent water to a 1 L volumetric flask. Slowly add 500 mL of H_2SO_4 to the flask to dilute to volume. Store the solution in reagent bottle at room temperature. Assign an expiration date of 6 months from date of preparation unless the parent material expires earlier, in which case, use the earliest expiration date.

7.2 Standards

Purchase stock standards as certified solutions from commercial vendors. Prepare surrogate and spiking solutions in the laboratory by diluting a known volume of the stock standard solutions in an appropriate solvent. Record the preparation of standard in the LIMS (TALS) module established for this purpose.

Store prepared standard solutions in glass containers at 4°C or below. Unless otherwise specified, assign an expiration date of 6 months from the date of preparation or in accordance with the expiration date of the parent standard, whichever is sooner. The recommended formulation for each standard used in this procedure is provided in the analytical method along with the recommended source materials, expiration dates and storage conditions.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are the minimum sample size for collection, preservation for shipment to the laboratory and holding time requirements as specified by SW-846 Chapter 4, Table 4-1. Footnotes to this table specify this information is guidance and does not represent EPA requirements. Selection of containers, preservation techniques and sample collection size should be based on project specific data quality objectives.

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time[1]	Reference
Water	1L Amber Glass	4X1L	Cool to ≤ 6°	Extraction: 7 days	SW-846 Chapter 4

[1]Extraction holding time is determined from sampling date.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Analytical SOP
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Analytical SOP
Matrix Spike(s) MS/MSD	Client Request	See Analytical SOP
Sample Duplicate (SD)	Client Request	See Analytical SOP

9.2 Instrument QC

Refer to the analytical SOP for the determinative test method.

10.0 Procedure

10.1 Instrument Calibration

Check the calibration of the pH meter and the balance each day of use prior to use and record these checks in the logbook designated for this purpose.

Verify the calibration of any mechanical pipettes used is current and if it is not, notify QA. The QA department checks the calibration of pipettes quarterly in accordance with the procedures specified in laboratory SOP BR-QA-008.

10.2 Equipment Preparation

Prepare glassware using the procedures described in laboratory SOP BR-EX-017 and rinse with acetone and methylene chloride prior to use. Label the glassware for each field and QC sample clearly and unambiguously during each step of the extraction procedure. Solvent will erase grease pens and “sharpie ink”; so use caution to ensure labels are not obliterated during the procedure.

Assemble a drying funnel for each field and QC sample by placing a plug of glass wool in a 75mm glass funnel then add a sufficient amount of purified granular sodium sulfate to fill the funnel ¾ full. Rinse the funnel with ~30 mL of acetone and ~ 30 mL of methylene chloride each and discard the solvent rinse.

Assemble a KD setup for each field and QC sample by attaching a 10 mL concentrator tube to a 500 mL evaporation flask and attach this to the drying funnel.

10.3 Extraction

If samples were received in 1 L containers, mark the meniscus of the sample container with a permanent marker. If a different size container was provided, measure 1 L of sample into a graduated cylinder. Transfer 1 L of reagent water to individual separatory funnels to serve as the method blank and LCS.

Add the proper type and volume of surrogate solution to each sample container (or graduated cylinder) and to the MB and LCS. Add the proper type and volume of spike solution to any samples designated as MS/MSD and to the LCS. See the extraction conditions workbook for type, concentration and amount of surrogate and spike solution to use for each test method. If the sample container is filled to the top of the bottle, remove and discard an amount of sample sufficient to add the spike and surrogate solution.

Check the pH of each sample using a calibrated pH meter or wide range pH paper. Adjust the pH, if necessary, using 1:1 sulfuric acid solution or 6N sodium hydroxide solution. See the extractions conditions workbook to determine the extraction pH for each test method. If using the pH meter, thoroughly rinse the probe of the pH meter with reagent water between each sample in order to avoid cross-contamination. If using pH paper, do not dip the paper into the sample, instead aliquot a drop of sample onto the pH paper.

Quantitatively transfer the entire sample into the 2 L separatory funnel. Rinse the sample container with ~60 mL methylene chloride and pour the rinsate into the separatory funnel extractor. To measure the sample volume, fill the sample container with tap mater to the mark of

the meniscus and pour the water into a graduated cylinder for volume measurement. Record the sample volume in the TALS worksheet.

Note: If high analyte concentrations are anticipated, a smaller sample volume may be taken and diluted to 1L with reagent water. If smaller volumes are used, it may be necessary to adjust the volume of surrogate and spike solution. Consult with the Department Supervisor for further guidance.

Cap and shake each separatory funnel vigorously for 15-20 seconds. Immediately after the first shake, vent the separatory funnel into a fume hood. Place the separatory funnel on the Sep. Funnel shaker and shake for 2 minutes. After the 2 minute shake is complete allow sufficient time for the organic layer to separate from the water layer. If an emulsion forms and it is more than one-third the size of the solvent layer, filter, centrifuge, or stir the extract to remove the emulsion.

Drain the solvent portion from the extractor into the drying funnel/concentration apparatus and repeat the extraction two more times using fresh portions of methylene chloride, combining the three extracts. If further pH adjustment and secondary extraction is required adjust the pH and repeat the extraction three more times.

Concentrate the extract using KD technique and adjust the extract to the final volume required for the test method.

10.4 Extract Concentration (KD Technique)

10.4.1 Macro Concentration

Macro Snyder Column (K-D)

Add one or two clean boiling chips to the K-D evaporation flask and attach a three-ball Snyder column to the flask. Add ~1 mL of methylene chloride to the top of the column then place the K-D apparatus in a hot water bath (60-70°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in hot water vapor.

Attach the solvent vapor recovery glassware to the Snyder column. Adjust the vertical position of the apparatus and check the water bath temperature. The water bath temperature should be between 54.8 – 74.8°C when methylene chloride is the extraction solvent and 84-89°C when hexane is the extraction solvent. Higher water bath temperatures may be used so long as the recovery of target analytes is not impacted. The boiling point of each solvent is provided in the following table:

Solvent	Boiling Point	Water Bath Temperature
Hexane	69°C	84 – 89°C
Methylene Chloride	39.8°C	54.8 – 74.8°C

Monitor the concentration and do not let the extract evaporate to dryness. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with solvent.

When the apparent volume of the extract reaches desired amount remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

10.4.2 Micro Concentration

Add one or two clean boiling chips to the concentrator tube and attach a two ball micro-Snyder column to the tube. Place the concentrator tube into the water bath so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the concentrator tube and check the temperature of the water bath to ensure the proper temperature for the extract solvent.

Continuously monitor the distillation process to ensure sample extracts do not evaporate to dryness. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with solvent. Remove setup when desired sample volume is reached.

10.4.3 Nitrogen Blowdown

Nitrogen blow down may be used to concentrate extracts as needed.

Place the concentrator tube in a warm water bath maintained at a temperature of 35°C. Apply a steady stream of nitrogen until the desired final extract volume is achieved. Rinse the internal wall of the concentrator tube several times with the appropriate solvent during the evaporation and ensure the solvent level in the concentrator is positioned such to prevent water condensations. Monitor the concentration carefully and do not allow the extract to evaporate to dryness.

10.5 Extract Preparation & Handling

Transfer the extract to labeled Teflon lined screw cap vial.

Adjust the extract to volume using a reference vial for final extract volumes greater than 1 mL and by using the tip of concentration tubes for volumes 1 mL and less. Before using the reference vial, verify the reference vials were prepared on the same day by an analyst authorized to prepare reference vials. Place the extracts in refrigerated storage maintained at a temperature of 4°C±2 in preparation for subsequent cleanup or analysis as specified by the method chain.

If the TALS log-in does not include cleanup methods in the method chain and there is reason to believe the extract may require cleanup (color, odor, viscosity, etc.) notify the PM of the situation so he/she can determine if cleanup should be performed prior to analysis.

Complete the TALS batch worksheet and perform primary review of the extraction batch.

11.0 Calculations / Data Reduction

11.1 Calculations

Calculations are provided in the analytical SOP for each method parameter.

11.2 Data Review

Refer to laboratory SOP BR-QA-019 for the required elements of each step of data review.

11.2.1 Primary Review

Review the batch worksheet for correctness and completeness. Record any problems encountered during the extraction process with a nonconformance memo (NCM).

11.2.2 Secondary Review

Review the batch worksheet for correctness and completeness and to ensure the extraction performed is consistent the SOP and project specifications.

Print the output worksheets and release extracts and output worksheet to the analytical department or to the next step in the method chain such as extract cleanup.

If the TALS log-in does not include cleanup methods in the method chain and there is reason to believe the extract may require cleanup (color, odor, viscosity, etc.) notify the PM of the situation so he/she can determine if cleanup should be performed prior to analysis.

For additional guidance regarding the laboratory's protocol and required elements for data review refer to laboratory SOP BR-QA-019.

12.0 Method Performance

12.1 Detection Limit (DL), Limit of Detection (LOD) and Limit of Quantitation (LOQ)

Refer to the analytical SOP for DL, LOD and LOQ requirements.

12.2 Demonstration of Capabilities (DOC)

Each analyst must complete an Initial Demonstration of Capability prior to unsupervised performance of this method.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures

are incorporated by reference to BR-EH-001 *Hazardous Waste*. The following waste streams are produced when this method is carried out.

- Organic Solvents - Satellite container: 55 Gallon Covered and Vented Drum.
- Extracted water samples - Satellite Container: 55 Gallon Covered and Vented Drum.
- Vials containing extracts - Satellite Container: 5 Gallon Covered Bucket (inside fume hood).
- Solid Waste - Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood).

15.0 References / Cross-References

- SW-846 Method 3510C Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.
- Laboratory SOP BR-EX-017
- Laboratory SOP BR-QA-019
- Laboratory SOP BR-QA-005
- Laboratory SOP BR-EH-001
- Corporate Environmental Health and Safety Manual

16.0 Method Modifications

There are no modifications of the referenced method.

17.0 Attachments

- Table 1: Primary Materials Used
- Appendix A: Terms and Definitions

18.0 Revision History

Revision 9:
- Title Page: Update Approval Signatures and copyright date
- Section 8: Updated text to cite regulatory reference and add that this information is guidance per the language in SW-846 Chapter 4.
- Section 10.5: Added procedure for use of reference vials and final extract volume adjustment and added laboratory policy for cleanups when cleanups are not included in method chain.
- Section 11.0: Added statement about laboratory for cleanups when cleanups are not included in the method chain.

Revision 8:
- Updated Title Page
- All Sections: Added TALS terminology and removed reference to bench sheets and added reference to extraction conditions workbook.
- All Sections: Changed Sep Funnel rotator to Sep. Funnel shaker.
- Section 10.3: Changed sequence of procedure so that surrogate and spike solutions are added to sample container before transfer to extraction vessel and to perform pH measurement and adjustment before transfer to extraction vessel.

Revision 7:
- Sections 5.0 & 5.2: Updated with new language.
- Section 6.3: Remove balance and added glass wool.

Table 1: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Sulfuric Acid[1]	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

[1] *Always add acid to water to prevent violent reactions.*
[2] *Exposure limit refers to the OSHA regulatory exposure limit.*

Appendix A: Terms and Definitions

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

Title: HOMOGENIZATION OF BIOTA & TISSUE

Approval Signatures:

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Approval Date: August 1, 2012

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This SOP describes the laboratory procedure for the whole body homogenization and extraction of biota and tissue samples by tissuemizer in preparation for analysis by a variety of chromatographic procedures.

NOTE: Pre-preparation of tissue such as dissection, sportsman filet, sample compositing, shucking, etc. may be performed by the laboratory per customer specification. These procedures are project specific and instructions are not included in this SOP.

2.0 Summary of Method

2.1 Homogenization

Tissue samples are homogenized using a titanium blade homogenizer. Biota samples are homogenized using stainless steel knives and/or a food processor. The homogenized sample(s) are transferred to labeled glass jars and stored in a freezer maintained at a temperature of -15ºC (±5ºC) in preparation for extraction.

The laboratory's standard procedure is to perform whole body homogenization of the tissue sample. Any customer specifications for dissection or homogenization of certain parts of the tissue sample, compositing multiple samples or other must be negotiated with the laboratory during project initiation and specific instructions for sample processing must be prepared and provided to the extraction laboratory by the laboratory PM. In the absence of instruction, the laboratory will homogenize the entire sample.

The homogenization procedure is a laboratory developed procedure based on the procedures described in the Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, National Status and Trends Program for Marine Environmental Quality.

2.2 Extraction

A portion of homogenized sample is mixed with anhydrous sodium sulfate then macerated for 3 minutes in an appropriate extraction solvent using the Tissumizer. The solvent layer decanted poured through sodium sulfate and collected in a collection vessel. The extraction is repeated two more times with fresh portions of extraction solvent. After extraction, the combined extracts are concentrated to an appropriate final volume using K-D Technique. Percent lipids are determined following procedures given in laboratory SOP BR-EX-016 Percent Lipid Determination and extract cleanup is performed when necessary.

The extraction procedure is a laboratory developed procedure based on the procedures described in the Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, National Status and Trends Program for Marine Environmental Quality.

3.0 Definitions

- Biota: flora and fauna. For this SOP, all reference to "biota" refers to plant material.

- Tissue: an aggregate of cells usually of a particular kind together with their intercellular substance that form one of the structural materials of a plant or animal. For this SOP, all reference to "tissue" refers to structural materials from an animal.

A list of general terms and definitions are provided in Appendix A.

4.0 Interferences

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment that can cause interference and/or elevated baselines in chromatography. All reagents and solvents used during this procedure should be reagent grade or high purity in order to minimize interference. All glassware must be cleaned in accordance with laboratory SOP BR-EX-017 Glassware Cleaning, and rinsed with acetone and methylene chloride prior to use.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Homogenization Equipment

- Cutting Board- High density polyethylene 16X23"

- Homogenizer equipped with 55 mm Titanium Blade Omni International or equivalent.
- Food Processor Cuisinart or equivalent
- Stainless steel knives
- Glass Jars, wide mouth; 125 mL-1000 mL. ESS or equivalent.
- Meat Grinder

6.2 Extraction Equipment

- Tissuemizer equipped with a 20 mm x 195 Generator probe. Omni International PowerGen 700 or equivalent.
- Filter Funnels – 100 mm diameter for filtration/drying. Fisher Scientific or equivalent.
- No. 54 Filter paper. Whatman 18.5 cm, or equivalent.
- Beakers – 400 mL. Fisher Scientific or equivalent.

6.3 Extract Concentration (KD Apparatus)

- Concentrator Tube, 10 mL Graduated: ChemGlass Catalog # CG-1316-11 or equivalent
- Snyder Column: Three ball macro, AMK Catalog # SC2-01 or equivalent
- Snyder Column: Two ball micro, AMK Catalog # SC3-01 or equivalent
- Evaporation Flask: 500 mL attached to concentrator tube with clip, AMK Catalog Number KDF-500 or equivalent.
- Boiling Chips: Silicon carbide, approximately 10/40 mesh, solvent extracted in methylene chloride, Troemner Catalog # 133B or equivalent.
- Heating Mantle: Rheostat controlled for water bath capable of temperature control (±5°C). ChemGlass Catalog # PL3122 or equivalent.
- Water Bath, capable of temperature control to ±5°C. Barnstead Corporation Catalog # HM0500-HS1 or equivalent.
- Solvent Vapor Recovery System, Kontes K-54000-1006, K-547300-000, Ace Glass Catalog # 6614-30 or equivalent.

6.4 Miscellaneous

- Disposable Glass Pasteur Pipette and bulb: Fisher Scientific or equivalent.
- Top Loading balance: Capable of measuring to 0.01 gram accuracy, Mettler Model # PM4800 or equivalent.
- Vials and caps: 2, 4, 8, and 16 mL with Teflon lined septa and screw caps, Fisher Scientific or equivalent.
- Teflon and Stainless Steel Spatulas, Fisher Scientific or equivalent.
- Adjustable Pipette: Finnpipette or equivalent
- 0.5 mL – 2.0 mL Hamilton Gastight® syringes or equivalent.
- Paper towels

7.0 Reagents and Standards

7.1 Reagents

- Sodium Sulfate (Na_2SO_4), Granular Anhydrous: J.T. Baker or equivalent. Purify by heating at 400°C for at least 4 hours.
- Methylene Chloride (CH_2C_{12}): Pesticide Quality, J.T Baker or equivalent.

- Hexane, (C_6H_{14}): Pesticide Quality, J.T. Baker or equivalent.
- Acetone, ($(CH_3)_2CO$): Pesticide Quality, J.T. Baker or equivalent.
- Reagent Water: RO water filtered through a Nanopure System.
- Alkaline Liquid Detergent: Contrex or equivalent.

Methylene Chloride/Acetone (1:1): In a 4 L amber glass bottle mix 2 L methylene chloride with 2 L acetone. Store the solution in a fume hood. Assign an expiration date of 6 months from date of preparation unless the parent material expires earlier, in which case, use the earliest expiration date.

Hexane /Acetone (1:1): In a 4 L amber glass bottle mix 2 L hexane with 2 L acetone. Store the solution in a fume hood. Assign an expiration date of 6 months from date of preparation unless the parent material expires earlier, in which case, use the earliest expiration date.

7.2 Standards

Purchase certified stock standards from commercial vendors and from these prepare surrogate and spiking solutions by diluting a known volume of the stock standard solutions in an appropriate solvent. Record the preparation of all standards in the LIMS module. The formulations for the preparation of surrogate and spiking standards are provided in analytical SOPs.

Unless otherwise specified in the analytical SOP store prepared in glass containers at 4°C or below and assign an expiration date of 6 months from the date of preparation unless the parent standards expire earlier in which case use the earliest expiration date.

Assay surrogate and spike solutions before each use to verify the made to concentration is within specifications. Maintain records of the assay per the procedure established for the work section.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP.

The laboratory recommends that tissue and biota samples be collected in glass jars or sealable plastic bags. Immediately following collection, biota samples should be iced to a temperature of 4°C (±2°C) and tissue samples should be frozen and maintained at a temperature of -15°C (±5°C) until the time of homogenization. After homogenization, all homogenized samples must be stored in a freezer maintained at a temperature of -15°C (±5°C).

Tissue and biota samples must be extracted within the holding time specified in the client's quality assurance plan. In the absence of client specifications, the following HT will be used:

SVOA GC/MS: 14 Days from Date of Collection
Pesticides: 14 Days from Date of Collection
PCB: 365 Days from Date of Collection
Mercury: 28 Days from Date of Collection
Metals: 180 Days from Date of Collection

After extraction any remaining sample should be returned immediately to the freezer or stored per the project specifications.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Analytical SOP
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Analytical SOP
Matrix Spike(s) MS/MSD	Client Request .	See Analytical SOP
Sample Duplicate (SD)	Client Request	See Analytical SOP

10.0 Procedure

10.1 Instrument Calibration

Check the calibration of the balance each day of use prior to use.

Check the calibration of the adjustable pipettes quarterly.

Perform periodic maintenance on the tissuemizer's generator probe as necessary. Maintenance may include but is not limited to the replacement of Teflon bearings and rotor shafts when a loud squealing noise is heard. Refer to the PowerGen 700 Homogenizer Instruction Manual for further guidance and for the manufacturer's recommended maintenance program.

10.2 Whole Body Homogenization of Tissue Samples

Remove the samples from storage and let the sample(s) thaw completely.

Wash all equipment with detergent and hot water, then rinse with reagent water and acetone prior to use and also after each sample.

Prepare a equipment blank to ensure the equipment is clean. To prepare the blank, transfer a representative amount of reagent water (similar to the size of the samples) to a clear glass jar. Let the water come into contact with all equipment that will be used to homogenize the samples. This process should be done randomly during homogenization to verify the cleaning process.

If possible obtain a glass jar large enough to accommodate the entire sample. Label the jar with the sample's lab ID and place the jar on the analytical balance. Tare the balance. Put on a pair of nitrile gloves and with your hands transfer the sample from the storage container to the labeled jar. Record the pre-homogenization weight of the sample in the worksheet.

Transfer the sample to a pre-cleaned cutting board. Cut the sample into 1-3" sections using a stainless steel knife then place the sections of samples in the labeled jar.

Insert the titanium blade into the jar and homogenize the sample at 2000-4000 RPM until the sample becomes slurry. Manual mixing with a stainless steel or Teflon spatula may be required

to insure complete homogenization. Remove the blade from the sample jar and scrape any remaining sample from the blade into the labeled jar. Place the jar on the top-loading balance and record the post homogenization weight in the worksheet.

NOTE: Samples greater than 12 inches or samples with weight measurements that exceed 1-2 pounds should be homogenized in a stainless steel meat grinder prior to use of the titanium blade. If the sample is extremely large homogenization with a knife may be necessary. If the homogenized tissue sample cannot fit into a single container, pass the sample through the meat grinder multiple times, homogenize the slurry and transfer to multiple containers.

10.3 Homogenization of Biota Samples

Wash all equipment with detergent and hot water, then rinse with reagent water and acetone prior to use and also after each sample.

Prepare a equipment blank to ensure the equipment is clean. To prepare the blank, transfer a representative amount of reagent water (similar to the size of the samples) to a clear glass jar. Let the water come into contact with all equipment that will be used to homogenize the samples. This process should be done randomly during homogenization to verify the cleaning process.

Remove the samples from storage and let warm to ambient temperature. Select a glass jar large enough to accommodate the entire sample. Label the jar with the sample's lab ID and place the jar on the top-loading balance. Tare the balance. Remove the biota sample from the storage container and place in the labeled jar. Record the pre-homogenization weight of the sample into the worksheet.

Remove the sample and place on a pre-cleaned cutting board. Slice the material into very fine sections using a stainless steel knife or a food processor. Return homogenized sample back into the jar and place on the top-loading balance. Record the post homogenization weight into the worksheet.

10.4 Extraction

Clean all glassware prior to use following the procedure given in laboratory SOP BR-EX-017. Label all glassware with field and QC samples ID numbers clearly and unambiguously during each step of the extraction procedure. Solvents will erase grease pens and "sharpie ink", so caution must be taken to ensure that the labels are not obliterated during the procedure.

Assemble a KD apparatus set-up and prepare a glass funnel for each sample to be extracted. Fold a 185 mm Whatman® 54 filter into quarters and place a filter in each funnel. Fill each funnel ~3/4 full with purified granular anhydrous sodium sulfate. Rinse the funnel with ~30 mL acetone and methylene chloride each and discard the solvent rinse. Place a prepared funnel onto each K-D setup.

Assemble the Tissuemizer by attaching the 20 mm x 195 mm-generator probe to the Tissumizer motor. Place the Tissuemizer in the fume hood and attach to the aluminum staging using clamps. Clean the Tissuemizer prior to use by running the generator probe for 10 seconds in a 400 mL beaker filled with ~ 200 mL of reagent water. Discard the reagent water and repeat with another aliquot of reagent water. Repeat the cleaning step two more times each with ~250 mL of acetone.

Mix the sample using a stainless steel or Teflon spatula. Place a labeled 400 mL beaker onto the top-loading balance and depress the "tare" button. Referring to the extraction condition spreadsheet, weigh out the appropriate amount of sample and record sample weight ± 1 gram into TALS. Repeat for all samples. Transfer two additional aliquots of the sample selected for the MS and MSD into labeled 400 mL beakers. Transfer the same weight of sodium sulfate each into labeled 400 mL beakers to serve as the method blank (MB) and laboratory control sample (LCS).

Add a sufficient volume of granular sodium sulfate to each sample and mix thoroughly with a stainless steel spatula until a free-flowing mixture is formed.

Add the appropriate volume of surrogate spike to each field sample and QC sample. Add the appropriate volume of spike solution to the laboratory control samples and the MS/MSD.

Add 100 mL of the appropriate extraction solvent to each beaker. Use 1:1 $MeCl_2$/Acetone for samples to be analyzed by GC/MS and 1:1 Hexane/Acetone for GC/ECD.

Refer to the Extractions Condition Worksheet to determine the type and amounts of solution added and the extraction solvent used for each test method.

Immerse the generator probe in the first sample beaker so that it is approximately ½" into the extraction solvent. Turn on the Tissuemizer. Adjust the speed on the motor until the solvent begins to vortex in the beaker, but does not splash out of the beaker. Extract the sample for 3 minutes. During extraction move the beaker in a circular motion to ensure that the entire sample is subject to extraction. Remove the beaker and decant the extraction solvent into the sample's corresponding funnel and K-D apparatus. Repeat the extraction 2 more times with ~100 mL of extraction solvent. After the 3rd extraction, pour the entire contents of the beaker into the funnel, rinse the beaker with more of the extraction solvent, and pour this into the funnel as well.

Rinse the funnel with ~30 mL of extraction solvent and allow the solvent to completely drain into the K-D apparatus. Remove the funnel from the K-D apparatus and discard the contents of the funnel. Clean the generator probe and repeat the extraction for each sample.

Concentrate the extracts following the procedure given in section 10.5 in preparation for percent lipids determination and extract cleanup. After concentration and prior to extract cleanup, set aside a 1 mL aliquot of the concentrated extract and determine the percent lipids following procedures given in laboratory SOP BR-EX-016 *Percent Lipids Determination.*

Perform extract cleanup as appropriate following procedures given in laboratory SOPs BR-EX-002 and BR-EX-011. Refer to extraction condition spreadsheet for details. After cleanup, concentrate the extracts following the procedure given in section 10.5.

Enter the extraction data into the TALS. Assemble any associated paperwork and submit the extracts to the supervisor for a final project check. After review is complete, relinquish the extracts to the appropriate analytical department and place in the refrigerated storage area.

Note: Immediately following concentration, all sample extracts must be stored in a refrigerator maintained at a temperature of 4ºC (±2°C) in order to maintain thermal preservation.

10.5 Extract Concentration (KD Apparatus)

10.5.1 Macro Concentration

Macro Snyder Column (K-D)

Add one or two clean boiling chips to the K-D evaporation flask and attach a three-ball Snyder column to the flask. Add ~1 mL of methylene chloride to the top of the column then place the K-D apparatus in a hot water bath (60-70°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in hot water vapor.

Attach the solvent vapor recovery glassware to the Snyder column. Adjust the vertical position of the apparatus and check the water bath temperature. The water bath temperature should be between 54.8 – 74.8°C when methylene chloride is the extraction solvent and 84-89°C when hexane is the extraction solvent. Higher water bath temperatures may be used so long as the recovery of target analytes is not impacted. The boiling point of each solvent is provided in the following table:

Solvent	Boiling Point	Water Bath Temperature
Hexane	69°C	84 – 89°C
Methylene Chloride	39.8°C	54.8 – 74.8°C

Monitor the concentration and do not let the extract evaporate to dryness. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with solvent.

When the apparent volume of the extract reaches desired amount remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

Micro Synder Column (K-D)

Add one or two clean boiling chips to the concentrator tube and attach a two ball micro-Snyder column to the tube. Place the concentrator tube into the water bath so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the concentrator tube and check the temperature of the water bath to ensure the proper temperature for the extract solvent.

Continuously monitor the distillation process to ensure sample extracts do not evaporate to dryness. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with solvent. Remove setup when desired sample volume is reached.

Nitrogen Blowdown

Nitrogen blow down may be used to concentrate extracts as needed.

Place the concentrator tube in a warm water bath maintained at a temperature of 35°C. Apply a steady stream of nitrogen until the desired final extract volume is achieved. Rinse the internal wall of the concentrator tube several times with the appropriate solvent during the evaporation and ensure the solvent level in the concentrator is positioned such to prevent water condensations. Monitor the concentration carefully and do not allow the extract to evaporate to dryness.

11.0 Calculations / Data Reduction

11.1 Data Review

11.1.1 Primary Review

Review the TALS worksheet for correctness and completeness. Record any problems encountered during the extraction process into TALS or complete a NCM, when necessary. Set aside the extracts and paperwork for secondary review.

11.1.2 Secondary Review

Review the TALS worksheet against the extraction conditions spreadsheet and/or project notes to ensure the extraction performed is consistent with project specifications. Authorize release of the extracts to the appropriate analytical department.

For additional guidance regarding the laboratory's protocol and required elements for data review refer to laboratory SOP BR-QA -019 *Data Review*.

12.0 Method Performance

12.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ)

Refer to the SOP for the test method for requirements for determination of LOD and LOQ. These procedures are not included in sample preparation SOPs.

12.2 Demonstration of Capabilities (DOC)

Each analyst must complete an Initial Demonstration of Capability prior to unsupervised performance of this method.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 *Hazardous Waste*. The following waste streams are produced when this method is carried out.

- Organic Solvents - Satellite container: 55 gallon covered and vented drum.
- Vials containing extracts - Satellite container: 5 gallon covered bucket in fume hood.

- Methylene Chloride-Waste-Satellite Container: 55 Gallon Waste Drum
- Sulfuric Acid Waste-Satellite Container: 2.5L Waste Bottle Labeled with appropriate acid type (sulfuric).
- Solid Waste-Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood)

15.0 References / Cross-References

- Comprehensive Descriptions of Trace Organic Analytical Methods given in the Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, National Status and Trends Program for Marine Environmental Quality.

- GERG Trace Organic Contaminant Analytical Techniques published in NOAA Technical Memorandum NOS Orca 71, Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, Comprehensive Descriptions of Trace Organic Analytical Methods, July 1993.

- CW-E-M-001 *Corporate Environmental Health and Safety Manual*
- BR-EX-016 *Percent Lipid Determination*
- BR-EX-017 *Glassware Cleaning*
- BR-EX-002 *Extract Cleanup Procedure*
- BR-EX-011 *Gel-Permiation Cleanup*
- BR-QA -019 *Data Review*
- BR-QA-005 *Determination of LOD, LOQ, & RLs*
- BR-EH-001 *Hazardous Waste*

16.0 Method Modifications

Not applicable.

17.0 Attachments

- Table 1: Primary Materials Used
- Appendix A: Terms and Definitions

18.0 Revision History

Revision 5, Effective Date 5/20/08:
- Title Page: Updated approval signatures.
- Section 6.0: Inserted vendor information
- Section 8.0: Inserted table
- Section 15.0: Added cross referenced methods with the SOP
- All Sections: Fixed typographical errors

Revision 6, Effective Date 05/20/10:
- Title Page: Updated approval signatures
- Section 6.1: Addition of Meat Grinder to equipment list
- Section 10.1: Changed pipette calibrations to be done quarterly
- All Sections: Fixed typographical errors
- All Sections: Changed benchsheets reference to extraction condition spreadsheet
- All Sections: Changed LIMS references to TALS

Revision 7, Effective Date 08/01/12

- Section 8.0: Updated Holding Time requirements
- Section 9.1: Changed frequency of MS/MSD to client request

Table 1: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

[1] *Always add acid to water to prevent violent reactions.*
[2] *Exposure limit refers to the OSHA regulatory exposure limit.*

Appendix A: Terms and Definitions

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

SOP Change in Progress Attachment (CIPA)

SOP Number	SOP Title	SOP Revision	SOP Effective Date	CIPA Effective Date
BR-EX-027	Soxtherm Extraction (SW-846 3541)	0	11/10/10	10/01/12

The following revisions were made to this standard operating procedure (SOP). These changes are effective as of the CIPA Effective Date. This change to this document is authorized by the laboratory's QA Department.

Page 7 of 12, Section 10.5 Extraction Concentration (KD-Apparatus):

Remove the phrase (KD-Apparatus) from header 10.4 so that the text is:

10.5 Extraction Concentration

Insert the following text between 10.5 and 10.5.1:

The following sections describe the procedures for the concentration of extracts. Any of the three techniques described may be used. However some techniques are more efficient than others to achieve the final extract volume. Use the following guidelines to select the concentration technique: For the concentration of volumes greater than 5 mL, use Macro Concentration. To concentrate extract volumes between 5 and 1 mL use Micro Concentration. To concentrate extract volumes below 1 mL, use Nitrogen Blowdown.

Change the text in the header for 10.5.1 from Micro Snyder Column Concentration to Macro Snyder Column Concentration.

10.5.1 Macro Snyder Column Concentration

Title: AUTOMATED SOXHLET EXTRACTION (SW-846 3541)

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Approval Date: November 10, 2010

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1.0 Scope and Application

This SOP describes the laboratory procedure for automated soxhlet extraction of organic analytes from soils, sediment and waste solids and the subsequent concentration of the extracts in preparation for analysis by GC or GC/MS.

1.1 Analytes, Matrix(s), and Reporting Limits

Refer to analytical methods for analyte lists and reporting limits.

2.0 Summary of Method

A portion of sample is dried with anhydrous sodium sulfate, placed in an extraction thimble and extracted with an appropriate solvent for 3 hours. Following extraction the extract is concentrated using a Kuderna-Danish (K-D) apparatus to an appropriate final volume in preparation for cleanup and/or determinative analysis.

This procedure is based on the following reference method:

SW-846 Method 3541, Automated Soxhlet Extraction, Revision 0, 1994. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Third Edition, September 1986.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment. These contaminants can lead to elevated baselines and discrete artifacts that appear in gas chromatograms. These materials are demonstrated to be free of interferences by the analysis of method blanks. Interferences from phthalate esters can also pose a problem for the chlorinated pesticides analysis. To avoid this type of interference, the use of plastics in the laboratory must be minimized.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens:benzo(a)anthracene,benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC,beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds should be prepared in hood.

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The KD apparatus has ground glass joints which can become stuck. Technicians must use Kevlar or other cut/puncture resistant gloves when separating stuck joints.

5.2 Primary Materials Used

Table 1 lists those materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Extraction Equipment

- Automated Soxhlet Extraction System: Gerhardt SX001-6 place extraction unit or equivalent.
- 54 mm Glass beaker, AMK Glass or equivalent.
- 45 mm Extraction Thimble w/holder, AMK Glass or equivalent.
- Water Chiller, VWR or equivalent.
- Ultra-High Purity Nitrogen.

6.2 Extract Concentration (KD Apparatus)

- Concentrator tube, 10 mL graduated. ChemGlass Catalog Number CG-1316-11 or equivalent
- Snyder Column, Three ball macro AMK Catalog Number SC2-01 or equivalent
- Snyder Column, Two ball micro AMK Catalog Number SC3-01 or equivalent
- Evaporation Flask, 500 mL attached to concentrator tube with clip. AMK Catalog Number KDF-500 or equivalent
- Boiling Chips, silicon carbide, approximately 10/40 mesh, solvent extracted in methylene chloride. Troemner Catalog Number 133B or equivalent.
- Heating mantle rheostat controlled for water bath capable of temperature control (± 5°C). ChemGlass Catalog Number PL3122 or equivalent.
- Water Bath, capable of temperature control to ±5°C. Barnstead Corporation Catalog Number HM0500-HS1 or equivalent.
- Solvent Vapor Recovery System, Kontes K-54000-1006, K-547300-000, Ace Glass Catalog Number 6614-30 or equivalent.

6.3 Miscellaneous

- Disposable glass Pasteur pipette and bulb.
- Top Loading balance: Capable of measuring to 0.01 gram accuracy. Mettler Model Number PM4800 or equivalent.
- Teflon and Stainless Steel Spatulas
- Adjustable pipette, capable of measuring 200 uL to 5 mL, Finnpipette or equivalent.
- 0.5 mL – 2.0 mL Hamilton Gastight® syringes or equivalent.
- 2, 4, 8, or 16 mL Teflon lined screw vial Fisher Scientific or equivalent

7.0 Reagents and Standards

7.1 Reagents

- Sodium Sulfate (granular, anhydrous), Na_2SO_4. J.T. Baker or equivalent. Purify by heating at 400°C for at least 4 hours
- Methylene Chloride (CH_2C_{12}), Pesticide quality, J.T Baker or equivalent.
- Hexane, (C_6H_{14}), Pesticide Quality. J.T. Baker or equivalent.
- Acetone, ($(CH_3)_2CO$), Pesticide quality. J.T. Baker or equivalent.

7.2 Prepared Reagents

Methylene Chloride/Acetone (1:1): In a 4 L amber glass bottle mix 2 L methylene chloride with 2 L acetone. Store the solution in a fume hood.

Hexane /Acetone (1:1): In a 4 L amber glass bottle mix 2 L hexane with 2 L acetone. Store the solution in a fume hood.

7.3 Standards

Purchase stock standards as certified solutions from commercial vendors. Prepare surrogate and spiking solutions in the laboratory by diluting a known volume of the stock standard solutions in an appropriate solvent. Record the preparation of standard in the LIMS (TALS) module established for this purpose.

Store prepared standard solutions in glass containers at 4°C or below. Unless otherwise specified, assign an expiration date of 6 months from the date of preparation or in accordance with the expiration date of the parent standard, whichever is sooner. The recommended formulation for each standard used in this procedure is provided in the analytical method along with the recommended source materials, expiration dates and storage conditions.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time[1]	Reference
Solid	Glass with PTFE lined lid	50g	4°C	Extraction: 14 days	SW-846 3540C
Tissue	Glass with PTFE lined lid	50g	-10°C	Extraction: 14 days	SW-846 3540C

[1]Extraction holding time is determined from sampling date.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Analytical SOP
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Analytical SOP
Matrix Spike(s) MS/MSD	Client Request	See Analytical SOP
Sample Duplicate (SD)	Client Request	See Analytical SOP

9.2 Instrument QC

For information regarding instrument QC refer to the analytical SOP for the determinative test method.

10.0 Procedure

10.1 Instrument Calibration

Check the calibration of the balance each day of use prior to use and record these checks in the logbook designated for this purpose.

Check the calibration of any mechanical pipettes quarterly in accordance with the procedures specified in laboratory SOP BR-QA-008.

10.2 Glassware & Equipment Set-Up

Prepare glassware using the procedures described in laboratory SOP BR-EX-017 and rinse with acetone and methylene chloride prior to use. Label the glassware for each field and QC sample clearly and unambiguously during each step of the extraction procedure. Solvent will erase grease pens and “sharpie ink”; so use caution to ensure labels are not obliterated during the procedure.

Turn on the water chiller to the soxtherm unit. Check to ensure there is sufficient water in the unit and refill if necessary.

Start the flow of ultra-high purity nitrogen to the extraction units and set the pressure to 70 psi. Check the pressure gauge of the nitrogen cylinder and replace the tank if the pressure reading is less than 500 psi.

Initiate the following cleaning program using 120 mL of the 1:1 acetone/methylene chloride solution then discard the solution to the appropriate waste collection vessel after use.

Pre-Cleaning Operating Conditions (Program #2)

Temp Max:	150°C
Boiling Time:	20 Minute
Solvent Reduction A:	2 X 15 mL
Extraction Time:	20 Minute
Solvent Reduction B:	1X15 mL
Cool Time:	10 Minute
Total Process Time:	1 hours and 3 minutes.

The recommended operating conditions for the soxtherm units are:

Operating Conditions (Program #1)

Temp Max:	150°C
Boiling Time:	55 Minute
Solvent Reduction A:	5 X 15 mL
Extraction Time:	55 Minute
Solvent Reduction B:	1X15 mL
Cool Time:	20 Minute
Total Process Time:	2 hours and 55 minutes.

10.3 Sample Preparation

Mix samples thoroughly following the procedures specified in laboratory SOP BR-QA-020. Do no decant water from sediment samples if a large amount of water is present, contact the Project Manager for further guidance.

Initiate an extraction batch in TALS module ADII.

Place a labeled beaker on the balance and tare the balance. Measure 15 g (+/- 0.05 g) into the tared beaker and upload the sample mass into the TALS worksheet. Repeat for each sample and any designated MS/MSD or sample duplicates. Use anhydrous granular sodium sulfate for the method blank and LCS.

Mix each sample with a sufficient amount of granular anhydrous sodium sulfate to ensure a free-flowing mixture then transfer the sample into an extraction thimble.

Add ~6-12 silicon carborundum chips to each extraction beaker then place the extraction thimble into an extraction beaker.

Pipette the proper type and volume of surrogate solution to the sample container (or graduated cylinder) and add the proper type and volume of spike solution to the sample container for the

MS/MSD and LCS. See the extraction conditions workbook for type, concentration and amount of surrogate and spike solution used for each test method.

Add 120 mL of extraction solvent to each extraction beaker. For GC methods use 1:1 acetone/hexane solution as the extraction solvent and for GC/MS, use 1:1 acetone/methylene chloride.

10.4 Extraction

Place the extraction beakers into the extraction unit to ensure a tight seal between the extraction beaker and extraction unit. Check to ensure the chilled water and nitrogen systems are on and operating properly.

Turn on the soxtherm controller, press number "1" on controller pad to select program 1 and depress the enter key twice to begin the extraction process.

Periodically check the extraction units and do not allow the solvent in the extraction beaker to evaporate to dryness. If the solvent volume becomes low, lift the beakers off the boiling plate by switching the pneumatic control switch and add an additional amount of solvent to assure sufficient solvent volume until the extraction cycle is complete.

When the extraction cycle is finished, remove the beakers from the extraction unit and allow them to cool. Shut the soxtherm and Chiller "off" by depressing the "on" button. Shut the nitrogen off by turning the gas regulator counter clockwise.

Concentrate the extracts to an appropriate final volume.

10.5 Extract Concentration (KD Apparatus)

10.5.1 Micro Snyder Column Concentration

Transfer the extract to a 25 mL concentrator tube. Rinse the extraction beaker with 1~2 mL of the appropriate extraction solvent and transfer the rinsate to the concentrator tube to complete quantitative transfer.

Add one or two clean boiling chips to the K-D evaporation flask and attach a three-ball Snyder column to the flask. Add ~1 mL of methylene chloride to the top of the column then place the K-D apparatus in a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in hot water vapor. Attach the solvent vapor recovery glassware to the Snyder column. Adjust the vertical position of the apparatus and check the water bath temperature.

The water bath temperature should be between 54.8 – 74.8°C when methylene chloride is the extraction solvent and 84-89°C when hexane is the extraction solvent. Higher water bath temperatures may be used so long as the recovery of target analytes is not impacted. The boiling point of each solvent is provided in the following table:

Solvent	Boiling Point	Water Bath Temperature
Hexane	69°C	84 – 89°C
Methylene Chloride	39.8°C	54.8 – 74.8°C

Monitor the concentration and do not let the extract evaporate to dryness. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with solvent.

When the apparent volume of the extract is near the desired final volume remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. If samples require a solvent exchange add approximately 5-6 mL of the exchange solvent to the concentrator tube and return to the hot water bath for further concentration. Further concentrate the extract to final volume using micro-synder concentration or nitrogen blow down. Refer to the Extraction Conditions Spreadsheet for final extract volume for each test method, method.

Caution: *Do not allow extracts designated for 8270 PAH to concentrate to less than 1 mL in order to minimize volatilization of target compounds.*

Micro-Snyder Concentration: Add one or two clean boiling chips to the concentrator tube and attach a two ball micro-Snyder column to the tube. Place the concentrator tube into the water bath so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the concentrator tube and check the temperature of the water bath to ensure the proper temperature for the extract solvent.

Continuously monitor the distillation process to ensure sample extracts do not evaporate to dryness. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with solvent. Remove setup when desired sample volume is reached.

Nitrogen Blow Down: Place the concentrator tube in a warm water bath maintained at a temperature of 35°C. Apply a steady stream of nitrogen until the desired final extract volume is achieved. Rinse the internal wall of the concentrator tube several times with the appropriate solvent during the evaporation and ensure the solvent level in the concentrator is positioned such to prevent water condensations. Monitor the concentration carefully and do not allow the extract to evaporate to dryness.

10.6 Extract Preparation & Handling

For final extract volumes less than or equal to 1.0 mL adjust the extract volume using a graduated concentrator tube. For final extract volumes greater than 1.0 mL use a comparison vial. Transfer the extract to labeled Teflon lined screw cap via and store refrigerated. Complete the batch worksheet and perform primary review.

11.0 Calculations / Data Reduction

11.1 Calculations

Calculations are provided in the analytical SOP for each method parameter.

11.2 Data Review

Primary Review: Review the batch worksheet for correctness and completeness. Record any problems encountered during the extraction process with a nonconformance memo (NCM).

Secondary Review: Review the batch worksheet for correctness and completeness and to ensure the extraction performed is consistent the SOP and project specifications. Print the output worksheets and release extracts and output worksheet to the analytical department or to the next step in the method chain such as extract cleanup.

For additional guidance regarding the laboratory's protocol and required elements for data review refer to laboratory SOP BR-QA-019.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) Study must be determined for each test method associated with this extraction procedure during initial method set-up or prior to the analysis of field samples. The MDLs are verified annually or after major instrument maintenance. The procedure for the determination of MDLs is described in laboratory SOP BR-QA-005.

12.2 Demonstration of Capabilities (DOC)

Each analyst must complete an Initial Demonstration of Capability prior to unsupervised performance of this method.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 *Hazardous Waste*. The following waste streams are produced when this method is carried out.

- Solid Waste – Satellite Container: 5 Gallon Polyethylene Bucket
- Solvent Waste- Satellite Container: Steel 55 Gallon Drum

15.0 References / Cross-References

- SW-846 Method 3541, Automated Soxhlet Extraction, Revision 0, 1994. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Third Edition, September 1986.
- Laboratory SOP BR-EX-017 *Glassware Cleaning*
- Laboratory SOP BR-WC-006 *Percent Solids*
- Corporate Environmental Health and Safety Manual (CW-E-M-001)
- Laboratory SOP BR-QA-020 *Procedures for Sample Homogenization & Subsampling*
- Laboratory SOP BR-QA -019 *Data Review*
- Laboratory SOP BR-QA-005 *Determination of LOD, LOQ, & RL*

- Laboratory SOP BR-EH-001 *Hazardous Waste*
- Laboratory SOP BR-EX-002 *Extract Cleanup Procedures*

16.0 Method Modifications

None

17.0 Attachments

- Table 1: Primary Materials Used
- Appendix A: Terms and Definitions

18.0 Revision History

This is the first version of this SOP.

Table 1: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

[1] *Always add acid to water to prevent violent reactions.*
[2] *Exposure limit refers to the OSHA regulatory exposure limit.*

Appendix A: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

Title: Polychlorinated Biphenyls (PCBs) by GC/ECD (SW846 8082A)

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1.0 Scope and Application

This SOP describes the laboratory procedure used to determine the concentration of polychlorinated biphenyls (PCBs) as Aroclors using dual column gas chromatography with electron capture detectors (GC/ECD).

This SOP is applicable to instrument analysis only. Extraction and extract cleanup procedures are provided in separate SOPs.

1.1 Analytes, Matrices, and Reporting Limits

This procedure may be used for a variety of matrices including: water, soil, sediment and tissue.

The list of target compounds that can be determined from this method along with the associated reporting limits (RL) is provided in Table 1.

2.0 Summary of Method

2 uL of extract is injected into a dual capillary column gas chromatograph equipped with electron capture detectors (GC/ECD). The chromatographic data is used to determine the list of analytes provided in Table 1.

This SOP is based on the following reference method:

- SW-846 Method 8082A Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 0, February 2007.

If the laboratory procedure is modified from the above reference method, a list of modifications will be provided in Section 16.0 of this SOP.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

- Method interference may be caused by contaminants in the extraction solvent. Solvents should be stored away from organochlorine compounds to minimize contamination.

- Non-target compounds co-extracted from the sample matrix can also cause interference, the extent of which will vary depending on the nature of the samples. Elemental sulfur is often found in sediment samples and its presence will result in broad peaks. Samples are screened prior to analysis, and those samples that contain high levels of sulfur are subject to sulfur cleanup (SW-846 3660B). Cleanup procedures that may be used for this method include: GPC (SW-846-3640A), silica gel (SW-846 3630C), Florisil (SW-846 3620B), and Sulfuric acid Cleanup (SW-846 3665A).

- Phthalate esters introduced during sample preparation can pose a problem in the determination of target analytes. Common flexible plastics contain varying amounts of

phthalate esters. These phthalate esters can be easily extracted or leached during extraction. To minimize this interference, avoid contact with any plastic materials.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats, and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst must be aware of the locations of those zones and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off or disconnect it from its source of power.

5.2 Primary Materials Used

Table 2 lists materials used in this method which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Miscellaneous

- Autosampler Vials, National Scientific or equivalent.
- Hydrogen Generator: Parker Balston.
- Volumetric Syringes, Class “A” (10µl, 25µl, 50µl, 100µl, 250µl and 500µl), Hamilton or equivalent.

6.2 Analytical System

- Computer Hardware/Software: GC Acquisition Platform - VAX 4505 (GVAX) Multichrom V2.11. Data Processing - Hewlett-Packard 9000-series computers, an HP 9000 K200 (Chemsvr5)/ HP-UX 10.20 and Target V3.5 or higher.

- GC/ECD: with dual columns, dual ECDs, and auto-sampler capable of a 2-µl injection split onto two columns: HP 5890 with Leap Technology CTC A200SE and A200S Fisons autosamplers, Agilent Technologies 6890N with 7683 Series injector, or equivalent.
- GC Columns: A dual fused silica capillary column system that will provide simultaneous primary and confirmation analyses:

    - RTX-5, (30m x 0.25 mmID x 0.25um)
    - RTX-35, (30m x 0.25 mmID x 0.25um)

Equivalent columns may be used provided the elution orders are documented and compound separations are maintained.

7.0 Reagents and Standards

7.1 Reagents

- Hexane, Ultra-Resi Analyzed, JT Baker or equivalent.

7.2 Standards

Purchase stock standard solutions from commercial vendors and from these prepare calibration and working standards by diluting a known volume of stock standard in an appropriate solvent to the final volume needed to achieve the desired concentration. The recommended formulation for each standard used in this procedure is provided in Appendix B along with the recommended source materials, expiration dates and storage conditions.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection, so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method. Listed below are minimum sample size, preservation, and holding time requirements needed for this test.

Matrix	Sample Container	Minimum Sample Size	Preservation	Extract Holding Time	Reference
Water	Glass	1 L	Chilled to 4°C	40 Days	SW-846 8082A
Solid	Glass	50 g	Chilled to 4°C	40 Days	SW-846 8082A

[1]Analytical holding time is determined from date of initiation of extraction.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Table 3
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Table 3

Matrix Spike(s) MS/MSD	Client Request	See Table 3
Sample Duplicate (SD)	Client Request	See Table 3

9.2 Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initially; when ICV or CCV fail	See Table 3
Second Source Calibration Verification (ICV)	Once, after each ICAL	See Table 3
Continuing Calibration Verification (CCV)	Daily, every 10 samples, end of sequence	See Table 3
Retention Time Windows	As Needed	See Table 3

10.0 Procedure

10.1 Instrument Operating Conditions

Install a five meter deactivated guard column into the injection port and connect the guard column to the separate analytical columns using a glass "Y". The analytical columns are installed into independent ECD detectors.

The recommended instrument operating conditions are as follows:

Initial Temperature:	130°C for 1 minute Temperature Program: 20°C per minute to 190°C to 5°C per minute to 225°C to 20.0°C per minute to 300°C. Hold for 6 minutes.
Detector Temperature	300°C
Injector Temperature:	200°C
Injection volume:	2μL
Carrier Gas:	Hydrogen (supplied by hydrogen generators)

Optimize the flow rate of the carrier gas by injecting an un-retained substance onto the column at an isothermal oven state and adjusting the flow to obtain the recommended dead volume time.

10.2 Retention Time Window Establishment

Whenever a new GC column is installed, establish RT windows for each analyte by analyzing three standards over a 72-hour period. Calculate the mean RT and Standard Deviation (SD). The RT window is calculated as the mean RT ± 3SD. If the SD is <0.01 minutes, a default SD of 0.01 minutes may be used.

If this procedure results in RT windows that are too tight, favoring false negatives, the laboratory may opt to use an alternate method to determine the RT windows. An alternate method consists of using a RT window of ± 0.05 minutes. The center of the RT window is set at the midpoint calibration level in the initial calibration sequence. RT windows are then updated daily (minimum frequency), re-centering the windows on the retention times established in a CCV.

10.3 Instrument Calibration

10.3.1 Initial Calibration (ICAL)

Clean the injection port and column with a hexane instrument blank prior to calibration.

To calibrate the instrument analyze a standard containing a mixture of Aroclor 1016 and Aroclor1260 (AR1660) at a minimum of five concentrations and use this multi-point calibration to determine the concentration of AR1016 and AR1260 in sample.

The mixed AR1660 standard includes most of the peaks represented in the other Aroclors so the multi-point calibration can also be used to demonstrate linearity of the instrument and that a sample does not contain peaks that represent the other Aroclors but it is not sufficient for pattern recognition. For the remaining Aroclors analyze a single-point standard at a concentration near the mid-point of the calibration and use these standards for pattern recognition and calculation of a single-point calibration factor. The laboratory does not perform a multi-point calibration for the remaining Aroclors unless requested for the project or by regulatory requirement.

Prepare the calibration standards using the formulations provided in Appendix B then transfer ~100 ugL to an autosampler vial insert. Place the vials in the autosampler, set the autosampler to inject 2-µl of each standard onto the instrument and initiate the analytical sequence.

A minimum of 3 peaks must be chosen for each Aroclor, and preferably 5 peaks. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 5 peaks should include at least one peak that is unique to that Aroclor. Use at least five peaks for the Aroclor 1016/1260 mixture.

The data processing system calculates the Calibration Factor (CF), mean CF, and Percent Relative Standard Deviation (%RSD) for each analyte on both columns. The %RSD for each target analyte must be less than or equal to 20% in order to use the mean CF for quantification. This evaluation is performed for each quantitation peak chosen for each Aroclor. All peaks must pass the 20% evaluation, not the average of the 5 peaks chosen for quantitation. If this criterion is not met, use another suitable quantification method for that analyte or correct the problem and repeat the calibration. Once a method of quantification is chosen for a specific compound, it must be consistent throughout the entire analytical sequence until a new initial calibration is performed.

The calibration factor is used to determine the linearity of the calibration.

Alternate Quantification Option:

Linear Regression: Generate a curve of concentration vs. response for each analyte and calculate the correlation coefficient. The calibration must have a correlation coefficient (r) $\geq$ 0.995. If this criterion is not met, correct the problem and repeat the calibration. The use of linear regression requires a minimum of 5 calibration points.

10.3.2 Second Source Calibration Verification (ICV)

Immediately after each calibration and prior to the analysis of any QC or field samples, verify the accuracy of the initial calibration by analyzing a second source ICV.

Prepare the ICV using the formulation provided in Appendix B. Inject 2 µl of the ICV standard onto the instrument in the same manner as performed for the initial calibration standards.

The percent recovery of the average concentration of the peaks chosen for quantitation must be within ± 20% of the expected value (%R 80-120). If this criterion is not met, correct the problem and reanalyze the ICV. If reanalysis fails, remake the calibration standards and/or perform instrument maintenance and recalibrate. The acceptance criteria must be met on both columns.

10.3.3 Continuing Calibration Verification (CCV)

Analyze a CCV (1660) at or below the mid-calibration range each day before sample analysis, after every ten sample injections and at the end of each analytical sequence.

Note: The laboratory does not perform a CCV for the remaining Aroclors unless requested for the project or by regulatory requirement.

The data system calculates the calibration factor (CF) and percent difference using the average percent difference of the peaks chosen for quantitation.

The percent difference or drift must be within ±20% and the retention time (RT) must be within the established RT window. Acceptance criteria must be met on both columns.

If the CCV fails, it may be repeated once. If repeat analysis fails, corrective action must be taken. If the two CCVs do not meet the criteria, recalibration is required prior to running samples. Samples must be bracketed by passing CCVs. Samples analyzed before and after CCV failures must be reanalyzed, unless the CCV is high and there are no detects in the associated samples. (NELAC Requirement)

10.4 Troubleshooting

Check the following items in case of calibration failures:

- ICAL Failure – Perform injection port maintenance, install new guard column, check detector ends to see if detector jet has slipped. In extreme cases, install new columns, particularly if the chromatography has degraded as evidenced by peak shapes.
- CCV Failure – Perform Injection port maintenance; if injection port maintenance does not restore CCV, install a new guard column and remove one or more loops from each analytical column.
- Needle crushed during injection - Replace the needle and check the injection port for obstructions and check the autosampler for misalignment.
- Auto-sampler failure - Reset the auto-sampler.
- Power failure - Reset run in Multichrom and re-acquire or re-initiate run sequence.

10.5 Analysis

Remove the extract from refrigerated storage and warm to room temperature.

Transfer approximately 100 uL of extract to an autosampler vial and place the vials in the autosampler in a sequence that begins with the calibration standards followed by the analysis of an ICV, QC samples, field samples and continuing calibration verification standards (CCVs).

Enter the sample ID's into the data acquisition program in the order that the samples were placed in the autosampler tray and initiate the analytical sequence.

An example analytical sequence that includes calibration is as follows:

Injection Number	Lab Description
1	Instrument Blank
2	Instrument Blank
3	Instrument Blank
4	AR1221 (200 ppb)
5	AR1232 (200 ppb)
6	AR1242 (200 ppb)
7	AR1248 (200 ppb)
8	AR1254 (200 ppb)
9	AR1262 (200 ppb)
10	AR1268 (200 ppb)
11	AR1660 (50 ppb)
12	AR1660 (100 ppb)
13	AR1660 (200 ppb)
14	AR1660 (400 ppb)
15	AR1660 (800 ppb)
16	Instrument Blank
17	ICV
18-27	10 injections
28	CCV (AR1660 200ppb)
	Repeat steps 18-28

Cleaning blanks (IBLK) consisting of hexane may be analyzed after high-level samples at the discretion of the analyst.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

The data processing system identifies the target analytes by comparing the retention time of the peaks to the established retention time windows.

Review and accept or reject the qualitative identifications made by the data processing system using the following guidelines:

Compare the retention time of the peak to the established RT window, taking into account the shift of the surrogate peaks. If the surrogate peaks have shifted, open the retention time window in the direction of the shift. The processing system identifies the peak in the retention time window that is closest to the expected retention time set in the Target method, so the peak may need to be re-identified if a shift has occurred. The data system does not recognize Aroclor patterns. The analyst manually identifies Aroclors by comparing the pattern in the samples to the patterns in the initial calibration standards. Weathering of PCB's in the environment may alter the PCB's to the point that the pattern no longer matches the pattern established for that Aroclor in

the initial calibration. The laboratory takes the best pattern match approach to the identification and quantification of weathered PCB's.

Look for shoulders on the side of large peaks that may be peaks of interest. The processing system does not always automatically integrate shoulders from larger peaks, so manual integration (split) of the shoulder may be necessary.

Each target analyte must be detected on each column for qualitative identification to be made.

11.2 Quantitative Identification

Using an average of the chosen quantification peaks per Aroclor the data system calculates the corrected concentration for each target analyte using the equations given in Appendix C. If sample interference is suspected, the laboratory may remove up to two quantification peaks per column. The higher value between the two columns is reported as the primary result unless there is evidence of chromatographic anomalies, in which case the lower value will be reported. This deviation must be noted in the project narrative.

11.3 Calculations

See Appendix C.

11.4 Data Review

See laboratory SOP BR-QA-019 for data review requirements.

11.4.1 Primary Review

Review project documents to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Confirm qualitative and quantitative identification criteria using the criteria provided in Sections 11.1 and 11.2. If the data system does not properly integrate the peaks perform manual integration in accordance with laboratory SOP BR-QA-006.

Upload the data files from the data processing system to the laboratory information management system (TALS). Complete the batch information for standards and reagents and verify ICAL and QC sample associations. Review the results and set results to primary, secondary, acceptable or rejected as appropriate. Dilute and reanalyze samples whose results exceed the calibration range. The dilution analysis should result in a determination within the calibration range, preferably in the upper half of the calibration range. A more concentrated analysis is not necessary unless the project requires it. Dilution analyses may be performed to minimize matrix interference.

If a sample was analyzed immediately following a high concentration sample, review the results of the sample for any sign of carryover. If carryover is suspected, reanalyze the sample.

Create a non-conformance report (NCM) for any calibration, QC and sample data that is reported outside established acceptance criteria and/or schedule necessary corrective action. Set batch to 1st level review and complete the data review checklist.

11.4.2 Secondary Data Review

Verify quantitative and qualitative identification in the initial calibration standards and spot check such for ~15% of the remaining data in the batch.

If manual integrations were performed:

- Review each integration to verify that the integration meets the requirements for manual integration as specified in laboratory SOP BR-QA-006. If an error is suspected or found consult with the analyst that performed the integration analyst and request correction or notify the Department Manager, Technical Director or QA Manager. Do not "fix" the integration. Reintegration by a secondary data reviewer must not be performed except in limited circumstances as approved by the department supervisor or other laboratory management. If those instances where the secondary reviewer performs the integration, this person is now considered the primary analyst and each integration performed by the secondary reviewer must be subsequently reviewed by a peer analyst or the department supervisor to verify the integration is consistent and compliant with the requirements specified in laboratory SOP BR-QA-006.

- Check to ensure an appropriate technical reason code is provided for each manual integration. Acceptable technical reason codes are provided in laboratory SOP BR-QA-005.

Review project documents to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Verify that the acceptance criteria for the calibration and QC items listed in Table 1 were met. If the results do not fall within the established limits verify the recommended corrective actions were performed. If not, initiate corrective actions and/or verify an NCM was created to document the criteria exception. Verify analytical results are qualified accordingly. Set batch to 2nd level review and complete the data review checklist.

11.5 Data Reporting

The report format, application of data qualifiers and creation of the data deliverable is performed by the LIMS using the formatter set by the project manager during log-in.

Records of electronic and hardcopy data are maintained as described in laboratory SOP BR-QA-014.

12.0 Method Performance

12.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ)

Establish a LOD and LOQ at initial method set up following the procedures specified in laboratory SOP BR-QA-005. Verify the LOD and LOQ at the frequency established for the method using the procedures specified in same SOP. The frequency of LOD and LOQ verification depends on the strictest frequency of the regulatory program for which the method supports. The frequency requirement is documented in a spreadsheet maintained by the QA Department.

12.2 Demonstration of Capabilities (DOC)

Perform a method demonstration of capability at initial set-up and when there is a significant change in instrumentation or procedure.

Each analyst that performs the analytical procedure must complete an initial demonstration of capability (IDOC) prior to independent analysis of client samples. Each analyst must demonstrate on-going proficiency (ODOC) annually thereafter. DOC procedures are further described in the laboratory's quality system manual (QAM) and in the laboratory SOP for employee training.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of the SOP.

Instrument analysts, prior to independent analysis of client samples, must also have documentation of demonstration of initial proficiency (IDOC) and annual on-going proficiency (ODOC) in their employee training files.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Vials containing sample extracts: Satellite container: 15 gallon bucket connected to a fume hood.
- Solvent Waste: Satellite container: 1 L glass bottle located in fume hood.

15.0 References / Cross-References

- SW-846 Method 8082A Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 0, February 2007.
- Corporate Environmental Health and Safety Manual (CW-E-M-001)
- Laboratory SOP BR-QA-011
- Laboratory SOP BR-LP-011
- Laboratory SOP BR-QA-014
- Laboratory SOP BR-QA-006
- Laboratory SOP BR-QA-005

16.0 Method Modifications

Not applicable.

17.0 Attachments

- Table 1: Target Compound List and Reporting Limit
- Table 1A: Accuracy and Precision Limits
- Table 2: Primary Materials Used
- Table 3: QC Summary & Recommended Corrective Action
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Tables
- Appendix C: Equations

18.0 Revision History

BR-GC-005, Rev. 11:

- Title Page: Updated method reference
- Section 2.0: Updated method reference
- Section 10.3: Changed CCV criteria from 15% to 20%
- Table 3: Changed CCV criteria from 15% to 20%

BR-GC-005, Rev. 10:

- Updated approval signatures
- Section 10: Inserted note regarding multi-point calibrations for other Aroclors.

BR-GC-005, Rev. 9

- Updated reference method in Section 2.0.
- Changed QC criteria for %D from 15% to 20%.
- Added language to Section 10.2 to allow for updating RT windows using CCVs.
- Added language to Section 11.4.1 to allow for dilution to minimize matrix interference.
- Added standard preparation tables to Appendix B to allow for the preparation of 5 point calibrations for each of the Aroclors

Table 1: Routine Target Analyte List & Reporting Limits (RL)

ANALYTE	Routine Reporting Limit (RL)[1,2]	
	Water (ug/L)	Solid (ug/Kg)
AR1016	0.50	17
AR1221	0.50	17
AR1232	0.50	17
AR1242	0.50	17
AR1248	0.50	17
AR1254	0.50	17
AR1260	0.50	17
AR1262	0.50	17
AR1268	0.50	17

[1]*The routine RL is the unadjusted value that can be achieved in a blank matrix.*
[2]*The RL for tissue matrix is project defined.*

Table 1A: Routine Accuracy and Precision Limits[1]

Analyte	In-House Limits (%R)		Precision (RPD) (≤)
	Water	Solid	
AR1016	55-120	55-120	30
AR1260	60-125	55-125	30
Surrogate: Decachlorobiphenyl (DCB)	30-150	45-125	NA
Surrogate:TCX (Advisory)[2]	55-120	30-130	NA

[1] *The limits in this table are those used as of the effective date of this SOP. Current limits are stored in the LIMS database.*
[2] *The control limits for TCX are advisory. Corrective action is not performed when recovery is outside limits.*

Table 2: Primary Materials Used

Material [1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.

[1] *Always add acid to water to prevent violent reactions.*
[2] *Exposure limit refers to the OSHA regulatory exposure limit.*

Table 3: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	Option 1: RSD for each analyte ≤ 20% Option 2: Linear Regression: r ≥ 0.995	Correct problem, reanalyze, repeat calibration.
ICV	After each initial calibration	(% R) ± 20% from expected value	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Daily before sample analysis, every 10 samples and at the end of the analytical sequence	% Difference or Drift ±20%	See Section 10.3
MB	One per extraction batch of 20 or fewer samples	Target Analyte < RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer samples	See Table 1A	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD SD	Per client request	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate	All field and QC samples	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.

[1]*The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.*

Appendix A: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

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.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are only produced in this range and qualified with the proper data reporting flag when a project requires this type of data reporting.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

Appendix B: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration of the stock standard is different than those noted in this table, adjust the standard preparation formulation accordingly. Unless otherwise specified, prepare the standard solutions in hexane using Class A volumetric glassware and Hamilton syringes. Unless otherwise specified for a standard solution, assign an expiration date of 6 months from date of preparation unless the parent standard expires sooner in which case use the earliest expiration date. Store the prepared solutions under refrigeration and protected from light at a temperature of 4ºC (±2). See laboratory SOP BR-QA-002 *Standard Preparation* for further guidance.

Intermediate Calibration Standards (10 mg/L)

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
AR1660[1]	Restek #32039	Aroclor 1016 Aroclor 1260	1000	0.40	40	10
AR1254	Restek #32011	Aroclor 1254	1000	0.40	40	10
AR1248	Restek #32010	Aroclor 1248	1000	0.40	40	10
AR1242	Restek #32009	Aroclor 1242	1000	0.40	40	10
AR1232	Restek #32008	Aroclor 1232	1000	0.40	40	10
AR1221	Restek #32007	Aroclor 1221	1000	0.40	40	10
AR1262	Restek #32409	Aroclor 1262	1000	0.40	40	10
AR1268	Restek #32410	Aroclor 1268	1000	0.40	40	10

[1] *Standard is a mix of AR1016/AR1260. Concentration shown is the concentration of each Aroclor in the mixed standard.*

Intermediate ICV Standard (10 mg/L)

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
AR1660	Ultra Scientific PPM8082	Aroclor 1016 Aroclor 1260	1000	0.40	40	10

Surrogate Solution (10 mg/L)

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Pesticide Surrogate	Restek #3200	TCX DCB	1000	0.40	40	10

Working ICV Standard (200 ug/L)

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
Intermediate ICV	Laboratory Prepared	Aroclor 1016 Aroclor 1260	10	0.80	40	200
Surrogate	Laboratory Prepared	TCX DCB	10	0.080		20

AR1660 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1660 Intermediate	Laboratory Prepared	Aroclor 1016 Aroclor 1260	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1660 calibration standards*

AR1660 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1660 Level 5	AR1660 CAL Level 4	800	20	40	400
AR1660 Level 5	AR1660 CAL Level 3	800	10	40	200
AR1660 Level 5	AR1660 CAL Level 2	800	5.0	40	100
AR1660 Level 5	AR1660 CAL Level 1	800	2.5	40	50

AR1221 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1221 Intermediate	Laboratory Prepared	Aroclor 1221	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1221 calibration standards*

AR1221 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1221 Level 5	AR1221 CAL Level 4	800	20	40	400
AR1221 Level 5	AR1221 CAL Level 3	800	10	40	200
AR1221 Level 5	AR1221 CAL Level 2	800	5.0	40	100
AR1221 Level 5	AR1221 CAL Level 1	800	2.5	40	50

AR1232 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1232 Intermediate	Laboratory Prepared	Aroclor 1232	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1232 calibration standards*

AR1232 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1232 Level 5	AR1232 CAL Level 4	800	20	40	400
AR1232 Level 5	AR1232 CAL Level 3	800	10	40	200
AR1232 Level 5	AR1232 CAL Level 2	800	5.0	40	100
AR1232 Level 5	AR1232 CAL Level 1	800	2.5	40	50

AR1242 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1242 Intermediate	Laboratory Prepared	Aroclor 1242	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1242 calibration standards*

AR1242 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1242 Level 5	AR1242 CAL Level 4	800	20	40	400
AR1242 Level 5	AR1242 CAL Level 3	800	10	40	200
AR1242 Level 5	AR1242 CAL Level 2	800	5.0	40	100
AR1242 Level 5	AR1242 CAL Level 1	800	2.5	40	50

AR1248 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1248 Intermediate	Laboratory Prepared	Aroclor 1248	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1248 calibration standards*

AR1248 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1248 Level 5	AR1248 CAL Level 4	800	20	40	400
AR1248 Level 5	AR1248 CAL Level 3	800	10	40	200
AR1248 Level 5	AR1248 CAL Level 2	800	5.0	40	100
AR1248 Level 5	AR1248 CAL Level 1	800	2.5	40	50

AR1254 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1254 Intermediate	Laboratory Prepared	Aroclor 1254	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1254 calibration standards*

AR1254 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1254 Level 5	AR1254 CAL Level 4	800	20	40	400
AR1254 Level 5	AR1254 CAL Level 3	800	10	40	200
AR1254 Level 5	AR1254 CAL Level 2	800	5.0	40	100
AR1254 Level 5	AR1254 CAL Level 1	800	2.5	40	50

AR1262 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1262 Intermediate	Laboratory Prepared	Aroclor 1262	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1262 calibration standards*

AR1262 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1262 Level 5	AR1262 CAL Level 4	800	20	40	400
AR1262 Level 5	AR1262 CAL Level 3	800	10	40	200
AR1262 Level 5	AR1262 CAL Level 2	800	5.0	40	100
AR1262 Level 5	AR1262 CAL Level 1	800	2.5	40	50

AR1268 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1268 Intermediate	Laboratory Prepared	Aroclor 1268	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1268 calibration standards*

AR1268 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1268 Level 5	AR1268 CAL Level 4	800	20	40	400
AR1268 Level 5	AR1268 CAL Level 3	800	10	40	200
AR1268 Level 5	AR1268 CAL Level 2	800	5.0	40	100
AR1268 Level 5	AR1268 CAL Level 1	800	2.5	40	50

Appendix C: Equations

Calibration Factor (CF_x) = $\frac{\text{Peak area or height}_{(x)}}{\text{Standard concentration}_{(ug/L)}}$

Mean Calibration Factor ($\overline{CF}$) = $\frac{\sum_{i=1}^{n} CF_i}{n}$

where: n = number of calibration levels

Standard Deviation of the Calibration Factor (SD) = $\sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)2}{n-1}}$

where: n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Calibration Factor =
$\frac{SD}{\overline{CF}} \times 100\%$

Percent Difference (%D) = $\frac{CF_v - \overline{CF}}{\overline{CF}} \times 100\%$ Add absolute value signs

where: CF_v = Calibration Factor from the Continuing Calibration Verification (CCV)

Percent Drift = $\frac{\text{Calculated Concentration – Theoretical Concentration}}{\text{Theoretical Concentration}}$ X 100%

Percent Recovery (%R) = $\frac{C_s}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD = $\frac{C_s - C_u}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (RPD) = $\dfrac{|C_1 - C_2|}{\left(\dfrac{C_1 + C_2}{2}\right)} \times 100\%$

where: C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration

Extract

$C_{extract}\,(ug/L) = \dfrac{\text{Peak Area (or Height)}}{\overline{CF}}$

Note: The concentrations of the 3-5 peaks chosen for quantification is calculated and the average is then taken for final calculation.

Water

$$C_{sample}(ug/L) = C_{extract}(ug/L) \times \frac{\text{extract volume (L)}}{\text{sample volume (L)}} \times DF$$

Solid

$$C_{sample}(ug/Kg) = C_{extract}(ug/L) \times \frac{\text{extract volume (L)}}{\text{sample weight (Kg)}} \times \frac{100}{\%\,\text{solids}} \times DF$$

Quality Assurance Manual

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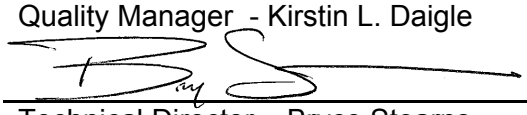
Title Page:

Quality Assurance Manual
Approval Signatures

	October 25, 2011
Laboratory Director – William S. Cicero	
	October 24, 2011
Quality Manager - Kirstin L. Daigle	
	October 24, 2011
Technical Director – Bryce Stearns	

SECTION 2. TABLE OF CONTENTS

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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	Method Compliance & Data Authenticity Audits
CA-Q-S-006	Detection Limits
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)
CW-L-S-002	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CW-L-P-004	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	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-007	Controlled Purchases Policy
CW-F-S-018	Vendor Selection
CA-Q-M-002	Corporate Quality Management Plan
CW-E-M-001	Corporate Environmental Health & Safety Manual

REFERENCED LABORATORY SOPs

SOP Reference	Title
BR-QA-003	Document Control
BR-QA-004	Complaint Resolution
BR-QA-011	Employee Training and Demonstration of Proficiency
BR-QA-005	Detection Limits, Limit of Detection and Limit of Quantitation
BR-QA-006	Manual Integration
BR-QA-020	Sample Homogenization and Subsampling
BR-SM-001	Sample Management

SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

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

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

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

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.*
- *U.S. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 4.2, October 2010.*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005) (DW labs only)*
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th, 21st, and on-line Editions.
- *U.S. Department of Energy* Order 414.1B, *Quality Assurance*, Approved April 29, 2004.
- *U.S. Department of Energy* Order 414.1C, *Quality Assurance*, June 17, 2005.
- *U.S. Department of Energy*, *Quality Systems for Analytical Services*, Revision 3.6, November 2010.
- U.S. Department of Defense, *Air Force Center for Environmental Excellence Quality Assurance Project Plan (QAPP),* Version 4.0.02, May 2006.
- Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.
- Marine Protection, Research, and Sanctuaries Act (MPRSA).
- Toxic Substances Control Act (TSCA).

3.2 Terms and Definitions

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

Refer to Appendix 2 for the Glossary/Acronyms.

3.3 Scope / Fields of Testing

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

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

3.4 Management of the Manual

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. The manual itself is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be

reviewed and approved by the senior laboratory management staff according to the laboratory's Document Control procedure (SOP No. BR-QA-003).

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

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

4.2 Roles and Responsibilities

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

4.2.1 Additional Requirements for Laboratories

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

4.2.2 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system.

The QA Manager reports directly to the Laboratory Director and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of QA staff to accomplish specific responsibilities, which include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.

- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Review a percentage of all final data reports for internal consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, 5% of calculations, format, holding time, sensibility and completeness of the project file contents.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule 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.
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- 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 12.
- Evaluation of the thoroughness and effectiveness of training.
- Compliance with ISO 17025. (where applicable)

4.2.3 Technical Manager (AKA Technical Director) & Department Manager (DM)

The Technical Director report(s) directly to the Laboratory Director. The Technical Director along with the Laboratory Director, the QA Manager and each Department Manager is accountable for compliance with the ISO 17025 Standard. The Technical Director works with QA and the Department Managers to solve day to day technical issues, provide technical training and guidance to laboratory staff, project managers, and clients, and assists with method development and validation.

The Department Managers report to the Laboratory Directory. The DMs maintain overall responsibility for a defined portion of the laboratory. These responsibilities include but are not limited to:

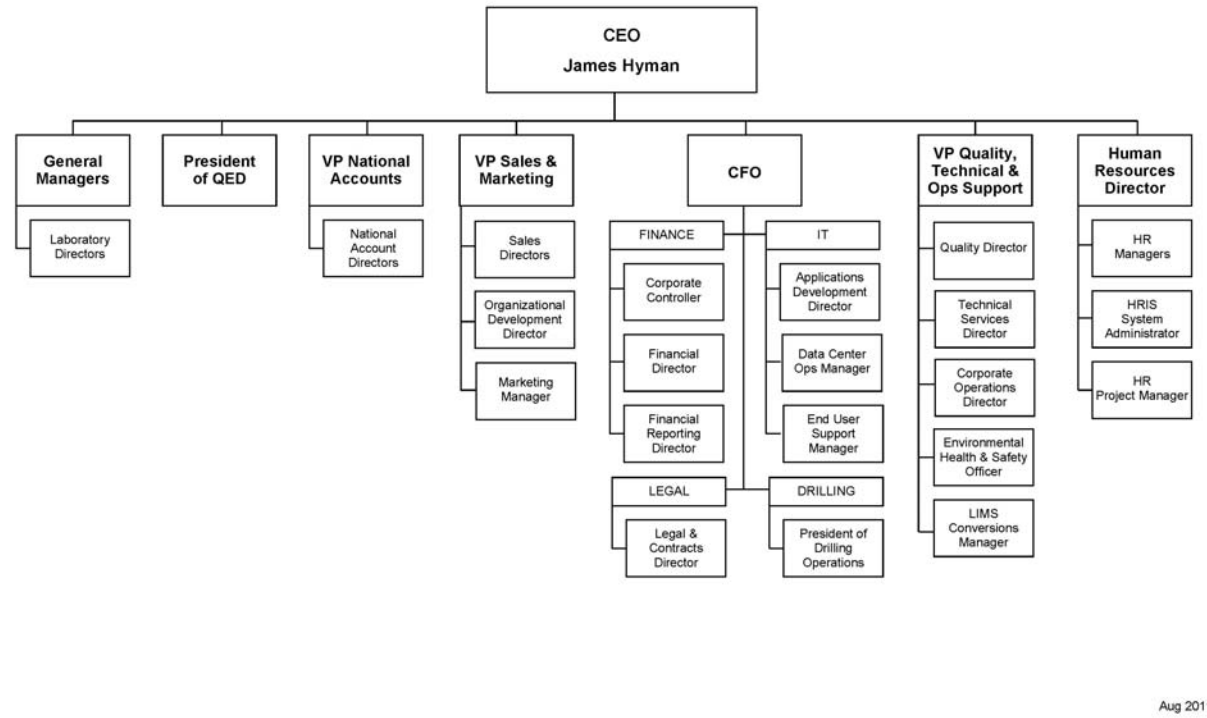
- Day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Working with the QA Manager to coordinate preparation of test method SOPs and performs subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples.
- Reviews and approves proposals from marketing, in accordance with the established procedure for the review of requests and contracts.
- Monitoring the validity of the analyses performed and data generated in the laboratory.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Working with the QA Manager to scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.

4.3 Deputies

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

Key Personnel	Deputy
William S. Cicero Laboratory Director	Bryce E. Stearns, Technical Director Kirstin L. Daigle, QA Manager
Kirstin L. Daigle QA Manager	William S. Cicero, Laboratory Director Bryce E. Stearns, Technical Director Frances S. Bertsch, QA Assistant
Bryce E. Stearns Technical Director	William S. Cicero, Laboratory Director Kirstin L. Daigle, QA Manager
Dan E. Helfrich EHS Coordinator	William S. Cicero, Laboratory Director

Figure 4-1. Corporate and Laboratory Organization Charts



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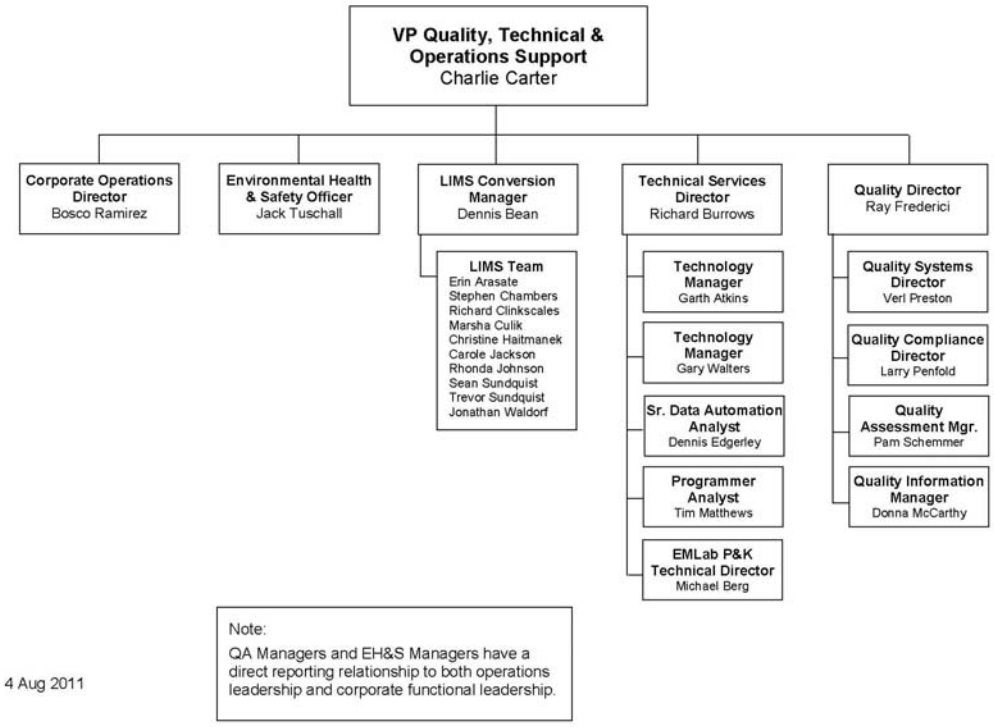
Quality, Technical & Operations Support



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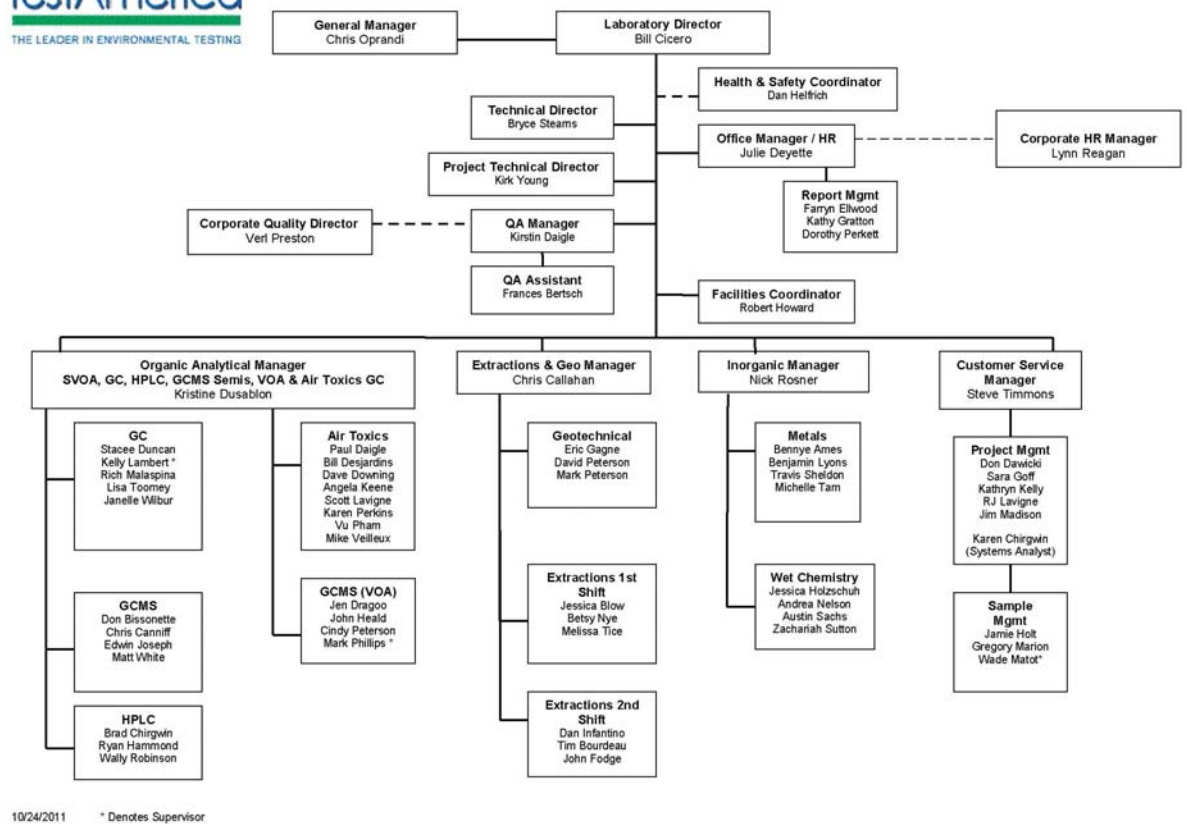
Burlington Laboratory Organization



SECTION 5. QUALITY SYSTEM

5.1 Quality Policy Statement

It is TestAmerica's Policy to:

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

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

5.2 Ethics and Data Integrity

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

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

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

5.3 Quality System Documentation

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

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

5.3.1 Order of Precedence

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

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

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

5.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately 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 their project QAPPs. The laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities. 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 it is finalized.

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

5.4.1 Precision

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

5.4.2 Accuracy

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

5.4.3 Representativeness

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

identical samples or sample aliquots.

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

5.4.4 Comparability

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

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

5.4.5 Completeness

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

5.4.6 Selectivity

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

5.4.7 Sensitivity

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

5.5 Criteria for Quality Indicators

The laboratory limits used quality control are stored in the LIMS database and may also be published in laboratory SOPs. Limits for accuracy and precision are laboratory generated or are

derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. The laboratory procedure for establishment of control limits is described in laboratory SOP BR-QA-013.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and by program. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. If a method requires the generation of limits from historical data the lab develops such limits from data stored in the LIMS database following the procedure specified in laboratory SOP BR-QA-013.

For each job analysts are instructed to use the current limits that are entered as reference data data in the Laboratory Information Management System (LIMS) On occasion, a client requests contract-specified limits for a specific project in which case project specific limits are entered into each LIMS job by the PM handling the project.

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

5.6.1 QC Charts

The laboratory's procedures for the creation of control charts are described in laboratory SOP BR-QA-013. Control charts are created from data stored in the LIMS. The charts are evaluated by QA or technical staff to determine if limits need to be updated or to assess the need for corrective actions to improve method performance.

5.7 Quality System Metrics

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

SECTION 6. DOCUMENT CONTROL

6.1 Overview

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

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies

- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP BR-QA-003.

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 records for 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 are kept by the QA department. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports are retained electronically, by each analytical section or by the QA department.

6.2 Document Approval and Issue

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

Controlled documents are authorized by the QA Department. To develop a new document, the department manager or any employee with approval from the department manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval QA personnel add the identifying version information to the document and retains a copy of the document as the official document on file. The document is then provided to all applicable operational units (may include electronic access) by either electronic or hardcopy distribution.

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

Quality System Policies and Procedures will be reviewed annually and revised as appropriate. Changes to documents occur when a procedural change warrants.

6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP BR-QA-003. Uncontrolled copies must not be used within the laboratory. Previous revisions are stored by the QA department. The current revision is located in the public controlled document folder accessible to all employees.

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure SOP.

Forms, worksheets, work instructions and information are organized by the QA department in accordance with the procedures specified in laboratory SOP BR-QA-003.

6.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP BR-QA-003.

SECTION 7. SERVICE TO THE CLIENT

7.1 Overview

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

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

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

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

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

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

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

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.
The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 Review Sequence and Key Personnel

Work requests are reviewed by appropriate personnel at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

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

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above. Appropriate personnel include but are not limited to:

- Legal & Contracts Director
- General Manager
- Laboratory Director
- Laboratory Project Manager
- Laboratory Technical Manager/Director
- Laboratory Department Manager
- Laboratory Customer Service Manager
- Information Technology Manager

- Account Executives
- Laboratory and/or Corporate Quality Managers
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements. The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility. The Project Manager, Sales Director, Legal Contracts Director, Account Executive or Proposal Coordinator then submits the final proposal to the client. The Legal & Contracts Director and facility Customer Service Manager 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. Records of review are organized and kept by the designated project manager (PM).

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM.

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. These records are retained by the laboratory PM.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project.

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

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

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

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

7.4 Special Services

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

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

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

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

7.5 Client Communication

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

Technical Managers and the Quality Assurance Manager are available to discuss any technical questions or concerns that the client may have.

7.6 Reporting

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

7.7 Client Surveys

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

SECTION 8. SUBCONTRACTING OF TESTS

8.1 Overview

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

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica's Corporate SOP's on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process (CA-C-S-001).

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

Project Managers (PMs), Customer Service Managers (CSM), or Account Executives (AE) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

Note: In addition to the client, some regulating agencies (e.g, USDA) or contracts (e.g, certain USACE projects) may require notification prior to placing such work**.**

8.2 Qualifying and Monitoring Subcontracators

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

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task. (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica: A listing of all approved subcontracting laboratories is available on the TestAmerica intranet site. Supporting documentation is maintained by corporate offices and by the TestAmerica laboratory originally requesting approval of the subcontract lab.

- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- NELAC or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

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

When the potential sub-contract laboratory has not been previously approved, management staff may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site and notify the finance group for JD Edwards.

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

8.2.3 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Laboratory Directors, 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 flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented and in the project folder. An example form that may be used for documentation is provided as Figure 8-1. For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

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

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

Non-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 laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

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

8.4 Contingency Planning

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs; however, this decision & justification must be documented in the

project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

Figure 8-1.

Example - Subcontracted Sample Form

Date/Time: _____

Subcontracted Laboratory Information:

- Subcontractor's Name: _____
- Subcontractor Point of Contact: _____
- Subcontractor's Address: _____
- Subcontractor's Phone: _____
- Analyte/Method: _____
- Certified for State of Origin: _____
- NELAC Certified: Yes_____No_____
- **USDA Permit (__Domestic __ Foreign)** **Yes**_____**No**_____
- ISO 17025 Certified: Yes_____No_____
- CLP-like Required: Yes_____No_____
  (Full doc required)
- Requested Sample Due Date: _____
  (Must be put on COC)
- **Client POC Approval on-file to Subcontract Samples to Sub Laboratory:** **Yes**_____**No**_____

Project Manager: _____

Laboratory Sample # Range: _____
(Only of Subcontracted Samples)

Laboratory Project Number (Billing Control #): _____

All subcontracted samples are to be sent via bonded carrier and Priority Overnight. Please attach tracking number below and maintain these records in the project files.

PM Signature_____**Date**_____

SECTION 9. PURCHASING SERVICES AND SUPPLIES

9.1 Overview

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

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

9.2 Glassware

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

9.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001.

9.3.1 Purchasing

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

9.3.2 Receiving

It is the responsibility of the manager that placed the order to receive the shipment. It is the responsibility of the manager or their designee who ordered the materials to document the date

materials where received. Once the ordered reagents or materials are received the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. 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 Specifications

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

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

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer's or SOPs expiration date unless 'verified' (refer to item 3 listed below).

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

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 120 psig for Helium, 100 psig for liquid Argon and 30 psig for Nitrogen or the tank must be replaced. To prevent a tank from going to dryness, close observation of the tank gauge must take place as pressure decreases towards the minimum 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 1- µmhom/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25^{o}C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical Managers must be notified

immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

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

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

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

Records of manufacturer's certification and traceability statements are maintained in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Manager, Technical Director or QA Manager.

9.3.4 Storage

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

9.4 Purchase of Equipment / Instruments / Software

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

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

9.5 Services

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

9.6 Suppliers

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

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

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

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.
As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

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

9.6.1 New Vendor Procedure

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

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

SECTION 10. COMPLAINTS

10.1 Overview

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

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

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

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

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following laboratory SOP BR-QA-004.

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to laboratory SOP BR-QA-004.

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

The general steps in the complaint handling process are:

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

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

10.3 Internal Complaints

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

10.4 Management Review

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

SECTION 11. CONTROL OF NON-CONFORMING WORK

11.1 Overview

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

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. Any modifications to the routine procedure are documented in the project record and described in the case narrative submitted with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Any project specific modifications to the procedure are documented in the project record.

11.2 Responsibilities and Authorities

TestAmerica's Corporate SOP entitled *Internal Investigation of Potential Data Discrepancies and Determination for Data Recall* (SOP No. CW-L-S-002) outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

The Laboratory Director, a Technical Manager or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of

procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be documented. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Director of Quality & Client Advocacy and the laboratory's Quality Director within 24 hours of discovery.

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

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

11.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation & Recall Procedure (SOP No. CW-L-S-002) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-L-S-002.

11.4 Prevention of NonConforming Work

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

11.5 Method Suspension / Restriction (Stop Work Procedures)

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

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

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.
The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

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

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

SECTION 12. CORRECTIVE ACTION

12.1 Overview

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

documented using Non-Conformance Reports (NCR) and Corrective Action Reports (CAR) (refer to Figure 12-1).

12.2 General

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

The purpose of a corrective action system is to:

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

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

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

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

- Questionable trends that are found in the review of NCRs.
- Issues found while reviewing NCRs that warrant further investigation.
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports

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

12.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis,

Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

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

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness.

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

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

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

12.3.4 Monitoring of the Corrective Actions

- The Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Corrective actions are tracked by the QA department.
- The QA Manager reviews NCMs and CARs monthly for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.1.4, Special Audits.)

12.4 Technical Corrective Actions

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

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to specific method SOPs.

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

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with

an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented**.**

12.5 Basic Corrections

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

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

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

Figure 12-1.
Example - Corrective Action Report

CORRECTIVE ACTION REPORT (CAR)			Tracking Number:
Initiated By:		Assigned To:	
Initiation Date:		CC:	
Due Date:			
Section 1: Describe Problem & Attach Supporting Documentation As Needed			
Corrective Action Prompted By:			
Recurring NCR	Internal Audit	External Audit	Complaint / Other
Section 2: Root Cause Analysis			
Section 3: Describe Actions Required to Correct & Prevent Problem			
Section 4: QA Review and Close Out			
Action Taken Was:	Acceptable	Not Acceptable	Other
Comments:			
Close Out Date:		Closed By:	
Section 5: Follow Up (From Close-Out Date)			
Time Frame:	Performed By:	Date:	Is action taken preventing recurrence?
1 Month			
3 Month			
6 Month			
Comments:			

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Table 12-1. Example – General Corrective Action Procedures

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

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

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

SECTION 13. PREVENTIVE ACTION / IMPROVEMENT

13.1 Overview

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

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

Opportunities for improvement may be discovered during management reviews, the monthly QA Metrics Report, evaluation of internal or external audits, results & evaluation of proficiency testing (PT) performance, data analysis & review processing operations, client complaints, staff observation, etc.

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

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

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

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-Out 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 and management review.

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

13.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory such as the addition of new equipment or personnel.

Procedures for minimization of potential risks inherent with a new event or change are described in various laboratory standard operating procedures.

SECTION 14. CONTROL OF RECORDS

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

14.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA department. Records are of two types; 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 each laboratory section.

Table 14-1. Record Index[1]

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

	Record Types [1]:	Retention Time:
Administrative Records	Finance and Accounting	10 years
	EH&S Manual, Permits	7 years
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	7 Years (HR Personnel Files must be maintained indefinitely)
	Administrative Policies Technical Training Records	7 years

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

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

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

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

14.1.2 Programs with Longer Retention Requirements

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

Table 14-2. Example: Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	**5 years (project records)** **10 years - Radiochemistry (project records)**
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
NY Potable Water NYCRR Part 55-2	10 years
Ohio VAP	10 years and State contacted prior to disposal
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

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

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

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

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved.

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

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purpose. Instrument data is stored by instrument. Run logs

are maintained for each instrument. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required.

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

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

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

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

- Also refer to Section 19.14.1 ‘Computer and Electronic Data Related Requirements’**.**

14.2 Technical and Analytical Records

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

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

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

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

- laboratory sample ID code**;**
- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters.
- analysis type**;**
- all manual calculations and manual integrations**;**
- analyst's or operator's initials/signature**;**

- sample preparation
- 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.

14.3 Laboratory Support Activities

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

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records)**;**
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value**;**
- copies of final reports**;**
- archived SOPs**;**
- correspondence relating to laboratory activities for a specific project**;**
- all corrective action reports, audits and audit responses**;**
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

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

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

protect the integrity of samples.

14.4 Administrative Records

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

14.5 Records Management, Storage and Disposal

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

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

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

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. The procedures for document are described in laboratory SOP BR-QA-003.

14.5.1 Transfer of Ownership

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

14.5.2 Records Disposal

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

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

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

SECTION 15. AUDITS

15.1 Internal Audits

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

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CA-Q-S-004. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or Corporate QA	All areas of the laboratory annually
Method Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CA-Q-S-004)	Methods Audits Frequency: 50% of methods annually 100% of methods annually (DoD Labs)
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI field of proficiency testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, the TNI quality systems requirements, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.2 QA Technical Audits

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

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Quality Assurance Manager or qualified designee at least every two years.

15.1.4 Special Audits

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

15.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: water, soil, air.

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

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

15.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time

allotted by the client or agency performing the audit. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

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

15.2.1 Confidential Business Information (CBI) Considerations

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

15.3 Audit Findings

Audit findings are documented in audit reports and tracked by the QA department. The laboratory's corrective action responses for internal and external audits include action plans and date for completion. If a completion date cannot be met, a new a completion date must be set and agreed to by the QA Manager.

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

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

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

SECTION 16. MANAGEMENT REVIEWS

16.1 Quality Assurance Report

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

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

16.2 Annual Management Review

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

This management systems review (Corporate SOP No. CA-Q-S-008 & Work Instruction No. CA-Q-WI-020) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

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

- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

16.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/Recall SOP shall be followed (SOP No. CW-L-S-002). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's COO, VP of Client & Technical Services, General Managers and Quality Directors receive a monthly report from the Director of Quality & Client Advocacy summarizing any current data integrity or data recall investigations. The General Manager's are also made aware of progress on these issues for their specific labs.

SECTION 17. PERSONNEL

17.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular

area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page.

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience

Specialty	Education	Experience
Technical Manager / Department Manager– **General**	Bachelors Degree in an applied science or engineering. The Technical Manager must also have 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified peer or supervisors and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics – Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.

- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP BR-QA-011.

17.4 Data Integrity and Ethics Training Program

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy (Policy No. CW-L-P-004) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.
Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.

- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 Overview

The laboratory is a 22,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

18.2 Environment

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

18.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

18.4 Floor Plan

A floor plan can be found in Appendix 1.

18.5 Building Security

Building cards are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook. Signs are posted in the laboratory designating employee only areas - "Authorized employees beyond this point".

SECTION 19. TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPS)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002.
- SOPs are reviewed at a minimum of every 2 years except for SOPs for Drinking Water and DoD SOPs which are reviewed annually. Whenever necessary, SOPs may be revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*

- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *NIOSH Manual of Analytical Methods, 4th ed., August 1994.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability (DOC) is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel (e.g., analyst hasn't performed the test within the last 12 months).

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

19.4.3.1 The spiking standard used should be prepared independently from those used in instrument calibration.

19.4.3.2 The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

19.4.3.3 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.4 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

19.4.3.5 When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

19.4.3.6 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance

criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

19.4.3.7 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (refer to Figure 19-1 as an example) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements. Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL.

Refer to the laboratory SOP No. BR-QA-005 for details on the laboratory's MDL process, including detection limit procedures specific to the CLP SOWs for ISM and SOM.

19.8 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

If IDL is > than the MDL, it may be used as the reported MDL.

19.9 Verification of Detection and Reporting Limits

Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP

Scope of Accreditation. Refer to the laboratory SOP BR-QA-005 Method Detection Limits (MDLs/DLs) for further details.

The laboratory quantitation limit is equivalent to the DoD Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects. For DoD projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

19.10 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. Complete details are available in the laboratory SOPs.

19.11 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

19.12 Estimation of Uncertainty of Measurement

19.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly,

and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent 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.

19.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.13 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. Client specific or Contractual Terms & Conditions for reanalysis protocols may supersede the following items.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within $\pm$ 1 reporting limit for samples $\leq$ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and may reanalyze the sample a third time for confirmation if sufficient sample is available.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

19.14 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in corporate IT procedure and policies. The laboratory is currently running TALS which is a custom in-house developed LIMS system 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 an SQL database which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.14.1.1 Maintain the Database Integrity: Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.14.1.2 Ensure Information Availability: Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.14.1.3 Maintain Confidentiality: Ensure data confidentiality through physical access controls such as password protextion or website access approval when electronically transmitting data.

19.14.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices* and laboratory SOP BR-QA-006.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it

should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.14.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.14.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1**%.** Units are defined in each lab SOP.

19.14.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to the number of significant figured programmed in the LIMS formatter selected by the PM.

19.14.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

19.14.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.

- Worksheets are created with the approval of the QA Manager at the facility. The QA department controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

Review procedures are out lined in several SOP BR-QA-019 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 for manual integration, BR-QA-005. The general review concepts are discussed below, more specific information can be found in the SOPs.

19.14.4.1 The data review process at the laboratory 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.

19.14.4.2 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. To ensure data compliance, a different analyst 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. Approximately 15% of all sample data from manual methods and from automated methods, all GC/MS spectra and all manual integrations are reviewed. Manual integrations are also electronically reviewed utilizing auditing software to help ensure compliance to ethics and manual integration policies. Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.14.4.3 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Department Manager, Project Manager,

QA Manager or Technical Director, as necessary. Corrective action is initiated whenever necessary.

19.14.4.4 The results are then entered or directly transferred into the computer database and a report is prepared for the client.

19.14.4.5 As a final review prior to the release of the report, the Project Manager reviews the report for completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.

19.14.4.6 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002) as the guideline for our internal SOP BR-QA-006.

19.14.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.14.5.2 Analysts shall not increase or decrease peak areas ~~to~~ 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.

19.14.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.14.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Example - Demonstration of Capability Documentation

Analyst Demonstration of Capability

TestAmerica Burlington

Michelle Tam

10/12/2011

Preparation Method(s):	3010A
Analytical Method(s):	6020
Matrix:	Water
Method Description:	Metals (ICP/MS)
Preparation SOP No:	BR-ME-009R16
Analytical SOP No:	BR-ME-003R7

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the laboratory's Quality Assurance Plan, has completed the Demonstration of Capability(DOC).
2. The test method(s) was performed by the analyst identified on this certificate.
3. A copy of test method(s) and laboratory SOPs are available for all personnel on-site. **These documents have been reviewed by the analyst as part of this DOC.**
4. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility. The associated information is organized and available for review.

Michelle Tam — M. Chadkin Tam — 10/12/11
Analyst **Signature** **Date**

Bruce Stearns — — 10/12/11
Technical Director **Signature** **Date**

Kirstin Daigle — Kirstin Daigle — 10/13/11
Quality Assurance Officer **Signature** **Date**

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SECTION 20. EQUIPMENT and CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturers instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may be / are also outlined in analytical SOPs or instrument manuals.

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains “calibration only” ASTM type 1 weights).
All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly.

The mercury/digital NIST thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in support equipment logbooks.

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each day.

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in logbooks designated for this purpose.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and Glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is / can be applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated during initial instrument set up and as needed after that based on the instrument performance checks as specified in each test method. If instrument performance checks continue to indicate the calibration is valid over a time-frame that exceeds one calendar year; the company requires that the instrument be recalibrated at least annually.

Note: The following sections describe general requirements for instrument calibration and provide guidance for when the method does not specify a procedure or the procedure in the method is vague. These requirements may not be applicable to all test methods. The calibration procedure used for each test method is always specified in the laboratory's SOP for the test method.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points (exception being ICP and ICP/MS methods) will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed & documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable based upon discussion and approval of the client:

a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs. Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

20.6 GC/MS Tuning

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Example: Instrumentation List

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Analytical Balance	Mettler	AT200	113081164	UNKNOWN	UNKNOWN
Analytical Balance	Mettler	ML204	1123452701	2010	NEW
Analytical Balance	Metler	ML204	1123452699	2010	NEW
Analytical Balance	Sartorius	XM1000P	40090006	UNKNOWN	UNKNOWN
Automated Distillation Apparatus	Westco	Easy Dist	1090	2002	NEW
Automated Distillation Apparatus	Westco	Easy Dist	1091	2002	NEW
COD	HACH	45600-00	11000022452	UNKNOWN	UNKNOWN
Conductivity Meter	Oaklon	CON110	35607-85	2001	
CVAA	Leeman (CV3)	HydraAA112-0064-1	2031	2003	NEW
CVAA	Leeman (CV4)	HydraAA112-0064-1	8015	2008	NEW
GC/ECD/ECD	Agilent (7424)	6890N	US10332093	2003	NEW
GC/ECD/ECD	Agilent (3283)	6890N	US10805001	2008	NEW
GC/ECD/ECD	Hewlett-Packard (2618)Screen	5890II	3203A41055	1987	UNKNOWN
GC/ECD/ECD	Agilent (7227)	6890N	CN10602095	2006	NEW
GC/ECD/ECD	Agilent (0825)	6890N	US10202136	2002	NEW
GC/ECD/ECD	Agilent (1031)	7890A	CN10301031	2010	NEW
GC/ECD/ECD	Agilent (5253)	6890N	CN10723008	2007	NEW
GC/ECD/ECD	Agilent (0911)	6890N	US10230082	2002	NEW
GC/ECD/ECD	Agilent (5005)	6890N	CN10615005	2009	USED
GC/FID/ECD	Hewlett-Packard (Screen)	5890	GC 2415A01109	UNKNOWN	UNKNOWN
GC/FID/FID	Hewlett-Packard (3012)	5890II	3235A45259	1984	UNKNOWN
GC/FID/FID/TCD	Varian (CP3800)	CP-3800	S/N 10328	2003	NEW
GC/FID/TCD	Varian (2866)	VR-3600	2866	1998	UNKNOWN
GC/FPD/FPD	Hewlett-Packard (2860)	5890II	2950A27078	1990	UNKNOWN
GC/MS	Hewlett-Packard (N)	5890II / 5971	3203A40979	1998	NEW
GC/MS	Hewlett Packard (V)	5890II / 5972	3336A61485	1998	NEW
GC/MS	Agilent (B)	6890N/ 5973	CN10317006	2003	NEW
GC/MS	Agilent (C)	6890N / 5973	CN10424016	UNKNOWN	NEW

GC/MS	Agilent (G)	6890N / 5973	CN10437065	UNKNOWN	USED
GC/MS	Agilent (E)	6890N / 5973	CN10453004	2005	NEW
GC/MS	Agilent (F)	6890N/ 5973	CN10531065	2005	NEW
GC/MS	Agilent (S)	7890A/5975	CN10211095	2010	NEW
GC/MS	Agilent (T)	7890A/5975	CN10211037	2010	USED
GC/MS	Hewlett-Packard (L)	5890II / 5971	3203A40982	1998	NEW
GC/MS	Hewlett-Packard (M)	5890II / 5971	3203A40980	1998	NEW
GC/MS	Agilent (D)	6890N / 5973	CN10439015	2004	NEW
GC/MS	Hewlett-Packard (P)	5890II / 5971	3203A40985	1992	USED
GC/MS	Hewlett-Packard (Q)	5890II / 5971	3203A40983	1992	NEW
GC/MS	Hewlett-Packard (R)	5890II / 5971	3203A40984	1992	NEW
GC/MS	Hewlett-Packard (U)	5890II Plus/ 5972	3336A61535	1997	NEW
GC/MS	Agilent (H)	6890N / 5975	CN10608102	2006	NEW
GC/MS	Agilent (Z)	6890A/ 5973	US00036343	2000	NEW
GC/MS	Agilent (J)	6890N / 5973	CN10430052	2009	USED
GC/FID	Hewlett-Packard (6453-K) Screen	5890 II	3203A41768	UNKNOWN	UNKNOWN
GPC	J2 Scientific (I)	Autoinject 110	02D-1030-2.1	2002	NEW
GPC	J2 Scientific (H)	Autoinject 110	02D-1031-2.1	2001	NEW
GPC	J2 Scientific (J)	AccuPrep	03G1076-3.0	2003	NEW
HPLC/UV	Dionex (1488)	P680	1680407	1991	UNKNOWN
HPLC/UV/PDA	Waters (1208)	600	60004790RP	1988	NEW
Hydrogen Generator	Parker Hannafin	H2-800	h2-800081C	2006	NEW
Hydrogen Generator	Parker Hannafin	H2-800	h2-800099C	2006	NEW
ICP-MS	Thermo Elemental (2)	X7	X0288	2003	NEW
ICP-OES	Thermo Electron Corp (7)	iCAP 6000	ICP20063302	2006	NEW
LC/MS/MS	Waters (1111)	Acquity/Quattro micro	QAA929	2005	NEW
LC	Waters (3062)	616	MX5NM6829M	UNKNOWN	NEW
pH Meter	Denver Instruments	UB-5	UB503B365	UNKNOWN	UNKNOWN
Soxtherm	Gerhardt (SOXA)	SE3AS306A	4012396	UNKNOWN	UNKNOWN
Soxtherm	Gerhardt (SOXB)	SE3AS306A	4022047	UNKNOWN	UNKNOWN
Soxtherm	Gerhardt (SOXC)	SE3AS306A	4022046	UNKNOWN	UNKNOWN
Soxtherm	Gerhardt (SOXD)	SE3AS306A	4022045	UNKNOWN	UNKNOWN

Soxtherm	Gerhardt (SOXE)	SE3AS306A	4022030	UNKNOWN	UNKNOWN
Soxtherm	Gerhardt (SOXF)	SE3AS306A	4012397	UNKNOWN	UNKNOWN
TKN Digestion System	Aim Lab	AIM600 Block	5048A23014	2011	NEW
TOC	Carlo Erba	NA 1500	220465	1991	UNKNOWN
TOC	Carlo Erba	EA1108	249146	1991	UNKNOWN
TOC	Costech	4010	231009973	2005	UNKNOWN
TOC	Shimadzu	TOC-V CPH	H5131480032AE	2011	NEW
Turbidimeter	HF Scientific	Micro 100	208463	2001	UNKNOWN
UV/VIS	Genesys	Spectronic 20	3SGEO38002	1999	UNKNOWN
UV/VIS	Genesys	Spectronic 20	3SGE165024	2002	UNKNOWN
UV/VIS	Lachat	Quick Chem 8000	A83000-2167	2000	UNKNOWN

Table 20-2. Example: Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Leeman Mercury Analyzer	Check Peristaltic Pump tubing	As required
	Lubricate Autosampler rods	Monthly
	Clean Autosampler	Weekly
	Check and fill Rinse Vessel	As required
	Check and fill Stannous Chloride	As required
	Check Waste Vessel	Daily
	Empty Waste Vessel	As required
ICP	Check Peristaltic Pump tubing	As required
	Clean Torch	Daily
	Replace Torch	As required
	Check and fill Rinse Vessel	As required
	Check and fill IS Vessel	As required
	Fill Standards Cup	Daily
	Check Waste Vessel	Daily
	Empty Waste Vessel	As required
	Check and clean Cones	As required
	Perform Auto Peak Adjustment	As required
ICP MS	Check Peristaltic Pump tubing	As required
	Clean Torch	As required
	Check and fill Rinse Vessel	As required
	Check and fill IS Vessel	As required
	Fill standards cup	Daily
	Check Waste Vessel	Daily
	Empty Waste Vessel	As required
	Check and clean Cones	As required
UV-Vis Spectrophotometer	Clean ambient flow cell	As required
	Wavelength verification check	As required
	Clean Cuvette with Cuvette Cleaning Solution	As required
Hewlett Packard GC/MS (VOA)	Clean Injection Port and Liner	As required
	Change Septa	As required
	Cut 2-3 inches from GC Column	As required
	Fill Autosampler rinse vials	As required
	Clean Purge and Trap mount and purge vessel	As required
	Check Purge Flow	As required
Hewlett Packard GC/MS (SVOA)	Clean Injection Port and Liner	Daily
	Change Septa	Daily
	Replace or clip Guard Column	Daily
	Replace or clip Analytical Column	Daily
	Fill Autosampler rinse vials	Daily
Hewlett Packard GC/MS (Air)	Check GC / Entech Column Interface	As required
	Check Nitrogen Tank Volume	As required
	Check Nitrogen Valves Software and Valves	As required
	Cut 2-3 inches from GC Column	As required
Gas Chromatograph	Replace Septa	As required
	Clean and replace Injection Port Liner	As required
	Replace or clip Guard Column	As required
	Replace or clip Analytical Column	As required
	Bake, Re-foil, Refurbish Detector	As required

Instrument	Procedure	Frequency
Zero Air Generator	Change pre-filter cartridge Replace catalyst module Check Indicator Beads in Moisture Filters Bake and Refill Mol Sieve Dry Rite Beads	Annually Indicator Light Blinks Daily As required
Hydrogen Generator	Fill Water Reservoir Replace Water in Water Reservoir Replace Ionic Bags in Water Reservoir	Daily Semi-Annually Semi-Annually
HPLC	Change Transfer Lines Replace Guard Column Replace Analytical Column Replace or clean Pump Head Check Valves Change Plunger Seals Change Suppressor Change Eluent Generator Cartridge and CR-ATC	As required As required As required As required As required As required As required
LC/MS/MS	Replace Guard Column Replace Analytical Column Replace or clean Pump Head Check Valves Change Plunger Seals Change In Line Filter Clean or Change Sample Cone Clean Source	As required As required As required As required As required As required As required
Balances	Class “1” traceable weight check Clean pan and check if level Field service	Daily, when used Daily Annually
Latchat	Change Tubing Replace Bulb	As required As required
Conductivity Meter	Calibrate	Daily
Turbidimeter	Calibrate Check light bulb	As required Daily, when used
Drying Ovens	Temperature monitoring Temperature adjustments	Daily As required
Refrigerators/ Freezers	Temperature monitoring Temperature adjustment Defrosting/cleaning	Daily As required As required
pH/Specific Ion Meter	Calibrate Clean electrode	Daily As required
Centrifuge	Check brushes and bearings	Every 6 months or as needed
Water baths	Temperature monitoring Water replaced	Daily, when used Monthly or as needed

SECTION 21. MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware and Glass microliter syringes quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and Glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), 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.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors 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 unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the ‘true’ value does not exceed method requirements. The accuracy of calibration

standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. [Refer to TestAmerica's Corporate SOP (CA-Q-S-001), Solvent and Acid Lot Testing and Approval.]

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained **in each lab section.** 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.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

21.4.1 All standards, reagents, and reference materials 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
- Preparer's name

- Final volume
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained on the company's intranet and in test method SOPs.

21.4.3 In addition, the following information may be helpful:

- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

SECTION 22. SAMPLING

22.1 Overview

The laboratory does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. 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 holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots & subsampling are located in test method SOPs.

SECTION 23. HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form should include information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification

- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in log-in by date; it lists all receipts each date.

23.1.2 Legal / Evidentiary Chain-of-Custody

If the client requests legal COC sample management personnel will initiate an internal COC for laboratory use by analysts and a sample disposal record.

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

23.2.1 Laboratory Receipt

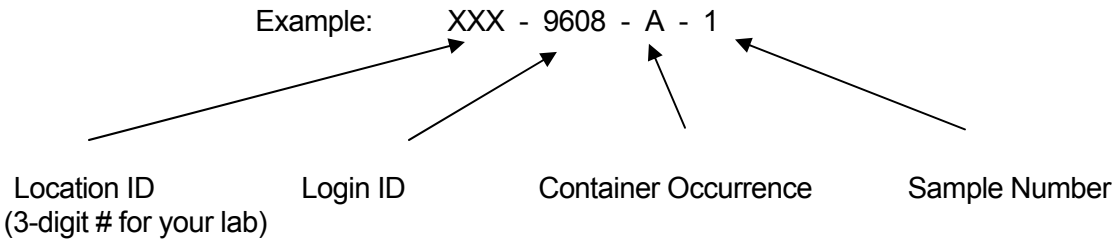
When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance,

irregularity, or compromised sample receipt must be documented*]* and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):



The above example states that TestAmerica <location> Laboratory (Location XXX). Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: ~~XXX - 9608 -~~ A - 1 - **A**

Secondary Container Occurrence

Example: 220-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a complete COC;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or

- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according to laboratory SOP BR-SM-001.

23.4 Sample Storage

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed weekly.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples, including empty sample containers, are returned to the secure sample control area until disposal.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 Hazardous Samples and Foreign Soils

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only. For any sample that is known to be hazardous at the time of receipt or, if after completion of analysis the result exceeds the acceptable regulatory levels, a Hazardous Sample Notice must be completed by the analyst. This form may be completed by Sample Control, Project Managers, or analysts and must be attached to the report. The sample itself is clearly marked with a red stamp, stamped on the sample label reading “HAZARDOUS” or “FOREIGN SOIL” and placed in a colored and/or marked bag to easily identify the sample. The date, log number, lab sample number, and the result or brief description of the hazard are all written on the Hazardous & Foreign Soil Sample Notice. A copy of the form must be included with the original COC and Work Order and the original must be given to the Sample Control Custodian. Analysts will notify Sample Control of any sample determined to be hazardous after completion of analysis by completing a Hazardous Sample Notice. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm that lab-packs all hazardous samples and removes them from the laboratory.

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

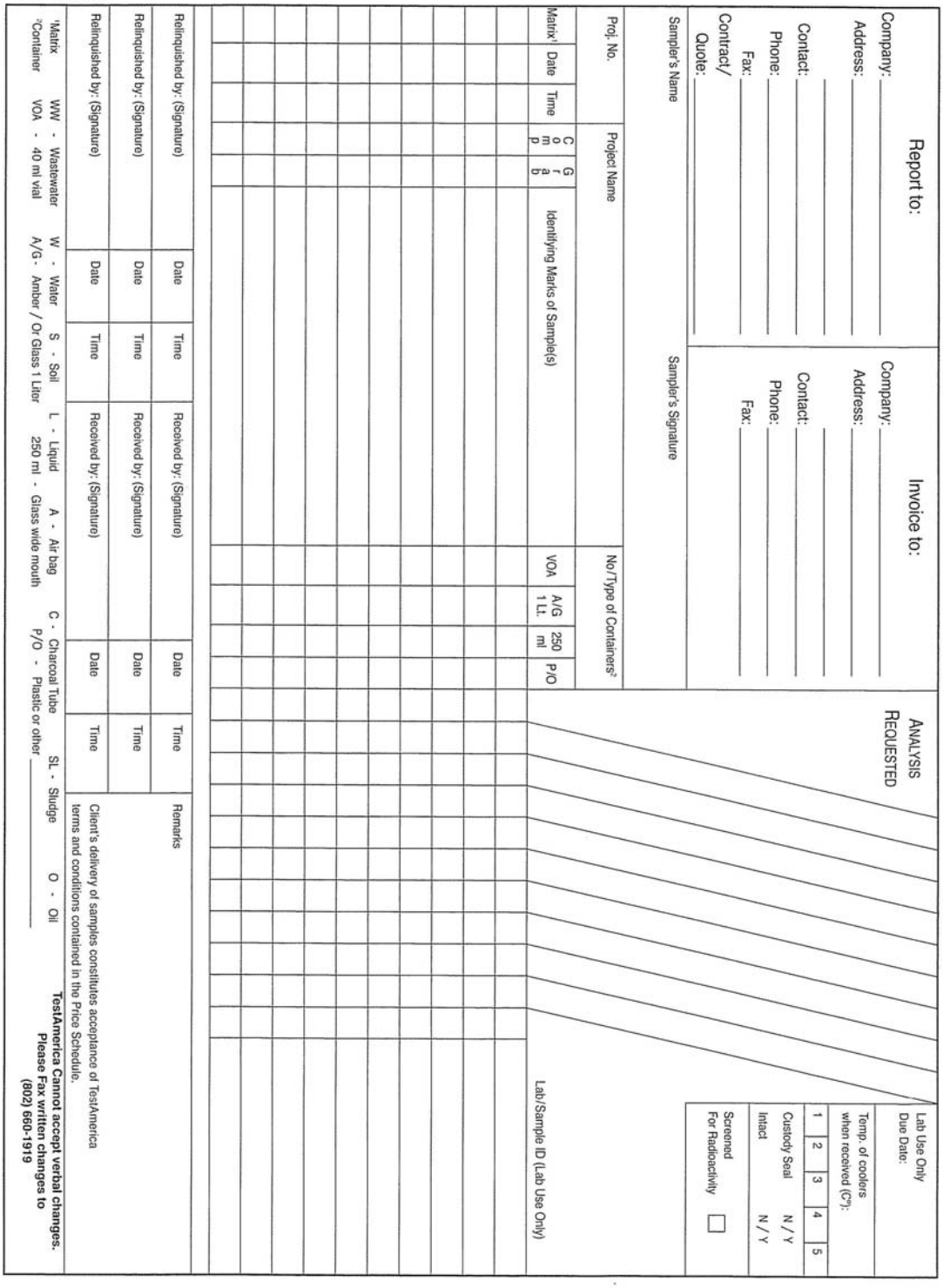
Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. 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: BR-EH-001) All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-1. Example: Chain of Custody (COC)



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Burlington
30 Community Drive, Suite 11
South Burlington, VT 05403 Tel: 802 660 1990

CHAIN OF CUSTODY RECORD

TAL-8234(1007)

Report to: Company: Address: Contact: Phone: Fax: Contract/ Quote: Sampler's Name

Invoice to: Company: Address: Contact: Phone: Fax: Sampler's Signature

Analysis Requested

Lab Use Only Due Date:

Temp. of coolers when received (C°): 1 2 3 4 5

Custody Seal N / Y
Intact N / Y

Screened For Radioactivity ☐

Proj. No. Project Name

No./Type of Containers²

Matrix¹	Date	Time	Comp	Grab	Identifying Marks of Sample(s)	VOA	A/G 1 Lt.	250 ml	P/O	Lab/Sample ID (Lab Use Only)

Relinquished by: (Signature) Date Time Received by: (Signature) Date Time Remarks

Relinquished by: (Signature) Date Time Received by: (Signature) Date Time

Relinquished by: (Signature) Date Time Received by: (Signature) Date Time

Client's delivery of samples constitutes acceptance of TestAmerica terms and conditions contained in the Price Schedule.

TestAmerica Cannot accept verbal changes. Please Fax written changes to (802) 660-1919

¹Matrix: WW - Wastewater, W - Water, S - Soil, L - Liquid, A - Air bag, C - Charcoal Tube, SL - Sludge, O - Oil
²Container: VOA - 40 ml vial, A/G - Amber / Or Glass 1 Liter, 250 ml - Glass wide mouth, P/O - Plastic or other ____

Figure 23-2. Example: Sample Acceptance Policy

The receipt of samples is acknowledged on the chain of custody (COC) form with the signature and date/time of the sample custodian. The condition of samples upon receipt is documented on checklists designated for this purpose. Any deficiencies identified during sample receipt are recorded and communicated to the laboratory project manager (PM), who will contact the client and fully document any decision to proceed with analysis in the project record. Consultation with the client should be immediate and timely (next business day or as specified in the project plan). Correspondence records and/or records of conversations concerning the decision to proceed with analysis and/or the disposition of rejected samples is maintained in the project record, and should be maintained in association with the sample receipt checklist. All data associated with samples that did not meet the sample acceptance criteria must be qualified with a Non-Conformance Report (NCR) and/or noted in the project narrative that accompanies the final test report.

Sample receipt is considered deficient when the following conditions are observed:

- Shipping cooler and/or samples are received outside the temperature specification
- Sample bottles are received broken or leaking
- Samples are received beyond holding time
- Samples are received without the appropriate preservation
- Samples are not received in appropriate containers
- Chain of Custody does not match the samples received
- Chain of Custody was not received or is incomplete*
- Custody seals are broken
- Evidence of tampering with the cooler and/or samples
- Headspace in 40mL or 22 mL VOA vials
- Seepage of extraneous water or other material into the samples
- Inadequate sample volume
- Illegible, impermanent ink, or non-unique sample labeling
- One or more coolers missing from a multi parcel shipment
- Shipping container is damaged

**Complete documentation shall include sample identification, the location date/time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample.*

Figure 23-3. Example: Cooler Receipt Form

TestAmerica Burlington
SAMPLE RECEIPT & LOG IN CHECKLIST

Client:	**Date Received:**	**Job #:**
Project #:	**Time Received:**	
PM:	**Received By:**	**Login#:**
Login Date:	**# Coolers Received:**	
	Samples Delivered By:	**ICOC Required? Y/N**
Initials:	⊐ Shipping Service	*If "Y", attach copy(s) of ICOC*
Signature:	⊐ Courier	
	⊐ Hand	

Receipt Info	**YES**	**NO**	**NA**	**COMMENTS**
There is *no* evidence to indicate tampering				
Custody seals are present and intact				
Custody seal numbers are present				
If yes, list custody seal numbers:				

IR Gun ID: **Correction Factor:** ° C

Thermal Preservation Type: ⊐ Wet Ice ⊐ Blue Ice ⊐ None ⊐ Other (specify)
Packing Material: ⊐ Bubble Wrap ⊐ Cardboard ⊐ Corrugated Paper ⊐ Shredded Paper ⊐ Styrofoam ⊐ Vermiculite ⊐ None

Cooler 1:	°C	Cooler 6	°C	Cooler 11	°C	Cooler 16	°C
Cooler 2:	°C	Cooler 7	°C	Cooler 12	°C	Cooler 17	°C
Cooler 3:	°C	Cooler 8	°C	Cooler 13	°C	Cooler 18	°C
Cooler 4:	°C	Cooler 9	°C	Cooler 14	°C	Cooler 19	°C
Cooler 5	°C	Cooler 10	°C	Cooler 15	°C	Cooler 20	°C

Unless otherwise documented, the recorded temperature readings are adjusted readings to account for the CF of the IR Gun
EPA Criteria: 0-6°C, except for air and geo samples which should be at ambient temperature and tissue samples, which may be frozen.
Some clients require thermal preservation criteria of 2-4°C or other such criteria. The PM must notify SM when alternate criteria is specified.
Comments:

Report Management-*Workshare (TALS Labs)*	**YES**	**NA**	**Initials**	**Date**
Workshare (TALS Labs)-Shipping and Receiving Documents scanned and attached to job.				
Email notification sent to job contact				
Report Management-*Login Review*	**YES**	**NA**	**Initials**	**Date**
Shipping and Receiving Documents: Scanned and attached to deliverable job folders.				
Project Manager-*Login Review*	**YES**		**Initials**	**Date**
Login Review Performed				
EDD Questions	**YES**	**NA**	**Initials**	**Date**
Applicable login/job questions answered				
Report Management-*Preliminary Deliverables*	**YES**	**NA**	**Initials**	**Date**
Lab Documents-Scanned and attached to appropriate job deliverables folder.				
Subcontract Data (if applicable)- Non-TALS Labs, 3rd Party Labs Scanned and attached to appropriate job deliverables folders				
Narrative-NCMs added, if present				
Preliminary Reports-run and assembled				
EDD(s)-run and created				
Project Manager	**YES**		**Initials**	**Date**
Final review and release completed				
Report Management-*Final Deliverables*	**YES**	**NA**	**Initials**	**Date**
Reports-Hardcopy printed (if requested), CD(s) burned				
Invoice-Approved in Invoice Desktop and printed (if hardcopy requested)				
Email-Report(s), EDD(s), and invoice (as requested)				
Delivery confirmed for report(s), EDD(s) and Invoice in TALS				

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TestAmerica

SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

Table 24-1. Example – Negative Controls

Control Type	Details
Trip Blank [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous

volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Table 24-2. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;

Table 24-2. Sample Matrix Control

Control Type	Details	
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects.If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated when necessary unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).

- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits.

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.

- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

24.6.3 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.4 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client).

24.7 Additonal Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.

SECTION 25. REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

The format of each report type is specific to the client or regulatory program and is therefore not included in the QAM. The reporting specifications for CLP contract samples must comply with the specifications for CSF organization, preparation and review as specified in the SOW. Procedures for preparation of the CSF are provided in laboratory SOP BR-RM-001.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report For Samples) with a "sample results" column header.

25.2.2 Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report 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.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

25.2.11 Practical quantitation limits or reporting limit.

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).

25.2.17 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.18 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

25.2.19 When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of TNI Standard or provide reasons and/or justification if they do not.

25.2.20 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.21 When soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

25.2.22 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.23 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, or how your lab identifies it). A complete report must be sent once all of the work has been completed.

25.2.24 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.28 A clear statement notifying the client that non-accredited tests were performed and directing the client to the laboratory's accreditation certificates of approval shall be provided when non-accredited tests are included in the report.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

25.3 Reporting Level or Report Type

The laboratory routinely offers four levels of quality control reporting.

- Level I is a report with the features described in Section 25.2 above except QC summary information is not included.
- Level II is a Level I report plus QC summary information.
- Level III contains all the information supplied in Level II, but presented on CLP-like summary forms, and relevant calibration information. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

The format of each report type is specific to the client or regulatory program and is therefore not included in the QAM. The reporting specifications for CLP contract samples must comply with the specifications for CSF organization, preparation and review as specified in the SOW. Procedures for preparation of the CSF are provided in laboratory SOP BR-RM-001.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. TestAmerica Burlington offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific

electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 Supplemental Information for Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP No. CA-L-S-002).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 Client Confidentiality

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at the 1-800-765-0980 (or for e-mails: please notify us immediately by e-mail or by phone (1-800-765-0980) and delete this material from any computer).

25.7 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "R" ***[indicate your naming scheme]***. The revised report will have the word "revised" or "amended" next to the date rather than the word "reported".

When the report is re-issued, a notation of "report re-issue "is placed on the cover/signature page of the report *or at the top of the narrative page* 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 11/3/08 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/08 at 10:47am.*

25.9 Policies on Client Requests for Amendments

25.9.1 Policy on Data Omissions or Reporting Limit Increases

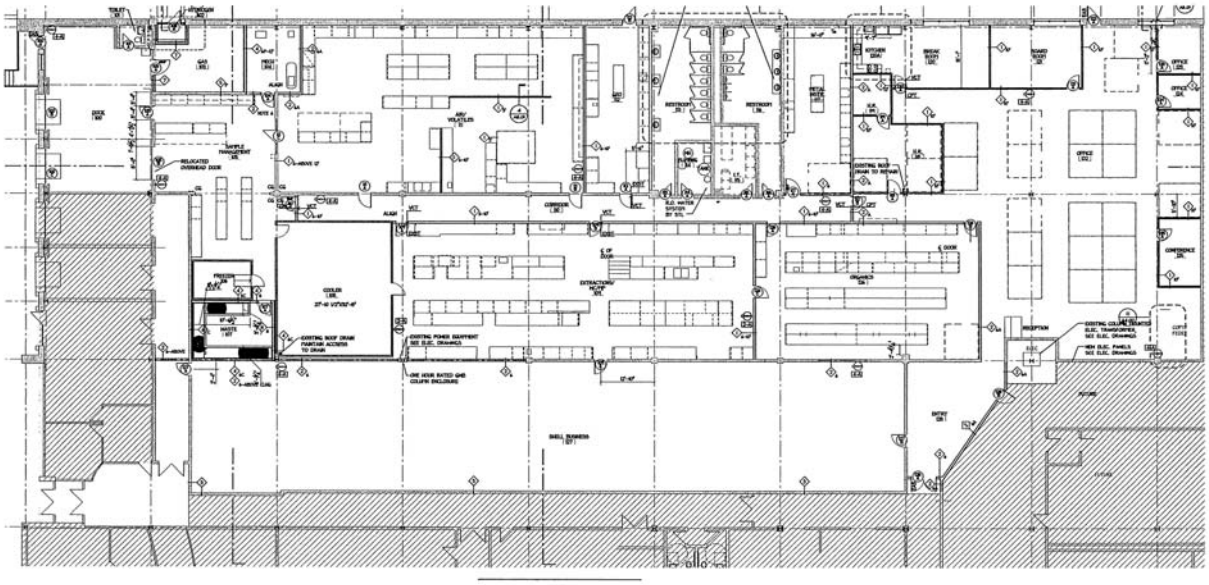
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. Laboratory Floor Plan



Appendix 2. Glossary/Acronyms (EL-V1M2 Sec. 3.1)

Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical ~~r~~relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material~~,~~ accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis**.** No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.
External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: hose matrix, technology/method, and analyte combinations for which the NELAP accreditation body offers accreditation.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as

total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

LOD Verification [a.k.a., MDL Verification]: A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

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

(QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater~~,~~ effluents, and TCLP or other extracts.

Drinking Water: Any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Air & Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test

result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

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

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, ~~product~~ or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2nd 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 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – ICP/Mass Spectrometry
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
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
TNI – The NELAC Institute
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP – Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Burlington maintains accreditation, certifications and approvals with numerous state and national entities. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Lab ID	Program	Program Type	Authority
NA	Delaware DNREC		Delaware
ADE-1492	DoD ELAP	DoD	ACLASS
200610	NELAC	Secondary AB	New Hampshire
VT972	NELAC	Primary AB	New Jersey
10391	NELAC	Secondary AB	New York
68-00489	NELAC	Secondary AB	Pennsylvania
E87467	NELAC	Secondary AB	Florida
176292	NELAC	Secondary AB	Louisiana
PH-0751	State Program		Connecticut
VT00008	State Program		Maine
050-999-436	State Program		Minnesota
LAO00298	State Program		Rhode Island
VT-4000	State Program		Vermont
P330-11-00093	USDA		USDA

The certificates and parameter lists are available upon request from a laboratory representative. A complete list of analytical capabilities may be found on the company's web site, the laboratory's public server or from a representative of the laboratory.

Title: Gasoline Range Organics – Total Area Quantification [Method No. 8015B/D]

Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):

8/30/13
Gary S. Rudz Date
GC Manager

8/30/13
Brad Prinzi Date
Quality Assurance Manager

8/30/13
Christopher Spencer Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This method is used to qualify and quantify Gasoline Range Organics by purge & trap techniques into a capillary column equipped gas chromatograph. This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and the integration of gas chromatograms.

1.2 Analytes, Matrix(s), and Reporting Limits

1.3 All soils and waters

1.4 Routine laboratory reporting limits are 50 ug/l for aqueous samples and 250 ug/kg for soil samples.

1.5 Method detection limits are established for each compound by analyzing seven replicate spiked samples and using the following equation. This is done on an annual basis.

$$MDL = (3.14)(S_c)$$

Where:

S_c = standard deviation of concentration for seven replicate samples

The MDL is determined annually and is on file with the QA dept.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

2.1 The Gasoline Range Organics Method provides gas chromatographic conditions for the detection of various aromatic volatile compounds using purge and trap techniques. An inert gas is bubbled through a 5ml water sample contained in a specifically designed purging chamber at ambient temperature. The aromatics are transferred efficiently from the aqueous phase to the vapor phase and swept through a sorbent trap where the aromatics are trapped. After purging is complete, the trap is flash-heated and flushed with the inert gas to desorb the aromatics onto the gas chromatographic column. The gas chromatograph is temperature programmed to separate the aromatics which are then detected by a flame ionization detector (FID).

3.0 Definitions

All abbreviations are explained in the text of this SOP, and additional definitions may be found in the Test America Buffalo LQM.

4.0 Interferences

4.1 Samples can be contaminated by diffusion of volatile organics through the sample container septum during shipment and storage. A field blank, a trip blank and a holding blank prepared from reagent water and carried through the sampling and/or subsequent storage and handling can serve as a check on such contamination.

4.2 Contamination by carryover can occur whenever a sample with high levels is analyzed. To reduce carryover the purging device, syringe and lines are flushed between every analysis. The trap is baked at 270°C between each analysis.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Gasoline	Flammable	N/A	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
2-Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

Gas chromatograph suitable for on-column injection and all required materials, i.e., syringes, columns, gases, detector and a data processing system capable of measuring peak areas and heights.

Hewlett Packard 5890 gas chromatograph
Tekmar, Dynatech, Purge & Trap
Archon Auto Sampler
Capillary columns as described in sections 9.2
Flame Ionization Detector
PE Nelson Totalchrom data system
Carrier Gas – Helium
Syringes - various

6.1 Instrumentation

Hewlett Packard 5890 gas chromatograph
Tekmar, Dynatech, Purge & Trap
Archon Auto Sampler

Columns - three different capillary columns are used in TestAmerica's 8015B/D (GRO) analysis:

Column analytical system
Column or equivalent
30 m x 0.53 mm ID RTX-VMS (Restek).
60 m x 0.53 mm ID RTX-VGC (Restek).
60 m x 0.53 mm ID ZB-624 (Phenomenex)

Sample introduction apparatus:
Purge – and – trap – See Method 5030

6.2 Supplies

Volumetric flasks - various sizes

Micro syringes - various sizes

Carbon treated tap water for volatile-free source of water

7.0 Reagents and Standards

7.1 All standards are stored in the freezer at a maximum of (-2°C).

7.2 Volatile standards are prepared covering a concentration range expected to be found in real samples. All standards are to be properly recorded in a given standard log.

Separate Standard Operating Procedures address standard preparation and standards recordkeeping.

7.3 Stock Standards
- **7.3.1** Unleaded Gasoline Standard purchased from Ultra Scientific at 5000 ng/ul in methanol.
- **7.3.2** Commercial grade gasoline – used as second source material (ICV)

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Aqueous samples should be collected in (2) 40 ml Level I VOA vials. Soil samples should be collected in (1) 4 oz Level I glass wide-mouth jar. All samples should be maintained with zero headspace.

8.2 Both aqueous and soil samples should be shipped with ice.

8.3 Both aqueous and soil samples must be stored and maintained at the lab at 4°C, ±2, until the analyst is prepared for the analytical process.

8.4 Both aqueous and soil samples are to be analyzed within 14 days of sample date.

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Level 1 VOA vial	50 mLs	Cool 4 ± 2°C	14 Days	40 CFR Part 136.3
Soils	Glass	3 grams	Cool 4 ± 2°C	14 Days	N/A

9.0 Quality Control

9.1 ICAL: Analysis can not begin without an acceptable calibration. Instrument maintenance may be required. Refer to TestAmerica Corporate Policy P-T-001, R3 for information on the proper selection of calibration points.

9.2 ICV: Reanalyze calibration curve if unacceptable ICV is obtained (<15%)

9.3 CCV: (%D<15%)
- **9.3.1** Reanalyze the CCV
  - **9.3.1.1** If the 2nd analysis is acceptable, analytical sequence may continue, however the previous 10 samples must be re-analyzed.
  - **9.3.1.2** If the 2nd analysis is unacceptable, analyze a new ICAL

9.3.2 If the CCV is out high and there are no positives in the samples, the results may be reported. This situation must however be noted in the logbook and on the Job Summary.

9.4 GVOA Decision Tree – running 2 consecutive CCVs
- **9.4.1** Run: CCV1
- **9.4.2** Run: CCV2
  - **9.4.2.1** CCV 1 Passes & CCV2 Passes
  - **9.4.2.2** Continue as Normal Routine

  - **9.4.2.3** CCV1 Passes & CCV2 Fails
  - **9.4.2.4** Rerun all runs after failed CCV

  - **9.4.2.5** CCV1 Fails & CCV2 Passes
  - **9.4.2.6** Rerun all runs before CCV1 and last Compliant CCV.

  - **9.4.2.7** CCV1 Fails & CCV2 Fails
  - **9.4.2.8** Rerun all samples before and after CCVs after Maintenance and/or Recalibration

9.5 Method Blank: Reanalyze all samples associated with an unacceptable method blank.

9.6 LCS:
- **9.6.1** If below limits: Reanalyze all samples associated with an unacceptable LCS
- **9.6.2** If above limits: Reanalyze all samples with detections. Reanalysis is *not required* if samples are ND.

9.7 MS/MSD:
- **9.7.1** Matrix interference can be assumed and corrective action is not required if both of the following conditions are met:
  - **9.7.1.1** LCS recovery is acceptable
  - **9.7.1.2** Recoveries in both the MS and MSD are consistent (RPD<30%)

    9.7.1.2.1 If sample appearance indicates that the MS/MSD pair may not provide reproducible results, the poor results may be accepted but this should be noted in the job summary and case narrative.

- **9.7.2** If LCS is unacceptable – reanalysis of batch is required.

- **9.7.3** If recoveries in MS/MSD are different (i.e.: one high, one low) further evaluation should be made. Matrix interference can not be assumed in this case. Discussion with the department supervisor, operations manager or QA manager should be included in the final decision process prior to releasing data.

9.8 Surrogate:

9.8.1 If below limits – reanalyze samples

9.8.2 If above limits – reanalyze sample if detections obtained. Reanalysis is *not required* if sample is ND.

CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

A Job Exception Form must be completed and filed with the Project Manager and QA Manager for any of the following conditions:

Holding times exceeded
Insufficient sample volume for re-analysis
Re-analysis required due to method blank, surrogate or LCS failure

In the event of unknown positives or sample matrix which present the analyst with questionable data, the project manager shall be notified so the client may be contacted and involved in the decision process and course of action

9.9 **Sample QC** - The following quality control samples are prepared with each batch of samples.

9.10 The reader is referred to TestAmerica's Laboratory Quality Manual (LQM) for general information and more specific detail. Often project specific quality assurance documents will provide overriding criteria to that presented below. Those criteria depending on project-specific data quality objectives may be more or less stringent than TestAmerica's LQM or the following criteria. The following criteria are subsequently presented as minimum criteria on those criteria deemed applicable in the absence of project-specific Data Quality Objectives.

9.11 Surrogates - All standards and samples are fortified with a surrogate material in order to monitor both system and method performance. The surrogate material routinely used by TestAmerica is A,A,A-Trifluorotoluene and is added to all samples at prescribed levels (usually 150ng/ul). Please refer to Laboratory GRO manual for specific recovery limits. Limits are client and state specific. Laboratory specific surrogate limits are calculated yearly by using the following equation:

Upper limit = p+3s
Lower limit = p-3s

where p=ave.% rec. and s=standard deviation

9.11.1 If the surrogate is out-of-control, re-analysis is required.

9.11.2 If upon re-analysis, surrogate recoveries are under control, report only the re-analysis.

9.11.3 If re-analysis again results in out-of-control surrogate recoveries, report both analyses and note matrix effects in the case narrative.

9.11.4 If initial surrogate failure is due to chromatographic interference which resulted in large portion bias, re-analysis may be inappropriate, but approval of the client, TestAmerica Program Manager and/or the Quality Manager is required.

9.12 Matrix Spike / Matrix Spike Duplicates - MS and MSD samples are to be prepared at a frequency of at least 5% (1 MS per 20 samples and 1 MSD per 20 samples) or with each analytical job/case. Batch MS/MSD (i.e., 1/20 over a number of jobs/cases) is an option to fulfilling the requirements, but at least one (1) set of MS/MSD should be run per day of instrument operation.

9.12.1 Both water and soil spikes should be at a level of 1000 total ng.

9.12.2 MS and MSD percent recoveries and Relative Percent Differences (RPDs) between MS and MSD recoveries are advisory, unless defined as mandatory by an applicable project-specific quality assurance plan. Advisory recovery limits are 50-150% and %RPD between MS/MSD recoveries $\leq$50%.

9.13 Laboratory Control Sample / Matrix Spike Blank - A minimum of one (1) matrix spike blank 12 hours (or batch) is required.

9.13.1 Spiking levels for LCS are the same as for MS/MSD. Please refer to Laboratory GRO manual for required recovery limits. Limits are client and state specific.

9.13.2 Inject 10.0ul of the unleaded gasoline standard (2000ug/ml) into 100 mls of volatile free water for a final concentration of 0.2 ug/ml. This concentration should yield 1000 ng on column.

9.13.3 To prepared soil sample add 100ul of the unleaded gasoline standard (500ug/mL) and 100ul of surrogate (75ug/mL).

9.13.4 In the event of LCS failure, re-analysis is required. If re-analysis continues to indicate LCS failure, all sample analyses completed relative to that LCS are subject to re-analysis.

9.14 Method Blank - A method blank must be analyzed once every 12 hours. If a method blank exhibits contamination above the Quantitation Limit all related sample results must be evaluated. Samples containing the same analytes found in the method blank must be re-analyzed.

9.15 Method Blank:
Detected concentrations < PQL or
Detected concentrations < 10X amount in associated samples

9.16 LCS (LCS):
Recovery within 50-150% (typical)

9.17 MS/MSD:
Recovery within 50-150% (typical)
%RSD <30%

9.18 Surrogate:
Recovery within lab historical limits. Typical limits are 55-140%.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.19 Instrument QC

ICAL: %RSD < 20% or calibration factor >0.990

ICV (second source): Within ±15% of true value

CCV: %D < 15%

Step	*Standards*	*Type*	*Control Limit*	*Frequency*
Method # 8015B/D GRO				
1-5	*5 Point Cal*	*Linear or Ave Cal Factor*	*>0.995 or <20.0 RSD*	*When ICV criteria cannot be met with routine maintainence*
6	*Second Source*	*Midrange Cal*	*</= 15.0 %D*	*After ICAL prior to analysis*
7	*ICV*	*Single Point*	*</= 15.0 %D*	*Daily prior to analysis*
8	*VBLK*	*VOA-free Water*	*<RL for all Target Analytes*	*Daily prior to analysis*
9	*CCV*	*Single Point*	*</= 15.0 %D*	*Every 10 injections*

10.0 Procedure

10.1 Volatile compounds are introduced into the gas chromatograph by purge–and–trap (Method 5030). Method 5030 may be used directly on groundwater samples or low-level contaminated soils and sediments. For medium-level soils and sediments, aqueous extraction, in accordance with this SOP may be necessary prior to purge-and-trap. All method blanks and standards are treated in the same manner as the samples.

10.2 Sample Preparation
10.3 Sample Preparation - Water

10.3.1 Aqueous samples are removed from the storage incubator and allowed to warm to ambient temperature before analysis.

10.3.2 For matrix spike/matrix spike duplicate and matrix spike blank analyses, the sample aliquot is also fortified with the appropriate spike solution.

10.3.3 All appropriate information including sample designations, etc. are recorded in the instrument injection logbook and appropriately transferred to calculation forms/software. Copies of these original analyst observations are included in their entirety in the job/case files.

10.4 Sample Preparation - Soil/Sediment (Med Level)

10.4.1 The sample consists of the entire contents of the sample container. Any supernatant liquids are mixed in to the sample with a clean spatula. Care is taken to minimize time which the sample is exposed to the air.

10.4.2 Approximately 5 grams of sample is weighted into a tared 20 ml vial. Surrogate and/or spiking materials (if appropriate to the particular sample) are added along with 5.0 ml of methanol.

10.4.3 The vial is sealed and shaken for 2 minutes. The contents are allowed to settle or are centrifuged to obtain a clear (not necessarily colorless) aqueous supernatant. Using a clean 1.0 ml syringe, 1000ul of supernatant is drawn off and placed into a 50mls flask of volatile free water, capped and shaken. 44ml of this mixture is poured into a VOA vial, capped and inserted into the tray on the gas chromatograph for purge-and-trap analysis.

10.4.4 Dry weights for soil analysis are determined independently of the procedure but are used in order to report results on a dry weight basis.

10.4.5 All appropriate information including sample weights, and sample designations are recorded in the instrument injection logbook and transferred to calculation forms/software. Copies of these original analyst observations are included in their entirety in the job/case files.

10.5 Instrument Performance Specifications – The gas chromatograph is set up to reflect the following general operating conditions.

Injection B Temp.	250^{o}C
Detector B	250^{o}C
Oven Maximum	250^{o}C
Range 1	2
Range 2	0
Signal 1	Att. 0
Signal 2	Att. 0

10.6 Column conditions reflect the constituents of interest in a particular analysis.

GRO Analysis:

Initial Temperature	50°C
Initial Time	5 min
Rate	10°C/min
Final Temperature	210°C
Final Time	2.5 min
Rate A	20°C/min
Final temperature A	220°C
Final Time A	1.0 min
Equilibrium Time	1.0 min

10.7 Calibration

10.8 A five point calibration curve is established for each compound of interest. The method of continuing calibration verification on a daily basis is acceptable if QC criteria are met. A new initial calibration curve must be run whenever continuing calibration criteria can not be met.

Gasoline Component Standard and Concentrations

Level	2.0 Standard µls Injected	Ng on column
A	1100	250
B	2200	500
C	4400	1000
D	13200	2500
E	22000	5000

10.8.1 Using the unleaded gasoline standard, inject 20ul into 100 mls of volatile free water for a final concentration of 2.0 ng/ml.

10.8.2 For the initial calibration curve to be acceptable measurable response factors at all levels and the %Relative Standard Deviation (RSD) between response factors must be ≤20%.

10.9 For calibration verification (i.e. continuing calibration) of the analytical curve, a 2500 total ng standard must be analyzed every 12 hours sample analyses and at the end of each analysis sequence.

10.9.1 Inject 10.0ul of the unleaded gasoline standard (5000ng/mL) into 100 mls of volatile free water for a final concentration of 0.5. ug/mL. This concentration should yield 2500ng on column.

10.9.2 The continuing calibration verification standard response factor must agree with the initial calibration to within ±15% difference. Project-specific limits may be different from this criteria.

10.10 To obtain retention time windows, three levels from an initial calibration are used to calculate standard deviation and mean for all target compounds and surrogates. The width of the retention time window is + or – 3x the standard deviation of the mean absolute retention time from the 3 levels in the initial calibration. The retention times are updated daily with each ICV.

Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.

Calibration Controls	Sequence	Control Limit
Calibration Standards	5-point (minimum) linearity	≤20% RSD
Cont. Cal. Verif. (CCV)	Prior to / after every 10 injections	Prior to/after every 10 injections
RT Windows (RTW)	Init. CCV determines midpt. of RTW	±3X SD

10.11 Sample Analysis

10.12 Upon establishment of or verification of established instrumental operating conditions, the following instrument maintenance procedures are performed daily.

10.12.1 Check flows and column conditions

10.12.2 Analyze blank(s) before samples and determine system to be clean and free of interferences.

10.12.3 If interferences are present, (1) bake out column at 250°C, and/or (2) cut capillary column at injector end about 6-12 inches.

10.12.4 Log maintenance activities into the instrument maintenance log.

11.0 Calculations / Data Reduction

$$\text{Calibration factor} = \frac{\text{total area of peaks}}{\text{nanograms injected}}$$

$$\text{Percent Difference} = \frac{R1 - R2}{R1} \times 10$$

Where: R1 = Expected recovery in nanograms

R2 = Actual recovery in nanograms

Nanograms on column = $\frac{\text{total area minus surrogate's area}}{\text{Calibration factor}}$

Ug/L = $\frac{\text{nanograms on column}}{\text{ul injected (purged)}}$ X 1000

ug/Kg= medium level soils = $\frac{\text{(nanograms on column X final volume)}}{\text{(dry weight X ul injected)}}$ X 1000

low level soils = $\frac{\text{nanograms on column}}{\text{dry weight}}$

$R = \frac{Xs - Xu}{K} \times 100$

Where: Xs = measured value for spikes sample
Xu = measured value for unspiked sample
K = known value of spike in the sample

% dry weight = $\frac{\text{grams of dry sample}}{\text{grams of sample}}$ X 100

Rounding – for rounding off numbers to the appropriate level of precision, observe the following common rules:

If the figure following those to be retained is less than 5, round down.

If the figure is greater than 5, round up (increase the last digit by one).

If the figure following the last digit to be retained equals 5 (round up if the digit to be retained is odd, and down if that digit is even).

Organic Significant Figures – for volatile and semi volatile results, report analytical results to one significant figure if the value is less than 10, and two significant figures if the value is above 10.

Results must be defined as wet weight or dry weight.

12.0 Method Performance

12.1 The accuracy and precision obtained will be determined by the sample matrix, sample introduction technique, and by the calibration procedure used. Specific acceptance limits for both precision and accuracy are updated annually and are maintained in the TALs system.

12.2 Method Detection Limit Study (MDL)

12.3 Method detection limit studies are performed annually in accordance with 40 CFR, Part 136, Appendix B.

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.4 Demonstration of Capabilities

12.5 A minimum of 4 replicate QC check standards at 20ug/L will be performed by each analyst. The average recovery and standard deviation are keep in TALs and kept with each analyst's training file.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2 Waste Streams Produced by the Method

14.2.1 The following waste streams are produced when this method is carried out. VOA vials containing extracted acidic water and small amounts of methanol. Waste is disposed of in appropriate waste containers, either labeled "A" for aqueous waste or "C" for any solvent other than water.

14.2.1 VOA vials containing extracted soil samples, which will contain small

amounts of methanol. The methanol is decanted off the soil into a "C" waste container. The soil is wrapped in tin foil and placed in the solid waste receptacle. Soils used for dry weight measurements are also disposed of in this manner.

14.2.2 Glass waste such as pipettes and vials are rinsed and disposed of in approved glass receptacles.

15.0 References / Cross-References

15.1 Method D 4128-89, "Standard Practice for Identification of Organic Compounds in Water by Combined Gas Chromatography and Electron Impact Mass Spectrometry", American Society for Testing and Materials.

15.2 Method 8015B, "Non-halogenated Volatile Organics by Gas Chromatography", Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, Revision 2, September 1994.

15.3 Method 8015D, "Non-Halogenated Organics Using GC/FID", Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Fourth Edition, Revision 4, June 2003.

15.4 "Test America's Laboratory Quality Manual", Current Revision

15.5 "Chemical Hygiene Plan", Current Revision

16.0 Method Modifications: N/A

17.0 Attachments NA

18.0 Revision History

Revision 3 dated August 30, 2013

- Changed QA Manager, signature updated
- Changed all references from 8015B to 8015B/D

Revision 2 dated March 29, 2012

- Removed all ELEMENT references
- Changed QA Manager, signature updated.
- Section 7.1 clarification
- Changed MSB references to LCS
- Section 10.4.3 updated soil procedure
- Section 9.13.3 Clarified soil spiking procedure

Revision 1 dated March 18, 2010

- Removed all AIMS references

Revision 0 dated June 9, 2008

- Integration of TestAmerica operations
- Quality Manager change, signature updated

Title: Method 5030C: Purge and Trap

Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):

7/30/13	7/30/13
Denise Giglia Date Department Manager	Kenneth Kasperek Date EHS Manager
7/30/13	7/30/13
Brad Prinzi Date Quality Assurance Manager	Christopher A. Spencer Date Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

This method describes sample preparation and extraction for the analysis of volatile organics by a purge and trap procedure. The gas chromatographic determinative steps are found in Methods 8260C, 624, 524.2, NYSDEC Analytical Services Protocols, and USEPA OLMO4.3.

1.1 Analytes, Matrix(s), and Reporting Limits

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7.3.1 in the Quality Assurance Manual.

2.0 Summary of Method

An inert gas, helium, is bubbled through a sample (solution) at ambient temperature or an elevated temperature depending on analytes and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After sample purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column.

3.0 Definitions

Standard definitions are found in TestAmerica Buffalo's Laboratory Quality Manual.

4.0 Interferences

Purchasing high-quality helium minimizes impurities from the purge gas (helium). The purge and trap system is highly susceptible to carryover from high level samples. Sample lines are flushed with volatile free water after each sampling. The trap is baked at 260 degrees C for a minimum of eight minutes.

The laboratory analyzes weekly volatile holding blanks to ensure an environment free of volatile organic solvent vapors. Methylene chloride can permeate through a septum seal, a trip blank is carried through the sampling and handling protocols to serve as a check on such contamination.

The purge and trap system will also be demonstrated to be clean by the use of Method Blanks and IBLKs. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Unusually high-concentration samples should be followed by an analysis of organic-free reagent water to check for cross-contamination.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the

method to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats, and closed-toe, nonabsorbent shoes are a minimum.

Specific Safety Concerns or Requirements

Special precautions are taken when working with a purge and trap system. Due to the amount of gas utilized by the system, all employees are required to wear approved safety glasses. Parts of the system are under pressure, always allowing for the possibility of shattered glass.

Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

Purge and trap device that consists of three parts.

Sample purge vessels are designed to accept 5ml samples and have a total volume of less than 15 mls. In low level drinking water methods, 25ml sample purge vessels are utilized.

A VOCARB 3000 trap (or similar manufacturers trap) ~30cm long containing the following materials is utilized for all methods:

10cm Carbopack B
6cm Carboxen 1000
1cm Carboxen 1001

The desorber rapidly pre-heats the trap to 245 degrees C and then desorbs at 250 degrees C. The trap is then baked at 260 degrees C.

7.0 Reagents and Standards

Volatile free water for making sample dilutions and method blanks.
Purge and trap grade methanol for standards.

8.0 Sample Collection, Preservation, Shipment and Storage

Samples should be collected in 40 ml capped vials with zero headspace and stored at 4°C +/-2° until time of analysis. Aqueous samples preserved with HCl must be analyzed within 14 days of collection. Aqueous samples not preserved with HCl must be analyzed within 7 days of collection. Soil samples must be analyzed within 14 days of collection. TCLP volatile samples must be tumbled with in 14 days of collection and then analyzed within 14 days of the TCLP extraction.

9.0 Quality Control

A standard, LCS, and MB is analyzed in each run as well as a MS/SD every 20 samples.

9.1 Instrument QC

Instrument Operating Conditions (Suggested)

Purge temperature	<35-40ºC
Desorb Temperature	250ºC
Line Temperature	110ºC
Purge Gas (Helium)	40mL/min.
Purge Total Time	11 min.
Desorb Time	2 min.

Instrument Maintenance

Upon verification of established operating conditions, the following is performed on a sequence basis:

- check purge flow;
- analyze blank to insure system is free of contamination (daily);
- vessel and lines are flushed with volatile free water after each analysis.

Note: System must be leak free. System can be checked by purging 5mL water in sample vessel and capping off vent on purge device. If purge flow stops system is leak free, if purge flow continues (within 2-3 minutes) this means there is a leak within the system. Leak must be located and corrected.

10.0 Procedure

Instrument Operating Conditions (Suggested)

Purge temperature	<35-40ºC
Desorb Temperature	250ºC
Line Temperature	110ºC
Purge Gas (Helium)	40ml/min.
Purge Total Time	11min.
Desorb Time	2min.

10.1 Calibration

See appropriate determinative method(s).

11.0 Calculations

NA

12.0 Method Performance

MDLs are performed yearly, per analytical method, and kept on file with the Quality Department. The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20.7 of the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed.

Waste disposal procedures are incorporated by reference to BF-WM-01. The following waste streams are produced when this method is carried out:

14.1 Acidic material from the auto-sampler: Waste stream must be collected in "A" waste receptacles and neutralized before discharge to a sewer system.

14.2 Methanol waste from rinses and standards: Collect in "C" waste receptacles. In the case of medium level soil extractions, the methanol is decanted off the soil and collected in the "C" receptacle. Waste receptacles are then taken to sample control where they are disposed of properly. Excess samples (acidic and non-acidic). Collect in "A" waste receptacles and are neutralize before disposal into drain/sewer.

14.3 Excess soil sample from medium level extraction: Place in solid waste receptacle. Soils for dry weight measurements are also disposed in this manner.

15.0 References

U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984

U.S. EPA "Method 5030C, Purge and Trap for Aqueous Samples", Test Methods for Evaluating Solid Waste, Volume 1B, Revision 3, May 2003.

16.0 Tables, Diagrams, Flowcharts NA

17.0 Revision History

- Revision 0, July 30, 2013 initial release

Title: ULTRASONIC EXTRACTION (SW846 3550C)

Approval Signatures:

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Approval Date: December 17, 2013

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This SOP describes the laboratory procedure for ultrasonic extraction of nonvolatile and semivolatile organic compounds from solids such as soils, sediments and waste. This SOP includes the procedure for concentration of extracts in preparation for cleanup and/or determinative analysis.

This procedure is not applicable to the medium/high concentration method (individual organic components of greater than 20 mg/kg) as described in the referenced method.

1.1 Analytes, Matrix(s), and Reporting Limits

Refer to analytical methods for analyte lists and reporting limits.

2.0 Summary of Method

A measured amount of sample, usually 30 g, is mixed with anhydrous sodium sulfate to create a free-flowing mixture that is solvent extracted three times using ultrasonic extraction. The extract is dried, concentrated, and relinquished to the analytical department(s) or if necessary, exchanged into a solvent compatible for extract cleanup procedures.

This SOP is based on the following reference method:

SW-846 Method 3550C, Ultrasonic Extraction, Revision 3, February 2007.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment that can cause interference and/or elevated baselines in chromatography. All reagents and solvents used during this procedure should be reagent grade or high purity in order to minimize interference. All glassware must be cleaned in accordance with laboratory SOP BR-EX-017 *Glassware Cleaning Procedure*, and rinsed with acetone and methylene chloride prior to use.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP and should not be used.

Ultrasonic disrupters can produce high intensity noise and must be used in an area with adequate noise protection.

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The KD apparatus has ground glass joints which may become stuck. Technicians must use Kevlar or other cut/puncture resistant gloves when separating stuck joints.

Proper hearing protection is recommended when working with sonicators. Proper hearing protection must be worn if the Decibel value of the sonicators exceeds the regulatory limit. Closing the fume hood sash is recommended to reduce the cavitation sound.

The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds should be prepared in hood.

5.2 Primary Materials Used

Table 1 lists those materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Extraction Equipment

- Ultrasonic Disrupter: Vibra Cell 750-watt Model Dual Output with pulsing capability, 3/4" standard disrupter horn for low level extractions. Sonics and Materials Model Number VC750 or equivalent.
- Sonabox
- 400 mL Glass Beaker: Fisher Scientific or equivalent.
- Filter Funnels: 100mm diameter for filtration/drying, Fisher Scientific or equivalent.
- Filter Paper: No. 54 Whatman 18.5 cm or equivalent

6.2 Extract Concentration (KD Apparatus)

- Concentrator Tube: 10 mL graduated, ChemGlass Catalog Number CG-1316-11 or equivalent.
- Snyder Column: Three ball macro AMK Catalog Number SC2-01 or equivalent.
- Snyder Column: Two ball micro AMK Catalog Number SC3-01 or equivalent.
- Evaporation Flask: 500 mL attached to concentrator tube with clip, AMK Catalog Number KDF-500 or equivalent.
- Boiling Chips: silicon carbide, approximately 10/40 mesh, solvent extracted in methylene chloride, Troemner Catalog Number 133B or equivalent.
- Heating mantle rheostat controlled for water bath capable of temperature control (± 5℃). ChemGlass Catalog Number PL3122 or equivalent.
- Water Bath: capable of temperature control to ±5℃ , Barnstead Corporation Catalog Number HM0500-HS1 or equivalent.
- Solvent Vapor Recovery System: Kontes K-54000-1006, K-547300-000, Ace Glass Catalog Number 6614-30 or equivalent.

6.3 Miscellaneous

- Disposable Glass Pasteur Pipette and Bulb: Fisher Scientific or equivalent.
- Top Loading Balance: Capable of measuring to 0.01 gram accuracy, Mettler Model Number PM4800 or equivalent.
- Teflon and Stainless Steel Spatulas: Fisher Scientific or equivalent.
- 0.5 mL – 2.0 mL Syringes: Hamilton Gastight® Syringes or equivalent.
- 2, 4, 8 & 16 mL vials with PTFE-lined screw caps.

7.0 Reagents and Standards

7.1 Reagents

- Sodium Sulfate, granular anhydrous (Na_2SO_4): J.T. Baker or equivalent. Purify by heating at 400℃ for at least 4 hours.
- Methylene Chloride (CH_2Cl_2): Pesticide Quality, J.T. Baker or equivalent.
- Hexane, (C_6H_{14}): Pesticide Quality, J.T. Baker or equivalent.
- Acetone, ($(CH_3)_2CO$): Pesticide Quality, J.T. Baker or equivalent.
- Nitrogen, Ultra High Purity, Linde or equivalent.

7.1.1 Prepared Reagents

Methylene Chloride/Acetone (1:1): In a 4 L amber glass bottle mix 2 L methylene chloride with 2 L acetone. Store the solution in a fume hood. Assign an expiration date of 6 months from date of preparation unless the parent material expires earlier, in which case, use the earliest expiration date.

Hexane /Acetone (1:1): In a 4 L amber glass bottle mix 2 L hexane with 2 L acetone. Store the solution in a fume hood. Assign an expiration date of 6 months from date of preparation unless the parent material expires earlier, in which case, use the earliest expiration date.

7.2 Standards

Purchase stock standards as certified solutions from commercial vendors. Prepare surrogate and spiking solutions in the laboratory by diluting a known volume of the stock standard solutions in an appropriate solvent. Record the preparation of standard in the LIMS (TALS) module established for this purpose. The recommended formulation for each standard used in this procedure is provided in the analytical method along with the recommended source materials, expiration dates and storage conditions.

Store prepared standard solutions in glass containers at 4℃ or below. Unless otherwise specified, assign an expiration date of 6 months from the date of preparation or in accordance with the expiration date of the parent standard, whichever is sooner.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time[1]	Reference
Soil	Clear or Amber Glass	50 g	≤6℃	14 Days	SW-846

[1]Extraction holding time is determined from sampling date.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Analytical SOP
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Analytical SOP
Matrix Spike(s) MS/MSD	Client Request	See Analytical SOP
Sample Duplicate (SD)	Client Request	See Analytical SOP

9.2 Instrument QC

Refer to the analytical SOP for the determinative test method.

10.0 Procedure

10.1 Instrument Calibration

Check the calibration of the pH meter and the balance each day of use prior to use and record these checks in the logbook designated for this purpose.

Check the tune of the non-self-calibrating sonicator horns prior to use and inspect the horn tip for excessive wear. Attach the converter to the bottom connector. Set the pulsar switch to the off position, the output control to 10 (full power) and the % duty cycle to 50 (energy on 50% of time and off 50% of time). Briefly depress the tune switch and rotate the tuning control clockwise or counterclockwise until a minimum reading of less than 20% is achieved on the power meter. Re-attach the upper converter if using both converters and record the tune calibration in the logbook designated for this purpose.

10.2 Equipment Preparation

Prepare glassware using the procedures described in laboratory SOP BR-EX-017 and rinse with acetone and methylene chloride prior to use. Label the glassware for each field and QC sample clearly and unambiguously during each step of the extraction procedure. Solvent will erase grease pens and “sharpie ink”; so use caution to ensure labels are not obliterated during the procedure.

Assemble a drying funnel for each field and QC sample by placing filter in a 75mm glass funnel then add a sufficient amount of purified granular sodium sulfate to fill the funnel ¾ full. Rinse the funnel with ~30 mL of acetone and ~ 30 mL of methylene chloride each and discard the solvent rinse.

Assemble a KD setup for each field and QC sample by attaching a 10 mL concentrator tube to a 500 mL evaporation flask and attach this to the drying funnel.

10.3 Extraction

Mix sediment samples thoroughly and discard any foreign objects such as sticks, leaves and rocks. Homogenize the sample following the procedures given in laboratory SOP BR-QA-020 Sample Homogenization.

NOTE: The percent dry weight of solid samples is determined by the wet chemistry department following SOP BR-WC-006 Percent Solids Determination.

Label and place a 400 mL beaker on the balance and tare the balance. Weigh out approximately 30 g ±1 g of sample into the beaker and record sample weight to the nearest 0.01 g in the TALS worksheet tab. Use 30 g of purified sodium sulfate for the MB and LCS.

Add the proper type and volume of surrogate solution to each field and QC sample and add the proper type and volume of spike solution to the LCS and MS/MSD. (Refer to the Extraction Condition Spreadsheet for details).

Add a sufficient amount of anhydrous granular sodium sulfate to each sample and mix to create a free-flowing mixture.

Add 100 mL of the extraction solvent mixture to each field and QC sample. For GC Methods (8081 & 8082), use the 1:1 Acetone/Hexane solution as the extraction solvent. For GC/MS and 8015M-DRO, use the 1:1 Methylene Chloride/Acetone solution as the extraction solvent.

Check to ensure the extraction horn is appropriate for the extraction protocol.

Clean the tip of the sonicator horn using a lint-free cloth wetted with acetone. Place the bottom tip of the sonicator horn ~1/2 inch below the solvent, but above the sediment/sodium sulfate mixture layer. Close the hood sash to reduce noise produced by the sonicator.

Set the control knob set at 10 (full power) and with mode switch on Pulse (pulsing energy rather than continuous energy) and percent duty cycle knob set at 50% (energy on 50% of time and off 50% of time) extract ultrasonically for 3 minutes. Watch the extraction to ensure there is very active mixing of the sample and the solvent when the ultrasonic pulse occurs.

Decant the extract into the drying funnel. Repeat the extraction two more times with two additional 100 mL portions of extraction solvent. Decant the solvent between each extraction into the drying funnel/K-D setup. After the final extraction, pour the entire sample into the funnel and rinse with ~30 mL of extraction solvent. Allow the solvent to completely drain through the funnel into the K-D apparatus. Remove the funnel and discard the contents.

Concentrate the extract using K-D technique and when necessary, nitrogen blowdown.

10.4 Extract Concentration (KD Technique)

The following sections describe the procedures for the concentration of extracts. Any of the three techniques described may be used. However some techniques are more efficient than others to achieve the final extract volume. Use the following guidelines to select the concentration technique: For the concentration of volumes greater than 5 mL, use Macro Concentration. To concentrate extract volumes between 5 and 1 mL use Micro Concentration. To concentrate extract volumes below 1 mL, use Nitrogen Blowdown.

10.4.1 Macro Concentration

Macro Snyder Column (K-D)

Add one or two clean boiling chips to the K-D evaporation flask and attach a three-ball Snyder column to the flask. Add ~1 mL of methylene chloride to the top of the column then place the K-D apparatus in a hot water bath (60-70℃) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in hot water vapor.

Attach the solvent vapor recovery glassware to the Snyder column. Adjust the vertical position of the apparatus and check the water bath temperature. The water bath temperature should be between 54.8 – 74.8℃ when methylene chloride is the extraction solvent and 84-89℃ when hexane is the extraction solvent. Higher water bath temperatures may be used so long as the recovery of target analytes is not impacted. The boiling point of each solvent is provided in the following table:

Solvent	Boiling Point	Water Bath Temperature
Hexane	69℃	84 – 89℃
Methylene Chloride	39.8℃	54.8 – 74.8℃

Monitor the concentration and do not let the extract evaporate to dryness. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with solvent.

When the apparent volume of the extract reaches desired amount remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

10.4.2 Micro Concentration

Add one or two clean boiling chips to the concentrator tube and attach a two ball micro-Snyder column to the tube. Place the concentrator tube into the water bath so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the concentrator tube and check the temperature of the water bath to ensure the proper temperature for the extract solvent.

Continuously monitor the distillation process to ensure sample extracts do not evaporate to dryness. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with solvent. Remove setup when desired sample volume is reached.

10.4.3 Nitrogen Blowdown

Nitrogen blow down may be used to concentrate extracts as needed.

Place the concentrator tube in a warm water bath maintained at a temperature of 35℃. Apply a steady stream of nitrogen until the desired final extract volume is achieved. Rinse the internal wall of the concentrator tube several times with the appropriate solvent during the evaporation and ensure the solvent level in the concentrator is positioned such to prevent water condensations. Monitor the concentration carefully and do not allow the extract to evaporate to dryness.

10.5 Extract Preparation & Handling

Transfer the extract to labeled Teflon lined screw cap via and store refrigerated. Complete the batch worksheet and perform primary review.

If the TALS log-in does not include cleanup methods in the method chain and there is reason to believe the extract may require cleanup (color, odor, viscosity, etc.) notify the PM of the situation so he/she can determine if cleanup should be performed prior to analysis.

11.0 Calculations / Data Reduction

11.1 Calculations

Calculations are provided in the analytical SOP for each method parameter.

11.2 Data Review

11.2.1 Primary Review

Review the batch worksheet for correctness and completeness. Record any problems encountered during the extraction process with a nonconformance memo (NCM) and complete the data review checklist.

11.2.2 Secondary Review

Review the batch worksheet for correctness and completeness and to ensure the extraction performed is consistent the SOP and project specifications. Complete the data review checklist.

Print the output worksheets and release extracts and output worksheet to the analytical department or to the next step in the method chain such as extract cleanup.

If the TALS log-in does not include cleanup methods in the method chain and there is reason to believe the extract may require cleanup (color, odor, viscosity, etc.) notify the PM of the situation so he/she can determine if cleanup should be performed prior to analysis.

For additional guidance regarding the laboratory's protocol and required elements for data review refer to laboratory SOP BR-QA-019.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) Study must be determined for each test method associated with this extraction procedure during initial method set-up or prior to the analysis of field samples. The MDLs are verified annually or after major instrument maintenance. The procedure for the determination of MDLs is described in laboratory SOP BR-QA-005.

12.2 Demonstration of Capabilities (DOC)

Each analyst must complete an Initial Demonstration of Capability prior to unsupervised performance of this method.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 *Hazardous Waste*. The following waste streams are produced when this method is carried out.

- Organic Solvents - Satellite container: 55 Gallon Covered and Vented Drum.
- Extracted water samples - Satellite Container: 55 Gallon Covered and Vented Drum.

- Vials containing extracts - Satellite Container: 5 Gallon Covered Bucket (inside fume hood).
- Solid Waste - Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood).

15.0 References / Cross-References

- SW-846 Method 3550C, Revision 3, February 2007.
- Laboratory SOP BR-EX-017
- Laboratory SOP BR-QA-019
- Laboratory SOP BR-QA-005
- Laboratory SOP BR-QA-020
- Laboratory SOP BR-EH-001
- Corporate Environmental Health and Safety Manual

16.0 Method Modifications

There are no modifications of the referenced method.

17.0 Attachments

- Table 1: Primary Materials Used
- Appendix A: Terms and Definitions

18.0 Revision History

BR-EX-008, Revision 10.1

- Title Page: Updated approval signatures and copyright date.
- Section 6.1: Added Ultra High Purity Nitrogen to equipment list.
- Section 10.4: Added guidance for how to select concentration technique.

BR-EX-008, Revision 10

- Title Page: Updated approval signatures and copyright date.
- Section 7.1: Corrected methylene chloride formula to CH_2CI_2

BR-EX-008, Revision 9:

- Title Page: Updated approval signatures and copyright date.
- Section 6.3: Removed mechanical pipette from list.
- Section 10.1: Removed procedure to verify calibration of mechanical pipette.
- Section 10.3: Removed reference to Kimwipe and replaced with lint-free cloth.

BR-EX-008, Revision 8:

- Title Page: Updated method reference and approval signatures
- Section 2.0: Updated method reference to SW-846 3550C.
- Section 10.3: Changed the timing of the addition of surrogate and spike solutions from after the addition of sodium sulfate to before the addition of sodium sulfate.
- Section 15.0: Updated method reference to SW-846 3550C.

BR-EX-008, Revision 7:

- All Sections: Changed references from legacy system to TALS
- All Sections: Changed references from benchsheets to EX conditions spreadsheet
- All Sections: Fixed typographical errors
- Section 5.1: Removed vacuum system paragraph
- Section 6.1: Removed sonicator box
- Section 10.1 Addition of self calibrating sonicator note
- Section 10.3: Replaced sonicator box with fume hood sash

BR-EX-008, Revision 6:

- Section 6.3: Added vials.

Table 1: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Material[1]	**Hazards**	**Exposure Limit[2]**	**Signs and symptoms of exposure**
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.

[1] *Always add acid to water to prevent violent reactions.*
[2] *Exposure limit refers to the OSHA regulatory exposure limit.*

Appendix A: Terms and Definitions

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

Title: Diesel Range Organics by GC/FID (SW-846 8015B)

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Approval Date: January 8, 2013

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This SOP describes the analytical procedure used to determine the concentration of diesel range organics (DRO) in non-potable water, soil, sediment and waste. In the context of this SOP, DRO corresponds to the range of alkanes from C_{10} to C_{28}.

This SOP is applicable to the analytical procedure and does not include instructions for sample preparation or extraction. Sample preparation and extraction procedures are described in laboratory SOPs specific to the extraction technique employed.

This SOP describes the laboratory's routine analytical procedure and QC requirements for the test method.

Program or state specific requirements are not included in the SOP because these requirements do not apply to all jobs, vary between programs, are subject to frequent change, and/or may be mutually exclusive. Project specific or program specific QC requirements may be used in place of, or in addition to the requirements specified in this SOP when necessary.

Certain program requirements, such as Department of Defense (DOD) QSM or state specific requirements may be described in Program Requirement Summary (PRS). The PRS supplements and/or replaces procedures and/or QC requirements specified in this SOP when required. PRS are prepared by the QA department and issued to laboratory staff under controlled distribution to the electronic controlled distribution folder located in QA_Public folder.

The need to comply with the PRS shall be communicated by the PM with a method note or job comment in the TALS backlog. Analysts shall follow the instructions of the PRS when the requirement is specified for a job.

1.1 Analytes, Matrices, and Reporting Limits

This procedure may be used for a variety of matrices including: water, soil, sediment and waste.

The list of target compounds that can be determined from this method along with the associated reporting limits (RL) is provided in Table 1.

2.0 Summary of Method

Following solvent extraction a 2 uL aliquot of extract is injected onto a GC system equipped with a fused silica capillary column and a Flame Ionization Detector (FID). The GC system separates the organic compounds and detection is achieved by the FID.
This SOP is based on the following reference method:

- SW-846 Method 8015B Nonhalogenated Organics using GC/FID as applicable to the analysis of petroleum hydrocarbons including Diesel Range Organics (DRO), Revision 2, December 1996.

If the laboratory procedure is modified from the above reference method, a list of modifications will be provided in Section 16.0 of this SOP.

3.0 Definitions

A list of terms and definitions is provided in Appendix A.

4.0 Interferences

- Contaminated solvents, reagents or equipment can cause interferences. To reduce the occurrence of this type of interference, glassware used during the extraction process must be cleaned thoroughly before use following the procedure given in laboratory SOP BR-EX-017.

- Contamination by carryover is possible whenever high and low concentration samples are analyzed in sequence. To reduce the potential for contamination, set the syringe to rinse several times between injections. Samples are screened prior to analysis so that the proper dilution analysis is performed. Re-analysis is performed if carryover is suspected. Cleaning blanks may also be analyzed between samples at the discretion of the analyst.

- The FID is a non-selective detector, so there is a potential for interference from many non-target compounds.

- The chromatographic conditions in this method can result in significant column bleed and a resulting baseline rise. Baseline subtraction of the column bleed is employed by using the column compensation program on the GC.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst must be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

Table 2 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are instructed to be sure equipment used meets the specification of this SOP.

6.1 Miscellaneous

- Zero Air Generator
- Autosampler Vials, National Scientific or equivalent.
- Hydrogen Generator: Parker Balston
- Volumetric Syringes, Class "A" (10µl, 25µl, 50µl, 100µl, 250µl and 500µl), Hamilton or equivalent.
- Volumetric Flasks, Class "A" – appropriate sizes with ground glass stoppers.
- Helium

6.2 Analytical System

- Computer Hardware/Software: GC Acquisition Platform - VAX 4505 (GVAX) Multichrom V2.11. Data Processing - Hewlett-Packard 9000-series computers, an HP 9000 K200 (Chemsvr5)/ HP-UX 10.20 and Target V3.5 or higher.

- Hewlett Packard 5890 GC equipped with Flame Ionization Detector (FID) and an Auto-Sampler capable of a 2-µl injection split or equivalent.

- GC Column: RXI-5ms (30m x 0.25 mm ID x 0.25µm) or equivalent.

- Equivalent columns may be used so long as elution orders are documented and compound separations are maintained.

7.0 Reagents and Standards

7.1 Reagents

- Acetone, Ultra-Resi Analyzed: J.T. Baker or equivalent.

- Methylene Chloride, Ultra-Resi Analyzed: J.T. Baker or equivalent.

7.2 Standards

Purchase stock standard solutions from commercial vendors and from these prepare calibration and working standards by diluting a known volume of stock standard in an appropriate solvent to the final volume needed to achieve the desired concentration. The recommended formulation for each standard used in this procedure is provided in Appendix B along with the recommended source materials, expiration dates and storage conditions.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are recommendations for minimum sample size, preservation and the method specified holding time requirement:

Matrix	Sample Container	Minimum Sample Size	Preservation	Extract Holding Time	Reference
Water	Glass	1 L	Chilled to 4°C	40 Days	SW-846 8015B
Solid	Glass	50 g	Chilled to 4°C	40 Days	SW-846 8015B

Analytical holding time is determined from date of initiation of extraction.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Table 3
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Table 3
Matrix Spike(s) MS/MSD	Per Client Request	See Table 3
Sample Duplicate (SD)	Per Client Request	See Table 3

NOTE: Matrix spikes and sample duplicates indicate the effect of the sample matrix on precision and accuracy of results using the test method. Information from these QC are relevant only to the client's whose sample was used because matrix effects are sample and site-specific. These QC samples are performed when requested by the client and when the client provides sufficient sample volume. These QC samples are not used to judge laboratory performance. The method blank and LCS serve as the positive and negative controls for method performance by the laboratory. The laboratory does not perform LCS duplicates in lieu of MS/MSD because LCSD does not provide any information regarding matrix effects on precision or accuracy. Precision and bias of the test method in relation to laboratory performance is evaluated through demonstration of capability (DOC) and/or control charts.

9.2 Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initially; when ICV or CCV fail	See Table 3
Second Source Calibration Verification (ICV)	Once, after each ICAL	See Table 3
Continuing Calibration Verification (CCV)	every 10 samples	See Table 3
Retention Time Windows	As Needed	See Table 3

10.0 Procedure

10.1 Instrument Operating Conditions

Install a five meter deactivated guard column into the injection port and connect the guard column to the analytical column using a glass connector. Attach the analytical column to the FID.

The recommended instrument operating conditions are as follows:

Temperature Program:

Initial Temperature	50°C hold for 4 minutes
Program	50°C to 300°C at 20°C/min for 11 minutes
Final Temperature	300°C to 320°C, at 5°C/min, hold for 9.5 minutes
Carrier Gas:	Helium
Injector Temperature:	320°C
Detector Temperature:	330°C

10.2 Retention Time Window Establishment

DRO are distinguished on the basis of the retention time ranges for characteristic components in each type of fuel. The RT range for DRO is set during initial calibration based on retention times of the C_{10} to the C_{28} alkanes in the RT Standard.

The method recommends establishing retention time windows for $C_{10,}$ C_{28} and o-Terphenyl with the installation of each GC column by analysis of three standards over a 72-hour period. The mean RT and Standard Deviation (SD) are determined and the RT window is calculated as the mean RT $\pm$ 3SD. If the SD is <0.01 minutes, a default SD of 0.01 minutes may be used. If this procedure results in RT windows that are too narrow, favoring false negatives, the Technical Manager and the Department Manager may set a default RT window of $\pm$ 0.05 minutes centered around the C_{10} and C_{28} RT markers in the RT Standard and around the o-Terphenyl RT in the mid-level initial calibration standard.

10.3 Instrument Calibration

10.3.1 Initial Calibration (ICAL)

Initial Calibration

Prepare the calibration standards at a minimum of five different concentration levels using the formulations given in Appendix B.

Inject 2 µl of each of the calibration standards onto the system. The data processing system calculates the Calibration Factor (CF), mean CF, and Percent Relative Standard Deviation (%RSD).

The %RSD must be less than or equal to 20% in order to use the mean CF for quantification. If this criterion is not met, use an alternate quantification method for that analyte or correct the problem and repeat the calibration. Once a method of quantification is chosen, the same method of quantification must be used until a new calibration is performed.

Alternate Quantification Option:

Linear Regression: Generate a curve of concentration vs. response for each analyte and calculate the correlation coefficient. The calibration must have a correlation coefficient $r \geq 0.995$.

Non-Linear Regression (2nd Order): Generate a curve of concentration vs. response for each analyte and calculate the coefficient of determination (COD) r^2. The calibration model must have a COD $r^2 \geq 0.99$ and must include a minimum of 6 points.

10.3.2 Retention Time Standard (RT Standard)

Prepare the RT standard using the formulation in Appendix B. Analyze the RT standard to set the RT range.

10.3.3 Second Source Calibration Verification (ICV)

Verify the calibration with a second source standard. The second source standard must be prepared from a different manufacturer than the calibration standards unless only one manufacturer exists, in which case, the second source standard must be from a different lot than the lot used for the calibration standards.

Prepare the ICV using the formulation given in Appendix B. Inject 2 uL of standard onto the analytical system. The recovery of each analyte in the ICV must be within ± 20% of the expected value. The RT of the surrogate (o-Terphenyl) peak must be within the established RT window. If the criteria are not met, correct the problem and reanalyze the ICV. If reanalysis fails, repeat the calibration.

10.3.4 Continuing Calibration Verification (CCV)

Analyze the continuing calibration verification standard (CCV) at a concentration at or below the mid-range of the calibration each day before sample analysis, after every ten sample analyses, and at the end of each analytical sequence. The data system calculates the CF and percent difference for each analyte. The percent difference must be within ±15% and the RT of the surrogate (o-Terphenyl) peak must be within the established RT window.

NOTE: Use one of the mid-range calibration standards for the CCV.

If the results of the CCV are outside established acceptance criteria, a corrective action may be performed. If criteria are not met, stop work and recalibrate. Sample analysis may not proceed until the calibration has been verified.

Data with failing CCV may be usable and reportable under the following conditions:

- If the CCV result is biased high and there are no detections for the failing analytes in the sample, then the non-detects may be reported.

- If the CCV is biased low and the sample results for the failing analytes exceed a maximum regulatory limit or decision level, then the sample results may be reported.

Any sample result associated with a CCV failure must be qualified with a data flag.

NOTE: The conditions under which data may be reported may not be acceptable to all clients and/or regulatory programs and may only be used when not other requirement is specified. Always refer to the program requirements for which work is performed when making the decision to report data without corrective action.

10.3.5 Troubleshooting Guidance

- ICAL Failure: Perform injection port maintenance, install new guard column, check detector ends to see if detector jet has slipped. In extreme cases, install new columns, particularly if the chromatography has degraded as evidenced by peak shapes.

- CCV Failure: Perform Injection port maintenance. If injection port maintenance does not restore CCV, install a new guard column and remove one or more loops from each analytical column.

- Crushed Injection Needle: Replace the needle and check the injection port for obstructions and check the autosampler for misalignment.

- AutoSampler Failure: Reset the auto-sampler.

- Power Failure: Reset run and re-acquire or re-initiate run sequence.

10.4 Sample Preparation

Remove the sample extract from refrigerated storage and let it warm to room temperature.

Transfer ~100 uL of extract into an autosampler vial insert and cap the vial. Place the vials in the autosampler tray in order of the analytical sequence. See the next section for an example order.

Screen the extracts if they appear “dirty”, have a strong odor, or are suspected to contain high levels of DRO or other matrix interferences.

10.5 Sample Analysis

Scan the sample ID’s into the data acquisition program, place the samples on the autosampler in the order they were scanned and initiate the analytical sequence.

An example analytical sequence that includes initial calibration is provided in the following table:

Injection Number	Lab Description	
		Instrument Blank
		Instrument Blank
1	RT Standard	
2	ICAL Level 1	Diesel (100 ppm)
3	ICAL Level 2	Diesel (200 ppm)
4	ICAL Level 3	Diesel (500 ppm)
5	ICAL Level 4	Diesel (750 ppm)
6	ICAL Level 5	Diesel (1000 ppm)
7	ICV	
8-18	10 Samples	
19	CCV	
20-30	10 Injections	

31	CCV	
	Repeat	

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

Target analytes are identified by the processing software using summed peak areas in the RT range established during the initial calibration sequence.

Manual integration of the hydrocarbon envelope is typically required to ensure a straight baseline from C_{10} to $C_{28.}$ The o-Terphenyl peak is skimmed out of the hydrocarbon envelope. All manual integration must be performed in accordance with laboratory SOP BR-QA-006 *Manual Integration*.

11.2 Quantitative Identification

The data system calculates the corrected concentration for each analyte from the calibration curve using the equations given in Appendix C. If sample interference is suspected, or if atypical patterns are observed, the observations are noted in the case narrative.

11.3 Calculations

Example calculations are provided in Appendix C. All calculations are performed by the laboratory's LIMS system, TALS, using equations embedded in the TALS software.

11.4 Data Review

11.4.1 Primary Review

Confirm qualitative and quantitative identification criteria using the criteria provided in Sections 11.1 and 11.2. If the data system does not properly integrate the peaks perform manual integration in accordance with laboratory SOP BR-QA-006.

Upload the data files from the data processing system to the laboratory information management system (TALS). Complete the batch information for standards and reagents and verify and set ICAL and QC sample associations. Review the results and set results to primary, secondary, acceptable or rejected as appropriate.

Dilute and reanalyze samples whose results exceed the calibration range. The dilution analysis should result in a determination within the calibration range, preferably in the upper half of the calibration range. A more concentrated analysis is not necessary unless the project requires it. Dilution analyses may also be performed to minimize matrix interference.

If a sample was analyzed immediately following a high concentration sample, review the results of the sample for any sign of carryover. If carryover is suspected, reanalyze the sample.

Review project documents to ensure requirements are met. If not, immediately notify the project manager (PM) to determine an appropriate course of action.

Create a non-conformance report (NCM) for any calibration, QC and sample data that is reported outside established acceptance criteria and/or schedule necessary corrective action. Set batch to 1st level review and complete the data review checklist.

11.4.2 Secondary Data Review

Verify quantitative and qualitative identification in the initial calibration standards and spot check such for ~15% of the remaining data in the batch.

If manual integrations were performed:

- Review each integration to verify that the integration meets the requirements for manual integration as specified in laboratory SOP BR-QA-006. If an error is suspected or found consult with the analyst that performed the integration and request correction or notify the Department Manager, Technical Manager or QA Manager. Do not "fix" the integration. Reintegration by a secondary data reviewer must not be performed except in limited circumstances as approved by the department supervisor or other laboratory management. If those instances where the secondary reviewer performs the integration, this person is now considered the primary analyst and each integration performed by the secondary reviewer must be subsequently reviewed by a peer analyst or the department supervisor to verify the integration is consistent and compliant with the requirements specified in laboratory SOP BR-QA-006.

- Check to ensure an appropriate technical reason code is provided for each manual integration. Acceptable technical reason codes are provided in laboratory SOP BR-QA-005.

- Print the Manual Integration Summary Report. Document your review of manual integrations on the summary report and obtain any review signatures of integrations performed during secondary review as required.

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Verify that the acceptance criteria are met. If the results do not fall within the established limits verify the recommended corrective actions were performed. If not, initiate corrective actions and/or verify an NCM was created to document the criteria exception. Verify analytical results are qualified accordingly. Set samples to 2nd level review.

Run the LIMS QC Checker, investigate and correct any problems found. Run and review the deliverable. Fix any problems found then set the method chain to lab complete.

11.5 Data Reporting

The report deliverable and the application of data qualifiers is handled by TALS based on the TALS formatter set by the PM for each job.

The following default reporting scheme has been set for this method:

Results above the limit of quantitation (LOQ) or reporting limit (RL) are reported as the value found. Results between the limit of detection (LOD) and the LOQ or RL are reported as estimated. Results less than the LOQ and LOD are reported as non-detects to the LOQ or RL.

Electronic and hardcopy data are maintained as described in laboratory SOP BR-QA-014.

12.0 Method Performance

12.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ)

Establish a LOD and LOQ at initial method set up following the procedures specified in laboratory SOP BR-QA-005. Verify the LOD and LOQ at the frequency established for the method using the procedures specified in same SOP. The frequency of LOD and LOQ verification depends on the frequency of the regulatory program for which the method supports. The frequency requirement is documented in a spreadsheet maintained by the QA Department.

12.2 Demonstration of Capabilities (DOC)

An IDOC is required when there is a change instrument type, personnel or method, and when the method has not been performed by the laboratory or analyst in a 12 month period. Each analyst that performs the test method must complete an IDOC prior to independent analysis of client samples. Each analyst must demonstrate on-going proficiency (ODOC) annually thereafter.

Refer to laboratory SOP BR-QA-011 for DOC requirements and procedures.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of the SOP.

Instrument analysts, prior to independent analysis of client samples, must also have documentation of demonstration of initial proficiency (IDOC) and annual on-going proficiency (ODOC) in their employee training files.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices conducted are consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Vials containing sample extracts: Satellite container: 15 gallon bucket connected to a fume hood.
- Solvent Waste: Satellite container: 1 L glass bottle located in fume hood.

15.0 References / Cross-References

- SW-846 Method 8015B Nonhalogenated Organics using GC/FID as applicable to the analysis of petroleum hydrocarbons including Diesel Range Organics (DRO), Revision 2, December 1996.
- Laboratory SOP BR-QA-005
- Laboratory SOP BR-QA-011
- Laboratory SOP BR-EH-001
- Laboratory SOP BR-QA-014
- Laboratory SOP BR-QA-006
- Laboratory SOP BR-EX-017
- Laboratory Quality Assurance Manual BR-QAM
- Corporate Health and Safety Manual CW-E-M-001

16.0 Method Modifications

This laboratory procedure is modified from the reference method as follows:

Modification Number	Method Reference	Modification
1	8015B (7.6.5)	The method states that second column analysis is required when analytical interferences are indicated. The laboratory does not perform second column analyses for this method. If an analysis has an obvious interference or does not exhibit a typical fuel pattern, the observation is documented in the project narrative.
2	8015B (7.5.1)	The reference method states that calibration verification is performed by analyzing both the fuel standard and the RT standard. The laboratory does not analyze the RT standard with each CCV. The RT standard is only analyzed as part of the initial calibration sequence. RT drift is monitored by the surrogate RT in the CCV.

17.0 Attachments

- Table 1: Routine Target Analyte List & Reporting Limit (RL)
- Table 1A: Accuracy and Precision Limits
- Table 2: Primary Materials Used
- Table 3: QC Summary & Recommended Corrective Action
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Tables
- Appendix C: Equations

18.0 Revision History

BR-GC-004, Revision 14:

- Updated Title Page and Approval Signatures
- Section 9:0: Changed frequency for MS/MSD added explanatory NOTE.
- Section 10.0: Updated column type, temperature program and changed solvent. Changed CCV requirements to match TNI Standard. Clarified the analytical sequence.
- Section 11.0: Updated procedure to match TALS review process.

- Section 12.0: Updated DOC requirements to match TNI standard.
- Appendix B: Removed standard preparation tables and replaced with example formulary reports from TALS Reagent application.

BR-GC-004, Rev. 13

- Converted SOP to company format
- Updated approval signatures
- Added language on RT window establishment
- Removed all references to motor oil
- Updated standard information in Appendix B

Table 1: Routine Target Analyte List & Reporting Limit (RL)

Analyte	Routine Reporting Limit (RL)[1]	
	Water (mg/L)	Soil (mg/Kg)
C10-C28	0.10	6.7

[1]*The RL that can be achieved in a clean matrix assuming 100% solids for soils. Actual RL in sample will vary based on volume/mass extracted, extract final volume, dilution factor, percent moisture or other matrix interferences inherent to the sample.*

Table 1A: Routine Accuracy and Precision Limits[1]

	In-House Limits[2] (%R)		Precision (RPD) (≤)
	Water	Solid	
C10-C28	50-130	55-120	30
o-Terphenyl (Surrogate)	10-180	10-160	NA

[2] *In-House Control Limit Reference: 2012CC_Q2*

Table 2: Primary Materials Used

Material	Hazards	Exposure Limit[1]	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

[1] Exposure limit refers to the OSHA regulatory exposure limit.

Table 3: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	CF: RSD $\leq$ 20% Linear Regression: r $\geq$ 0.995 ($r^2 \geq$ 0.99- 6 POINTS REQUIRED)	Correct problem, reanalyze, repeat calibration.
ICV	After each initial calibration	(% R) ± 20% from expected value o-Terphenyl RT must be within RT window	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Daily before sample analysis, every 10 samples and at the end of the analytical sequence	% Difference or Drift ±15% o-Terphenyl RT must be within RT window	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs pass, otherwise repeat ICAL and all associated samples since last successful CCV. See Section 10.0
MB	One per extraction batch of 20 or fewer samples	Target Analyte < RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer samples	See Table 1A	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD SD	MS/MSD: Per extraction batch SD: Per client request	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate Spike	All field and QC samples	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.

[1]*The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.*

Appendix A: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

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.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration Verification (ICV): solution prepared from a separate source from that which is used to prepare the calibration curve.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are only produced in this range and qualified with the proper data reporting flag when a project requires this type of data reporting.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

Appendix B: Standard Preparation Information

Refer to the Formulary Reports appended to this SOP. The standard formulation reports are printed from the TALS Reagent Module and are attached to the SOP as PDF documents so they are not paginated.

Lab staff should use the formulary reports as the guide for standard preparation. The formulary report provides the name of the parent standard (source reagents); the vendor, the volume used, the solvent, solvent lot number, and the final concentration of the prepared standard. Keep in mind that if the concentration of the source reagents differ from those in the formulary report, the standard preparation procedure may need to be adjusted accordingly.

Prepare the standards using Class A volumetric glassware and glass syringes. Assign an expiration date of 6 months from date of preparation unless the parent standard expires sooner in which case use the earliest expiration date. Store the prepared solutions under refrigeration and protected from light at a temperature of 4ºC (±2). See laboratory SOP BR-QA-002 *Standard Preparation* for further guidance.

Appendix C: Equations

Calibration Factor (CF_x)

$$CF_x = \frac{Peak\ Area_x}{Standard\ concentration_{\ mg/L}}$$

Mean Calibration Factor ($\overline{CF}$)

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where:
n = number of calibration levels

Standard Deviation of the Calibration Factor (SD)

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(CF_i - \overline{CF}\right)2}{n-1}}$$

Where:
n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Calibration Factor

$$\text{Percent RSD} = \frac{SD}{\overline{CF}} \times 100\%$$

Where:
SD = Standard Deviation of the Calibration Factor
$\overline{CF}$ = Mean Calibration Factor

Percent Difference (%D)

$$\%D = \frac{CF_v - \overline{CF}}{\overline{CF}} \times 100\%$$

Where:
CF_v = Calibration Factor from the Continuing Calibration Verification (CCV)

Percent Drift

$$\text{Percent Drift} = \frac{\text{Calculated Concentration - Theoretical Concentration}}{\text{Theortical Concentration}} \text{ X } 100\%$$

Percent Recovery (%R)

$$\%R = \frac{C_s}{C_n} \times 100\%$$

Where:
C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD

$$\%R \text{ for MS/MSD} = \frac{C_s - C_u}{C_n} \times 100\%$$

Where:
C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (%RPD) - form 10

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

Where:
C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration

Extract

$$C_{extract}\,(mg/L) = \frac{\text{Peak Area}}{\overline{CF}}$$

Water

$$C_{sample}\,(mg/L) = C_{extract}\,(mg/L) \times \frac{\text{extract volume (L)}}{\text{sample volume (L)}} \times DF$$

Soil

$$C_{sample}\,(mg/Kg) = C_{extract}\,(mg/L) \times \frac{\text{extract volume (L)}}{\text{sample volume (Kg)}} \times \frac{100}{\%\text{ solids}} \times DF$$

Where:
DF = Extract Dilution Factor. If no dilution was made, DF=1.

SOP Change in Progress Attachment (CIPA)

SOP Number	SOP Title	SOP Revision	SOP Effective Date	CIPA Effective Date
BR-MS-001	SVOCs by GC/MS (SW-846 8270D)	7	04/01/11	06/29/11

The following change(s) are made to this standard operating procedure (SOP). These changes are effective on the CIPA Effective Date. The change to this document is authorized and issued by the laboratory's QA Department.

Page 5 of 31: Section 9.0 Quality Control

Insert the following paragraphs:

Section 9.0 describes the requirements for routine sample and instrument QC. The acceptance criteria and recommended corrective actions specified in the SOP are the criteria used in the absence of project-specific criteria. If the project manager (PM) has specified the use of project specific criteria in the job's method or sample comment notes, use the criteria specified in the project for the evaluation of QC. Always check the method comments in your back log for any special project requirements.

Project-specific reporting limits and control limits for LCS/MS/MSD and ICV are entered into the TALS job by the PM and these limits are automatically used for the evaluation of QC in the TALS batch. Project specific criteria for initial calibrations (ICAL) and continuing calibrations (CCV), and blank evaluations or non-routine QC may not be set on a project-basis in the LIMS system. The LIMS will apply the routine criteria set in the SOP; therefore any time project criteria or frequency is specified for these QC; the evaluation must be performed at the bench. The PM is responsible for communication of any special project requirements to the bench and verification that project specifications were followed.

DoD: If the project is Department of Defense (DoD) and the project specifies use of the QSM acceptance criteria ("F" Tables); the PM will identify the need to follow the "F" tables in the job log-in. The "F" tables are posted to the controlled SOP directory which is accessed using the shortcut located on each laboratory PC or with the following pathway: QA_items\SOPS\SOPs_Controlled_Distribution\Program Summary\DOD.

The DoD program summary includes a copy of the DoD QSM, the "F" tables, which specify required QC, frequency, criteria and corrective actions, "Appendix G" which specifies control limits that are to be used when no other project limits are specified, a copy of the latest version of the QSM and other relevant information such as which methods the laboratory hold DoD ELAP accreditation, the corporate SOP for internal DoD approval, comparisons between different versions of the QSM, and frequently asked questions about the QSM.

Title: SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS (SW-846 8270D)

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Approval Date: March 11, 2011

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This standard operating procedure (SOP) describes the laboratory's analytical procedure used to determine the concentration of semivolatile organic compounds in extracts from a variety of matrices.

This SOP is applicable to the analytical procedure and does not include instructions for sample preparation or extraction. Sample preparation and extraction procedures are described in laboratory SOPs specific to the extraction technique employed.

1.1 Analytes, Matrix(s), and Reporting Limits

This procedure may be used for a variety of matrices including: water, soil, sediment, sludge, and tissue.

The list of target compounds that can be determined from this method along with the associated reporting limit (RL) is provided in Attachment 1.

NOTE: The following problems have been associated with compounds analyzed by this method: Benzidine, 3,3'-Dichlorobenzidine and 4-chloroaniline may be subject to oxidative losses during solvent concentration; hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reactions in acetone solution, and photochemical decomposition; and n-nitrosodiphenylamine decomposes in the gas chromatograph inlet forming diphenylamine and, consequently, is detected as diphenylamine. Likewise, 1,2-Diphenylhydrazine is detected as Azobenzene and surrogate Phenol-d6 is detected as Phenol-d5 due to decomposition during the GC/MS analysis.

2.0 Summary of Method

Following solvent extraction and extract cleanup as needed, 2 uL of extract is injected onto a GC system which uses a temperature program to separate the target compounds which are then detected by a mass spectrometer (MS). Target analytes are identified by comparison of their mass spectra with the electron impact (or electron impact-like) spectra of standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

This procedure is based on the following reference method:

- SW-846 Method 8270D, Revision 4, February 2007.

If the laboratory's procedure is modified from the reference method, a list of these modifications can be found in Section 16.0 of this SOP.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

- Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the

extracted ion current profiles (EICPs). All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source.

- Injection syringes should be adequately flushed with solvent between injections in order to remove all traces of the prior sample.

- Contamination by carryover is possible whenever high and low concentration samples are analyzed in sequence. Samples are screened prior to analysis so that the proper dilution analysis is performed. Re-analysis is performed if carryover is suspected.

- Co-extracted Interferences may include lipids, polymers, copolymers, proteins, natural resins, cellular components, viruses, steroids, and high-molecular weight compounds. GPC, which is size exclusion chromatography, is appropriate for cleanup of these types of polar and non-polar interferences.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile or similar gloves must be used.

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials

section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Miscellaneous

2 mL Autosampler vials with 200 uL inserts, PFTE crimp top, National Scientific or equivalent.
4 mL sample vials with PFTE lined screw top caps, National Scientific or equivalent
Volumetric Syringes, Class “A”, Assorted sizes, Hamilton or equivalent.

6.2 Computer Hardware/Software

- GCMS Acquisition Platform - Hewlett-Packard ChemStation.
- Data Processing - Hewlett-Packard 9000-series computers, HP 9000 K200 (Chemsvr6)/ HP-UX 10.20 and Target V3.5.

6.3 Instrumentation

- SVOA Autosampler: HP7673A™, CTC A200S™ or equivalent.
- Gas Chromatograph: Hewlett-Packard™ 5890 GC, 6890 GC or equivalent.
- Mass Spectrometer: Hewlett-Packard™ 5971 MSD, 5972 MSD, 5973 MSD and 5975 MSD or equivalent.
- Primary Column, Crossbonded, 5% diphenyl – 95% dimethyl polysiloxane, 30 m x 0.25 mmID x 0.25 um film thickness: Restek™ RXi-5ms or equivalent.
- Guard Column: Restek™ Deactivated 5m x 0.25 mm ID or equivalent.
- Column unions: Restek Press-Tights™ or equivalent.
- Injection port liners: Single Goose Neck, borosilicate glass. Restek™ 20799 or equivalent.
- Injection Port Septa: HP™, 11 mm Thermo Red or equivalent.
- Data System: Hewlett-Packard Chem server™, Target 3.5 processing software and Hewlett-Packard ChemStation software for instrument control and acquisition

7.0 Reagents and Standards

7.1 Reagents

- Acetone: Pesticide Residue Analysis Grade, JT Baker or equivalent.
- Methylene Chloride (CH_2Cl_2): Pesticide Quality, J.T. Baker or equivalent.
- Methanol (MeOH): Pesticide Quality, J.T. Baker or equivalent.
- Hexane (CH_3 $(CH_2)_4CH_3$): Pesticide Quality, J.T. Baker or equivalent.
- Toluene ($C_6H_5CH_3$): Pesticide Quality, J.T. Baker or equivalent.

7.2 Standards

Purchase stock standard solutions from commercial vendors. Prepare calibration and working standards by diluting a known volume of the purchased stock standard in an appropriate solvent to the final volume needed to achieve the desired concentration. The recommended formulation for each standard used in this procedure is provided in Appendix B along with the recommended source materials, expiration dates and storage conditions.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method. Listed below are the laboratory recommended minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Tissue	Amber glass jar	30 grams	Chilled to <4°C or frozen	Analytical: 40 days	SW-846 Chapter 4
Soil, Sludge, Sediment	Amber glass jar	50 grams	Chilled to <4°C	Analytical: 40 days	SW-846 Chapter 4
Water	Amber glass bottle	1 Liter	Chilled to <4°C	Analytical: 40 days	SW-846 Chapter 4

Analytical holding time is determined from date of initiation of extraction.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Table 3
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Table 3
Matrix Spike(s) MS/MSD	Client Request	See Table 3
Sample Duplicate (SD)	Client Request	See Table 3

Surrogate spikes are added to all field and QC samples before preparation and/or analysis to assess the effect of the sample matrix on the accuracy of the method in the specific sample matrix.

Internal standards are added to all field and QC samples prior to analysis.

9.2 Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
DFTPP	Daily	See Table 3
Initial Calibration (ICAL)	Initially; when ICV or CCV fail	See Section 10.0
Second Source Calibration Verification (ICV)	Once, after each ICAL	See Table 3
Continuing Calibration Verification (CCV)	Daily, following a passing DFTPP	See Section 10.0

10.0 Procedure

10.1 Instrument Operating Conditions

Gas Chromatography/Mass Spectrometry: Set the instrument to acquire and store analytical data over the nominal mass range of 35-500 atomic mass units (amu) with a total cycle time (including scan overhead time) of one second per scan at 70 electron volts. Adjust the cycle time to measure five or more spectra during the elution of each GC peak.

A typical GC temperature program used by the laboratory is as follows:

Initial Temperature:	40°C for 2 minutes.
Temperature Program:	40°C to 320°C at 14°/min.
Final Temperature:	320°C for 5.6 min or until Benzo (g,h,i) perylene has eluted
Injector Temperature:	250°C
Transfer Line Temperature:	300°C
Injector:	Grob-like, splitless
Injection volume:	2 µL
Carrier Gas:	Helium

10.2 Tune Standard (DFTPP)

To initiate the analytical sequence inject a 2 µL aliquot of DFTPP (25 µg/mL) into the GC. The data processing system acquires and averages three scans (apex scan, scan prior, and scan following) and performs background subtraction of the single scan prior to the elution of the DFTPP. The results of the DFTPP must meet the tune criteria given in Table 2 before further analysis can proceed. If criteria are not met, retune the instrument.

The 12-hour analytical window begins at the time of injection of the tune standard. The injection of all samples and standards must be within 12 hours of the injection of the DFTPP. After 12 hours has elapsed initiate a new analytical window with analysis of another tune standard.

10.3 Column Performance and Injection Port Inertness Check

Evaluate the tune standard for GC column performance and injection port inertness. The degradation of DDT to DDE and DDD should not exceed 20 and benzidine and pentachlorophenol should be present at their normal responses and should not exceed a tailing factor of 2. If degradation is excessive and/or poor chromatography is noted perform instrument maintenance.

10.4 Instrument Calibration

10.4.1 Initial Calibration (ICAL)

Calibrate the instrument with a minimum of five calibration standards for each target analyte at concentrations that span the working range of the method. To accomplish this for the complete target analyte list, analyze eight ICAL standards at the recommended concentrations of 10, 25, 40, 50, 60, 70, 80, and 160 ng/uL. These values are equivalent to an on column concentration of 20, 50, 80, 100, 120, 140, 160, and 320 ng per 2 uL injection. Although eight standards are analyzed, not every standard is used for the calibration of each analyte. Each analyte has been assigned to an analyte group that includes a calibration range of at least five levels. The analyte group associations for each target analyte are provided in Table 1; the calibration range for each analyte group is as follows:

- Group A: This analyte group is associated with a seven point calibration curve. The calibration range is 10 to 80 ng/uL or 20 to 160 ng on column. The limit of quantitation (LOQ) for this group of analytes is 10 ng/uL (20 ng on column).

- Group B: This analyte group is associated with a six point calibration curve. The calibration range is 25 to 80 ng/uL or 50 to 160 ng on column. The limit of quantitation (LOQ) for this group of analytes is 25 ng/uL(50 ng on column).

- Group C: This analyte group is associated with a seven point calibration curve. The calibration range is 25 to 160 ng/uL or 50 to 320 ng on column. The limit of quantitation (LOQ) for this group of analytes is 25 ng/uL(50 ng on column).

Prepare each calibration standards in an autosampler vial using the formulations found in Appendix B. The final internal standard concentration in extract will be 20 ng/uL.

Inject 2 uL of each standard onto the instrument system. The data processing system acquires and calculates the mean Response Factor (RF), the Standard Deviation (SD) and the Percent Relative Standard Deviation (%RSD) for each analyte and surrogate compound. See Appendix C for the equations.

The following criteria must be met for a calibration to be considered acceptable. If criteria are not met, perform corrective action prior to further analysis. Recommended corrective actions are provided in Table 3.

- The minimum response factor (RF) must listed in the tables in Attachment 1 must be met for each analyte in each level of the ICAL. If the RSD is not met for any analyte, analyzed an RLCCV in each analytical batch to demonstrate sensitivity at the quantitation limit and a NCM is required to document the situation.

- The %RSD for each analyte must be less than 20%. If this criterion is not met for any analyte an alternative curve fit must be used for the analytes that fail criteria as follows:

  Use linear regression as the alternative curve fit. The regression coefficient (r^2) must be greater than 0.990 and the read back of the ICAL standard equal to the RL for each analyte must be within 30% of the expected value. To obtain the percent recovery, quant the low standard as a sample. If this criterion is not met, the curve may be evaluated using a quadratic curve or the compound may be quantitated using average response factor but if minimum RF is used the compound must be reported as estimated and the situation must

be documented with an NCM. If quadratic fit is used, the regression coefficient (r^2) must be greater than 0.990.

If an alternative curve fit is not performed; all sample results associated with the analyte that failed the %RSD criteria must be qualified and reported as estimated values.

- If more than 10% of the analytes in the calibration do not meet both %RSD criteria and the (r^2) criteria, the instrument must be recalibrated.

Note: SW-846 does not specify a cap for an individual analyte's %RSD. However, no individual analyte should exceed 60% RSD. If any analyte exceeds 60%RSD, the instrument should be recalibrated.

10.4.2 Second Source Calibration Verification (ICV)

Verify the calibration with a second source standard. The second source standard must be prepared from a different manufacturer than the calibration standards unless only one manufacturer exists, in which case, the second source standard must be from a different lot than the lot used for the calibration standards.

Prepare the ICV solution (50 ng/uL) using the formulations provided in found in Appendix B. The final internal standard concentration in extract will be 20 ng/uL. Inject 2 uL of the ICV onto the instrument system. Acquire the data and evaluate the results.

The percent recovery of the ICV must be ±30% of the expected value for each analyte. If this criterion is not met, correct the problem and reanalyze the ICV. If the reanalysis fails, remake the calibration standards and recalibrate.

NOTE: If after successful analysis of the ICV time remains in the 12-hour analytical window samples may be analyzed without analysis of a CCV; otherwise a CCV must be performed.

10.4.3 Continuing Calibration Verification (CCV)

Analyze a continuing calibration verification standard (CCV) immediately after the daily tune standard or the ICV. The concentration of the CCV should be at or near the mid-point of the calibration range for each analyte.

Inject 2 uL of the prepared CCV standard(s), acquire the data and evaluate the results. The data processing system calculates the RF and percent difference or drift for each target analyte and surrogate.

Evaluate the minimum RF against those listed in the table provided in Attachment 1. If criteria are not met, perform corrective action. Possible causes may include degradation of standards, injection port inlet contamination and/or active sites in the column or in the chromatographic system.

The %D must be <20% for 80% of the analytes included in the calibration. If more than 20% of anlaytes do not meet criteria, calibration verification fails and reanalysis is required. Note: the laboratory may set wider %D criteria for poor performing analytes but the number of analytes

with criteria greater than 20%D must not exceed 10% of the total number of analytes included in the calibration.

If less than 20% of analytes in the CCV exceed the %D criteria, analyze an RLCCV (CCV at the concentration of the RL). Analytes that do not meet the %D criteria may be reported if those anlaytes are qualitatively detected in the RLCCV. Report analytical results as follows:

If the analyte that does not meet criteria in the CCV is not detected in the field sample; report the analytical result for the analyte without qualification.

If the analyte that does not meet criteria in the CCV is detected in the filed, report the analytical result as an estimated value and document the exception with a NCM.

Troubleshooting:

Check the following items in case of calibration failures:

- Benzidine is subject to oxidative loss during extraction and chromatographs poorly, injection port and/or column maintenance may be required.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet, injection port and/or column maintenance may be required.
- N-nitrosodiphenylamine decomposes in the inlet and cannot be separated from diphenylamine. Compound is reported as N-nitrosodiphenylamine.
- 1,2-Diphenylhydrazine decomposes to Azobenzene in the analytical portion of the procedure and as such is reported as Azobenzene.
- Acid compounds are subject erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material, injection port and/or column maintenance may be required.
- Loss of sensitivity for higher boiling compounds and internal standards may be indicative of a leak at the inlet. Replace septa and/or re-tighten lower inlet connection.
- Carryover contamination may indicate empty rinse vials.

10.5 Screen Procedure

At the discretion of the laboratory, sample extracts may be screened prior to analysis with a GC-FID or a GC/MS system. To calibrate the screen instrument: analyze an instrument blank, a surrogate standards that includes surrogates at a concentration of 5 ng/uL, and a standard that contains target analytes at a concentration of 8 ng/ uL.

10.6 Analysis

Remove the extracts from storage and let them warm to room temperature. Transfer 100 uL of extract to a 1 mL auto-sampler vial with insert. Add 4 uL of the internal standard solution to each vial and seal the vial with a PTFE lined crimp top cap. If a different extract volume is used (e.g. 50 uL), adjust the internal standard volume proportionately (2 uL of IS).

If the extract was screened and the screen results indicate a primary dilution is required, dilute the extract in methylene chloride. If the relative volumes needed for a single dilution step exceed the accuracy of the syringes, perform serial dilutions. For example, if a sample requires a 0.1% analysis in order to have target constituents within the upper half of the calibrated range, 0.1 uL of a 100 uL extract aliquot is needed to perform the dilution but the

gradations of the syringe are to 0.2 uL. In this instances, perform a serial dilution of 1:100 (1.0%) and 10:100 (10%) to achieve an analysis concentration of 0.1%.

Arrange the samples in a sequence that begins with the calibration standards followed by the analysis of QC samples, field samples, and continuing calibration verification standards (CCVs).

An example analytical sequence for our routine compound list that includes initial calibration (ICAL) is provided below.

Injection Number	Lab Description
1	DFTPP Tune Standard
2	ICAL 7 (SSTD 160)
3	ICAL 6 (SSTD 140)
4	ICAL 5 (SSTD 120)
5	ICAL 4 (SSTD 100)
6	ICAL 3 (SSTD 080)
7	ICAL 2 (SSTD 050)
8	ICAL 1 (SSTD 020)
9	ICAL 8 (SSTD 320)
10	ICV (SSTD 100 ICV)*
11	DFTPP Tune Standard
12	CCAL 3 (SSTD 080)
	12 hour window of samples
1	DFTPP Tune Standard
2	CCAL 3 (SSTD 080)
	12 hour window of samples
	Continue until a new calibration is needed.

*If additional analytes are requested, a separate calibration is analyzed after the initial calibration noted above and before ICV and sample analysis begins.

Enter the sample ID's into the data acquisition program in the order the samples were placed in the autosampler and initiate the analytical sequence.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

The data system tentatively identifies target analytes by comparing the retention time of the peaks to a window set around the daily calibration standard, and searches in that area for the primary and up to two secondary ions characteristic of the target analyte. Tentative identifications made by the computer are reviewed and either accepted or rejected by the analyst using the following criteria:

- The target analyte is identified by comparison of its background subtracted mass spectrum to a reference spectrum in the user-created database. In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and their relative abundances should agree within 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10-50%. Some

ions, particularly the molecular ion, are of special importance if a tentative identification is to be made, and should be evaluated even if they are below 10% relative abundance.

- The GC retention time for the target analyte should be within 0.06 RRT units of the daily standard.

Identification is hindered when components are not chromatographically resolved from interfering analyte peaks or non-target constituents (background). When chromatographic peaks obviously indicate contribution from more than one component (broadened peak with shoulder(s) or a valley between two or more maxima), examine the EICPs to select the appropriate analyte spectra over the entire peak and use selective background subtraction in order to positively identify target analytes and account for extraneous ions. For coeluting compounds, the identification criteria will be met, but the analyte spectrum will contain extraneous ions contributed by the coeluting compound.

Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different GC retention times. Acceptable resolution is achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks. Otherwise, structural isomers are identified as isomeric pairs.

Complex environmental matrices, baseline upsets, coelution and peak shape variation can complicate automatic data system integration causing inaccurate and/or missed identification that should be corrected with manual integration. To assure accurate qualitative identification, optimize the data system integration parameters to ensure consistency in integration between standards and sample and evaluate each chromatogram to verify the identification for each target analyte.

11.1.1 Tentatively Identified Compounds (TICs)

If TICs are requested, perform the library search, and visually compare the sample spectra with the nearest library search and assign a tentative identification. The library search should not include peaks that are <10% of the nearest internal standard, target analytes, or peaks that elute earlier than 30 seconds before the first target analyte.

Use the following criteria to qualitatively identify the TIC compounds:

- Relative intensities of ions greater than 10% of the most abundant ion in the reference spectrum should be present in the sample spectrum.

- The relative intensities of the major ions should agree within ± 20%.

- Molecular ions present in the reference spectrum should be present in the sample spectrum.

- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

TICs identified by the automated processing system with a quality match of 85% or greater may be left as identified by the system. TICs with matches less than 85% may be marked as “Unknown”. Further compound classification is <u>not</u> routinely performed.

11.2 Quantitative Identification

The data system quantifies the concentration of the target compound based on the integrated abundance of the characteristic ion from the EICP using the equations given in Appendix C. If there is matrix interference with the primary ion, a secondary ion may be used for quantification by calculating a mean RF factor for that ion and using that ion to quantify the analyte in the sample. When secondary ion calculations are performed, initiate a nonconformance memo (NCM) to ensure the quantitation approach is reported in the project narrative.
The data system calculates TIC concentrations using an RF of 1.00 and the equations given in Appendix C.

11.3 Calculations

See Appendix C.

11.4 Data Review

11.4.1 Primary Review

Review project documents to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Confirm qualitative and quantitative identification criteria using the criteria provided in Sections 11.1 and 11.2. If the data system does not properly integrate the peaks perform and document manual integration in accordance with laboratory SOP BR-QA-006.

Upload the data files from the data processing system to the laboratory information management system (TALS). Complete the batch information for standards and reagents and verify ICAL and QC sample associations. Review the results and set results to primary, secondary, acceptable or rejected as appropriate. Dilute and reanalyze samples whose results exceed the calibration range. The dilution analysis should result in a determination within the calibration range, preferably in the upper half of the calibration range. A more concentrated analysis is not necessary unless the project requires it. Dilution analyses may be performed to minimize matrix interference.

If a sample was analyzed immediately following a high concentration sample, review the results of the sample for any sign of carryover. If carryover is suspected, reanalyze the sample.

Create a non-conformance report (NCM) for any calibration, QC and sample data that is reported outside established acceptance criteria and/or schedule necessary corrective action. Set batch to 1st level review and complete the data review checklist.

The following analytes have been identified as poor performing analytes in some matrices based on statistical data. Consequently corrective action will not be taken when LCS or MS/MSD recovery is not within established limits for these analytes. Initiate a NCM to document the exceedance and indicate poor performing analyte as the justification for not taking corrective action.

Poor Performing Analytes

Analyte	
Water	**Soil**

4-Nitrophenol	3,3'-Dichlorobenzidine
Benzoic Acid	4-Chloroaniline
Phenol	Benzoic Acid
Phenol-d5	Aniline
Benzidine	Benzidine
2-Chlorobenzoic Acid	2-Chlorobenzaldehyde
Hexachlorocyclopentadiene	2-Chlorobenzoic Acid
Pyridine	3-Chlorobenzaldehyde
	4-Chlorobenzaldehyde
	Pyridine

11.4.2 Secondary Data Review

Verify quantitative and qualitative identification in the initial calibration standards and spot check such for ~15% of the remaining data in the batch.

If manual integrations were performed:

- Review each integration to verify that the integration meets the requirements for manual integration as specified in laboratory SOP BR-QA-006. If an error is suspected or found consult with the analyst that performed the integration analyst and request correction or notify the Department Manager, Technical Director or QA Manager. Do not "fix" the integration. Reintegration by a secondary data reviewer must not be performed except in limited circumstances as approved by the department supervisor or other laboratory management. If those instances where the secondary reviewer performs the integration, this person is now considered the primary analyst and each integration performed by the secondary reviewer must be subsequently reviewed by a peer analyst or the department supervisor to verify the integration is consistent and compliant with the requirements specified in laboratory SOP BR-QA-006.

- Check to ensure an appropriate technical reason code is provided for each manual integration. Acceptable technical reason codes are provided in laboratory SOP BR-QA-005.

Review project documents to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Verify that the acceptance criteria for the calibration and QC items listed in Table 1 were met. If the results do not fall within the established limits verify the recommended corrective actions were performed. If not, initiate corrective actions and/or verify an NCM was created to document the criteria exception. Verify analytical results are qualified accordingly. Set batch to 2nd level review and complete the data review checklist.

11.5 Data Reporting

The report format, application of data qualifiers and creation of the data deliverable is performed by the LIMS using the formatter set by the project manager during log-in.

Records of electronic and hardcopy data are maintained as described in laboratory SOP BR-QA-014.

12.0 Method Performance

12.1 Limit of Detection (LOD) and Limit of Quantitation

Establish a LOD and LOQ at initial method set up following the procedures specified in laboratory SOP BR-QA-005. Verify the LOD and LOQ at the frequency established for the method using the procedures specified in same SOP. The frequency of LOD and LOQ verification depends on the strictest frequency of the regulatory program for which the method supports. The frequency requirement is documented in a spreadsheet maintained by the QA Department.

12.2 Demonstration of Capabilities (DOC)

Perform a method demonstration of capability at initial set-up and when time there is a significant change in instrumentation or procedure.

Each analyst that performs the analytical procedure must complete an initial demonstration of capability (IDOC) prior to independent analysis of client samples. Each analyst must demonstrate on-going proficiency (ODOC) annually thereafter. DOC procedures are further described in the laboratory's quality system manual (QAM) and in the laboratory SOP BR-QA-011.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

Instrument analysts, prior to independent analysis of client samples, must also have documentation of demonstration of initial proficiency (IDOC) and annual on-going proficiency (ODOC) in their employee training files.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 *Hazardous Waste*. The following waste streams are produced when this method is carried out.

- Vials containing sample extracts - Satellite Container: 30 gallon waste drum
- Methylene Chloride solvent waste - Satellite Container: 40mL vials and 4L bottle.

15.0 References / Cross-References

- Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (Method 8270D), Revision 4, February 2007
- Laboratory SOP BR-QA-005
- Laboratory SOP BR-QA-011
- Laboratory SOP BR-EH-001
- Laboratory SOP BR-QA-014
- Laboratory SOP BR-QA-006 Manual Integration Requirements
- Laboratory Quality Assurance Manual (QAM)
- DoD QSM

16.0 Method Modifications

Modification Number	Method Reference	Modification
1	8270D Sec. 7.4.2 and 7.7.2	The laboratory does not store standards at 6 °C (–10 °C recommended). All standards are stored at the recommendations of the manufacturer prior to initial use and then at 4 °C once prepared.
2	8270D Sec. 8.2	The laboratory does not store sample extracts at 6 °C. All extracts are stored at 4 °C once prepared.
3	8270D Sec. 11.5.2	4uL of internal standard is added to 100uL of sample extract just prior to analysis.

17.0 Attachments

- Table 1: Primary Materials Used
- Table 2: Tune Standard Criteria
- Table 3: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action
- Appendix A: Standard Terms and Definitions
- Appendix B: Standard Preparation Tables
- Appendix C: Equations
- Attachment 1: SW-846 8270D Method Information

18.0 Revision History

BR-MS-001, Revision 7:

Title Page: Updated method reference and approval signatures
Section 1: Updated method reference
Section 10.0: Updated criteria for ICAL and CCV to conform to 8270D requirements.
Section 15.0: Updated method reference.
Section 16.0: Updated method reference.
Section 18.0: Removed tables to Attachment 1
Table 3: Updated acceptance criteria or ICAL and CCV

Table 1: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.

[1] Always add acid to water to prevent violent reactions.
[2] Exposure limit refers to the OSHA regulatory exposure limit.

Table 2: DFTPP Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
51	10-80% of Base Peak
68	<2% of mass 69
69	Present
70	<2% of mass 69
127	10-80% of Base Peak
197	<2% of mass 198
198	Base peak, or >50% of mass 442
199	5-9% of mass 198
275	10-60% of Base Peak
365	>1% of mass 198
441	Present but less than 24% of mass 442
442	Base peak, or >50% of mass 198
443	15-24% of mass 442

Table 3: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC	Minimum Frequency	Acceptance Criteria	Recommended Corrective Action[1]
Tune Standard	Prior to initial calibration and every 12 hours	See Table 4	Reanalyze, retune mass spectrometer; no samples may be analyzed without a valid tune.
Breakdown Check	Daily prior to sample analysis	Degradation of DDT should not exceed 20%	If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to break of the first 6 to 12 in. of the capillary column. The use of a guard column between the injection port and the analytical column may help prolong analytical column performance life.
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	See Section 10.0	Correct problem then repeat calibration, no samples may be analyzed until criteria are met.
ICV	Immediately after each initial calibration	% Recovery within ±30% of expected value	Correct problem and verify second source standard. Reanalyze. If that fails, repeat initial calibration; no samples should be analyzed without an acceptable ICV.
CCV	Beginning of each 12-hour window after the tune standard	See Section 10.0	Correct problem then repeat CCV, no samples may be analyzed until criteria are met. If problem cannot be corrected by other measures, a new calibration must be generated.
MB	One per extraction batch of 20 or fewer samples	Routine: < RL (See Attachment 1)	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer samples	%R within limits (See Attachment 1)	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD	One per extraction batch of 20 or less samples.	%R within limits (See Attachment 1)	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate Standard	All field and QC samples	%R within limits (See Attachment 1)	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.
Internal Standard	All field and QC samples	EICP area between -50% to +100% of area of daily calibration internal standard area. RT ± 30 seconds from RT of midpoint of ICAL.	Inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

Appendix A: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

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.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Internal Standard: a known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

Appendix B: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration of the stock standard is different than those noted in this table, adjust the standard preparation formulation accordingly. Unless otherwise specified, prepare the standard solutions in methylene chloride using Class A volumetric glassware and Hamilton syringes. Unless otherwise specified for a standard solution, assign an expiration date of 6 months from date of preparation unless the parent standard expires sooner in which case use the earliest expiration date. Record the preparation of the solutions in the LIMS reagent module.
See laboratory SOP BR-QA-002 *Standard Preparation* for further guidance.

ISTD Mix

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/mL)	Volume Added (uL)	Final Volume (mL)	Final Conc. (ug/mL)
SV Internal Standard Mix	Restek (31006)	4000	1000	8.0	500

Decafluorotriphenylphosphine (DFTPP) Solution

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/mL)	Volume Added (uL)	Final Volume (mL)	Final Concentration (ug/mL)
SV GC/MS Tuning Standard	Ultra Scientific (GCM-150)	1000	400	16.0	25

8270 Calibration Stock Solution

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/mL)	Volume Added (mL)	Final Volume (uL)	Final Concentration (ug/mL)
8270 MegaMix	Restek (31850)	1000	1240	7440	167
Custom Rev. AP9 Adds w/ Benzoic Acid	Restek (564928)	1000	1240		

Benzidines Added Solution

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/mL)	Volume Added (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 Benzidines Mixture	Restek (31852)	2000	1000	2.0	1000

OLM04 Added Solution

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/mL)	Volume Added (uL)	Final Volume (mL)	Final Concentration (ug/mL)
CLP SOW OLM 04 Semi-volatiles Mix	Supelco/Sigma-Aldrich (47514-U)	2000	1250	2.5	1000

320 Acid Solution

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/L)	Volume Added (uL)	Final Volume (mL)	Final Concentration (mg/L)
Custom 2-Chlorobenzoic Acid	Restek (562264)	5000	800	2.0	2000
Benzoic Acid	Restek (31879)	2000	1000		1000

MASOGD Added Solution

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/L)	Volume Added (uL)	Final Volume (mL)	Final Concentration (mg/L)
Custom 2-Chlorobenzoic Acid	Restek (562264)	5000	128	4.0	160
Custom MMR 1	Restek (562265)	1000	320		80
Custom MMR 2	Restek (562266)	1000	320		

Appendix IX Mix A

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/mL)	Volume Added (uL)	Final Volume (mL)	Final Concentration (ug/mL)
Acid Surrogate Standard Mix (3/90)	Restek (31073)	7500	42.67	4.0	80
8270 Calibration Mix #1	Restek (31618)	2000	160		
8270 Calibration Mix #3	Restek (31620)	2000	160		
8270 Calibration Mix #4	Restek (31621)	2000	160		
8270 Calibration Mix #6	Restek (31623)	2000	160		
Custom AP9 Mix A	Restek (565078)	2000	160		

Appendix IX Mix B

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/mL)	Volume Added (uL)	Final Volume (mL)	Final Concentration (ug/mL)
B/N Surrogate Standard Mix (3/90)	Restek (31072)	5000	64	4.0	80
8270 Calibration Mix #2	Restek (31619)	2000	160		
8270 Calibration Mix #5	Restek (31622)	2000	160		
Appendix IX Mix #1	Restek (31625)	2000	160		
Ethyl Methacrylate	Restek (30289)	2000	160		
CLP SOW OLM 04 Semi-volatiles Mix	Supelco/Sigma-Aldrich (47514-U)	2000	160		

8270 ICV Solution

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/mL)	Volume Added (uL)	Final Volume (mL)	Final Concentration (ug/mL)
BNA Mix	NSI Solutions (C-701)	1000	400	4.0	100
Benzoic Acid	NSI Solutions (541)	5000	80		
Benzo(e)pyrene	Absolute (71016)	1000	400		
Perylene	Absolute (70471)	1000	400		

Benzidines Added ICV Solution

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/mL)	Volume Added (uL)	Final Volume (uL)	Final Concentration (ug/mL)
Benzidines Mix	NSI Solutions (C-402)	2000	1250	2500	1000

OLMO4 Added ICV Solution

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/mL)	Volume Added (uL)	Final Volume (mL)	Final Concentration (ug/mL)
CLP BNA Additionals Mix	NSI Solutions (C-601)	2000	1000	2.0	1000

8270C Routine Working Calibration Standard Preparation

Sample Type	Final Conc. (ng/uL)	Calibration Stock (uL)	Benzidines Added (uL)	Puf/Surrogate Added (uL)	OLM04 Added (uL)	ISTD (uL)	MeCl2 (uL)	Final Volume (uL)
CAL Level 1	10	6	1	1	1	4.0	91	104
CAL Level 2	25	15	2.5	2.5	2.5	4.0	77.5	104
CAL Level 3	40	24	4	4	4	4.0	64	104
CAL Level 4	50	30	5	5	5	4.0	55	104
CAL Level 5	60	36	6	6	6	4.0	46	104
CAL Level 6	70	42	7	7	7	4.0	37	104
CAL Level 7	80	48	8	8	8	4.0	28	104

Appendix IX & MASOGD Working Standard Preparation

Sample Type	Final Concentration (ng/uL)	80ppm CAL Std (uL)	ISTD (uL)	MeCl2 (uL)	Final Volume (uL)
CAL Level 1	10	12.5	4.0	87.5	104
CAL Level 2	25	30.3	4.0	69.7	104
CAL Level 3	40	50	4.0	50	104
CAL Level 4	50	62.5	4.0	37.5	104
CAL Level 5	60	75	4.0	25	104
CAL Level 6	70	87.5	4.0	12.5	104
CAL Level 7	80	100	4.0	0	104

320 Acid Working Standard Preparation

Sample Type	Final Concentration (ng/uL)	320 Acid Solution (uL)	ISTD (uL)	MeCl2 (uL)	Final Volume (uL)
CAL Level 8	320	16	4.0	84	104

8270C Routine Working ICV Standard Preparation

Sample Type	Final Conc. (ng/uL)	8270C ICV Solution (uL)	Benzidines ICV (uL)	OLM04 ICV (uL)	ISTD (uL)	MeCl2 (uL)	Final Volume (uL)
ICV	50	50	5	5	4.0	40	104

SURROGATE AND SPIKE STANDARDS

The following standards are used to spike field and QC samples prior to extraction. Assign an expiration date of 6 months from date prepared unless the stock standard expires sooner in which case use the earliest expiration date. Store the prepared solutions under refrigeration and protected from light at a temperature of 4ºC (±2).

8270 Matrix Spike

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/mL)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 MegaMix	Restek (31850)	1000	5.0	100	50
Custom Rev. AP9 Adds w/ Benzoic Acid	Restek (564928)	1000	5.0		

Solvent: Acetone

Benzidines Matrix Spike

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/mL)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/mL)
Benzidines Mixture	Ultra Scientific #US-105N	2000	6.25	250	50

Solvent: Acetone

OLM04 Matrix Spike

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/mL)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 Benzidines Mixture	Restek (31852)	2000	6.25	250	50

Solvent: Acetone

Benzoic Acid Matrix Spike

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/mL)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/mL)
Benzoic Acid	NSI Solutions (W-541)	5000	1.0	50	100

Solvent: Acetone

BNA Surrogate Spike

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/mL)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/mL)
Acid Surrogate Standard Mix (3/90)	Restek (31073)	7500	9.0	450	150
B/N Surrogate Standard Mix (3/90)	Restek (31072)	5000	9.0		100

Solvent: Acetone

MASOGD Matrix Spike

Stock Standard	Vendor (Catalog No.)	Stock Standard Concentration (ug/L)	Volume Added (uL)	Final Volume (mL)	Final Concentration (mg/L)
Custom 2-Chlorobenzoic Acid	Restek (562264)	5000	900	30	150
Custom MMR 1	Restek (562265)	1000	900		30
Custom MMR 2	Restek (562266)	1000	900		

Solvent: Acetone

Appendix C: Equations

Response Factor (RFx)

$$\text{Response Factor } (RF_x) = \frac{\text{Area}_x \times \text{Concentration}_{is}}{\text{Area}_{is} \times \text{Concentration}_x}$$

Where:
x = compound,
is = Internal Standard

Relative Retention Time (RRT)

$$\text{Relative Retention Time (RRT)} = \frac{\text{Retention Time}_x}{\text{Retention Time}_{is}}$$

Where:
x = compound
is = Internal Standard

Mean Response Factor ($\overline{RF}$)

$$\text{Mean Response Factor } (\overline{RF}) = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where:
n = number of calibration levels

Standard Deviation of the Response Factor (SD)

$$\text{Standard Deviation of the Response Factor (SD)} = \sqrt{\frac{\sum_{i=1}^{n} \left(RF_i - \overline{RF}\right)2}{n-1}}$$

Where:
n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Response

$$\text{Percent Relative Standard Deviation (RSD) of the Response} = \frac{SD}{\overline{RF}} \times 100\%$$

Percent Difference (%D)

$$\text{Percent Difference (\%D)} = \frac{RF_v - \overline{RF}}{\overline{RF}} \times 100\%$$

Where:
RF_v = Response Factor from the Continuing Calibration Verification (CCV)

Percent Drift

$$\text{Percent Drift} = \frac{\text{Calculated Concentration - Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100\%$$

Percent Recovery (%R)

$$\text{Percent Recovery (\%R)} = \frac{C_s}{C_n} \times 100\%$$

Where:
C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD

$$\text{Percent Recovery (\%R) for MS/MSD} = \frac{C_s - C_u}{C_n} \times 100\%$$

Where:
C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (%RPD)

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

Where:
C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration (for average RF quantification)

Aqueous Samples

$$C_x = \frac{A_x \times C_{is} \times V_t}{A_{IS} \times \text{MeanRF} \times V_o \times V_i} \times DF$$

Where:
C_x = Concentration of compound (ug/L)
A_x = Area of quantitation ion
C_{IS} = Concentration of associated internal standard (ng)
V_t = Extract Volume (uL)
A_{IS} = Area of quantitation ion for associated internal standard.
Mean RF = Mean Response Factor from initial calibration, or 1 for a TIC or Alkane

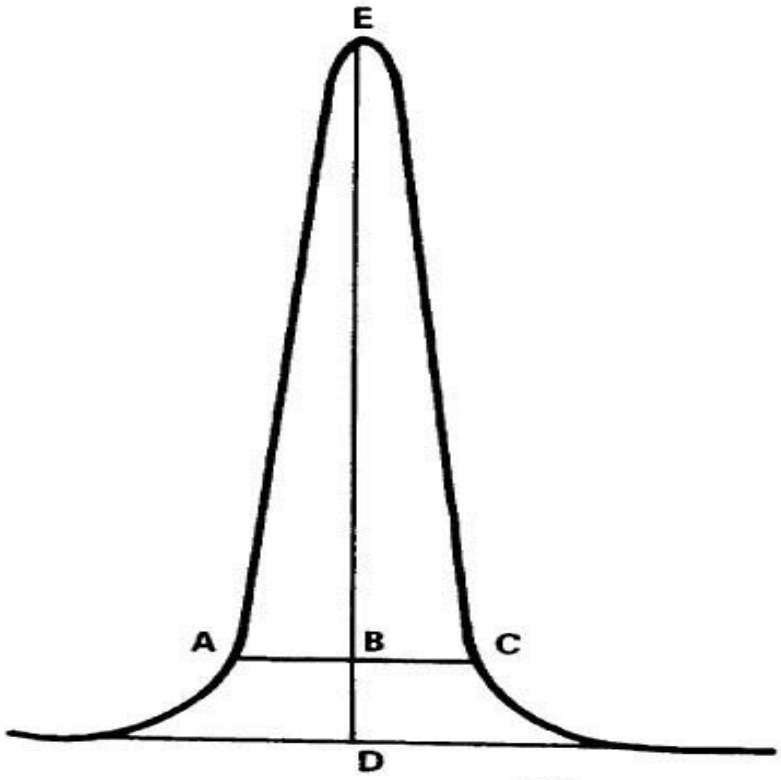
V_o = Sample volume (mL)
V_i = Volume injected (uL)
DF = Dilution Factor

Solid Samples

$$C_x = \frac{A_x \times C_{IS} \times V_t \times GPC \times 10^3 g/Kg}{A_{IS} \times MeanRF \times W_s \times S \times V_i \times 10^3 ng/ug} \times DF$$

Where:
C_x = Concentration of compound (ug/L)
A_x = Area of quantitation ion
C_{IS} = Concentration of associated internal standard (ng)
V_t = Extract Volume (uL)
AIS = Area of quantitation ion for associated internal standard.
Mean RF = Mean Response Factor from initial calibration, or 1 for a TIC or Alkane
Ws = Sample weight (g)
S = Percent Solid
Vi = Volume injected (uL)
DF = Dilution Factor

TAILING FACTOR CALCULATION



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor $= \frac{12}{11} = 1.1$

ATTACHMENT 1: SW-846 8270D Method Information

The tables associated to Attachment 1 summarize the laboratory's established RL for each matrix along with the in-house control limits for accuracy and precision, internal standard associations, characteristic ions used for quantitation and minimum response factor criteria for each analyte.

The RL's provided in these tables are those that can be achieved in a blank water or soil matrix using the routine extraction volume / mass of sample specified in the laboratory SOP for these matrices. These RLs are always adjusted for field samples based on actual sample amount used, final extract volume, percent moisture (solids) and dilutions.

The information provided in these tables is entered in the laboratory's management information system (LIMS) called TALS and is kept current by the local TALS system administrator. The values in TALS are the values used by the laboratory's data processing systems to evaluate and report data. The information presented in these tables is pulled from TALS and current as of the effective date of this SOP but is subject to change. Updates to these tables are made with each SOP revision. For the most current information, refer to the method, equipment and ICAL limit groups maintained in the TALS database.

Attachment 1: SW846 8270D Method Information (BR-MS-001)

Type	Analytes	CAS #	RL		Control Limits (%R)				Precision
			Water	Soil	Water		Soil		RPD
			(ug/L)	ug/Kg)	Lower	Upper	Lower	Upper	≤
Analyte	1,1'-Biphenyl	92-52-4	10	330	60	140	60	140	30
Analyte	1,2,4,5-Tetrachlorobenzene	95-94-3	10	330	60	140	60	140	30
Analyte	1,2,4-Trichlorobenzene	120-82-1	10	330	60	130	55	120	30
Analyte	1,2-Dichlorobenzene	95-50-1	10	330	55	130	55	120	30
Analyte	1,3,5-Trinitrobenzene	99-35-4	10	330	60	140	60	140	30
Analyte	1,3-Dichlorobenzene	541-73-1	10	330	50	125	50	115	30
Analyte	1,3-Diethyl-1,3-Diphenyl Urea	85-98-3	10	330	55	130	50	120	30
Analyte	1,3-Dinitrobenzene	99-65-0	10	330	60	140	60	140	30
Analyte	1,4-Dichlorobenzene	106-46-7	10	330	55	125	55	120	30
Analyte	1,4-Naphthoquinone	130-15-4	10	330	60	140	60	140	30
Analyte	1-Methylnaphthalene	90-12-0	10	330	60	140	60	140	30
Analyte	1-Naphthylamine	134-32-7	10	330	60	140	60	140	30
Analyte	2,2'-oxybis[1-chloropropane]	108-60-1	10	330	60	150	60	130	30
Analyte	2,3,4,6-Tetrachlorophenol	58-90-2	10	330	60	140	60	140	30
Analyte	2,4,5-Trichlorophenol	95-95-4	25	830	60	140	60	130	30
Analyte	2,4,6-Trichlorophenol	88-06-2	10	330	55	155	60	135	30
Analyte	2,4-Dichlorophenol	120-83-2	10	330	55	150	55	130	30
Analyte	2,4-Dimethylphenol	105-67-9	10	330	30	165	25	150	30
Analyte	2,4-Dinitrophenol	51-28-5	25	830	35	150	25	160	30
Analyte	2,4-Dinitrotoluene	121-14-2	10	330	60	130	50	120	30
Analyte	2,6-Dichlorophenol	87-65-0	10	330	60	140	60	140	30
Analyte	2,6-Dinitrotoluene	606-20-2	10	330	70	135	55	130	30
Analyte	2-Acetylaminofluorene	53-96-3	10	330	60	140	60	140	30
Analyte	2-Chlorobenzaldehyde	89-98-5	10	330	30	120	10	105	30
Analyte	2-Chlorobenzoic Acid	118-91-2	380	1660	10	35	10	285	30
Analyte	2-Chloronaphthalene	91-58-7	10	330	50	125	45	120	30
Analyte	2-Chlorophenol	95-57-8	10	330	65	140	60	125	30
Analyte	2-Methyl-3-Nitroaniline	603-83-8	10	330	10	95	10	75	30

Attachment 1: SW846 8270D Method Information (BR-MS-001)

Type	Analytes	CAS #	RL		Control Limits (%R)				Precision
			Water	Soil	Water		Soil		RPD
			(ug/L)	ug/Kg)	Lower	Upper	Lower	Upper	≤
Analyte	2-Methylnaphthalene	91-57-6	10	330	50	145	45	150	30
Analyte	2-Methylphenol	95-48-7	10	330	55	130	50	135	30
Analyte	2-Naphthylamine	91-59-8	10	330	60	140	60	140	30
Analyte	2-Nitroaniline	88-74-4	25	830	65	140	50	125	30
Analyte	2-Nitrodiphenylamine	119-75-5	10	330	45	125	50	120	30
Analyte	2-Nitrophenol	88-75-5	10	330	70	145	55	135	30
Analyte	2-Picoline	109-06-8	10	330	60	140	60	140	30
Analyte	2-Toluidine	95-53-4	10	330	60	140	60	140	30
Analyte	3 & 4 Methylphenol	15831-10-4	10	670	35	130	35	140	30
Analyte	3,3'-Dichlorobenzidine	91-94-1	10	330	10	140	10	120	30
Analyte	3,3'-Dimethylbenzidine	119-93-7	10	330	60	140	60	140	30
Analyte	3,5-Dinitroaniline	618-87-1	10	330	30	140	40	105	30
Analyte	3-Chlorobenzaldehyde	587-04-2	10	330	40	115	5	100	30
Analyte	3-Methylcholanthrene	56-49-5	10	330	60	140	60	140	30
Analyte	3-Nitroaniline	99-09-2	25	830	30	95	20	85	30
Analyte	4,6-Dinitro-2-methylphenol	534-52-1	40	330	55	180	55	160	30
Analyte	4-Aminobiphenyl	92-67-1	10	330	60	140	60	140	30
Analyte	4-Bromophenyl phenyl ether	101-55-3	10	330	55	150	55	130	30
Analyte	4-Chloro-3-methylphenol	59-50-7	10	330	50	155	60	140	30
Analyte	4-Chloroaniline	106-47-8	10	330	10	95	10	90	30
Analyte	4-Chlorobenzaldehyde	104-88-1	10	330	40	110	5	100	30
Analyte	4-Chlorophenyl phenyl ether	7005-72-3	10	330	60	140	50	120	30
Analyte	4-Nitroaniline	100-01-6	25	830	50	135	25	115	30
Analyte	4-Nitrophenol	100-02-7	25	830	10	110	30	155	30
Analyte	4-Nitroquinoline-1-oxide	56-57-5	10	330	60	140	60	140	30
Analyte	7,12-Dimethylbenz(a)anthracene	57-97-6	10	330	60	140	60	140	30
Analyte	Acenaphthene	83-32-9	10	330	60	135	55	120	30
Analyte	Acenaphthylene	208-96-8	10	330	60	125	51	115	30

Attachment 1: SW846 8270D Method Information (BR-MS-001)

Type	Analytes	CAS #	RL		Control Limits (%R)				Precision
			Water	Soil	Water		Soil		RPD
			(ug/L)	ug/Kg)	Lower	Upper	Lower	Upper	≤
Analyte	Acetophenone	98-86-2	10	330	60	140	60	140	30
Analyte	alpha,alpha-Dimethyl phenethylamine	122-09-8	10	330	60	140	60	140	30
Analyte	Aniline	62-53-3	25	830	10	110	10	105	30
Analyte	Anthracene	120-12-7	10	330	70	135	60	125	30
Analyte	Aramite, Total	140-57-8	10	330	60	140	60	140	30
Analyte	Atrazine	1912-24-9	10	330	60	140	60	140	30
Analyte	Azobenzene	103-33-3	10	330	65	135	60	140	30
Analyte	Benzaldehyde	100-52-7	25	330	60	140	60	140	30
Analyte	Benzidine	92-87-5	25	830	10	235	10	120	30
Analyte	Benzo[a]anthracene	56-55-3	10	330	70	135	55	130	30
Analyte	Benzo[a]pyrene	50-32-8	10	330	65	130	55	120	30
Analyte	Benzo[b]fluoranthene	205-99-2	10	330	40	150	45	130	30
Analyte	Benzo[e]pyrene	192-97-2	10	330	60	140	60	140	30
Analyte	Benzo[g,h,i]perylene	191-24-2	10	330	50	140	45	135	30
Analyte	Benzo[k]fluoranthene	207-08-9	10	330	60	140	60	125	30
Analyte	Benzoic acid	65-85-0	110	830	10	70	25	145	30
Analyte	Benzyl alcohol	100-51-6	10	330	45	150	50	155	30
Analyte	Bis(2-chloroethoxy)methane	111-91-1	10	330	45	160	55	125	30
Analyte	Bis(2-chloroethyl)ether	111-44-4	10	330	70	140	60	125	30
Analyte	Bis(2-ethylhexyl) phthalate	117-81-7	10	330	80	145	55	140	30
Analyte	Butyl benzyl phthalate	85-68-7	10	330	75	140	65	145	30
Analyte	Caprolactam	105-60-2	10	330	60	140	60	140	30
Analyte	Carbazole	86-74-8	10	330	60	140	55	125	30
Analyte	Chlorobenzilate	510-15-6	10	330	60	140	60	140	30
Analyte	Chrysene	218-01-9	10	330	65	130	60	125	30
Analyte	Diallate	2303-16-4	10	330	60	140	60	140	30
Analyte	Dibenz(a,h)anthracene	53-70-3	10	330	45	150	35	145	30
Analyte	Dibenzofuran	132-64-9	10	330	65	140	55	120	30

Attachment 1: SW846 8270D Method Information (BR-MS-001)

Type	Analytes	CAS #	RL		Control Limits (%R)				Precision
			Water	Soil	Water		Soil		RPD
			(ug/L)	ug/Kg)	Lower	Upper	Lower	Upper	≤
Analyte	Diethyl phthalate	84-66-2	10	330	55	140	50	125	30
Analyte	Dimethoate	60-51-5	10	330	60	140	60	140	30
Analyte	Dimethyl phthalate	131-11-3	10	330	65	140	55	120	30
Analyte	Di-n-butyl phthalate	84-74-2	10	330	60	135	50	120	30
Analyte	Di-n-octyl phthalate	117-84-0	10	330	70	135	60	135	30
Analyte	Dinoseb	88-85-7	40	330	60	140	60	140	30
Analyte	Di-n-propyl Adipate	106-19-4	10	330	50	130	50	115	30
Analyte	Disulfoton	298-04-4	10	330	60	140	60	140	30
Analyte	Ethyl methacrylate	97-63-2	10	330	60	140	60	140	30
Analyte	Ethyl methanesulfonate	62-50-0	10	330	60	140	60	140	30
Analyte	Ethyl Parathion	56-38-2	10	330	60	140	60	140	30
Analyte	Famphur	52-85-7	10	330	60	140	60	140	30
Analyte	Fluoranthene	206-44-0	10	330	50	140	50	120	30
Analyte	Fluorene	86-73-7	10	330	65	135	50	125	30
Analyte	Hexachlorobenzene	118-74-1	10	330	55	150	55	125	30
Analyte	Hexachlorobutadiene	87-68-3	10	330	35	140	50	130	30
Analyte	Hexachlorocyclopentadiene	77-47-4	10	330	10	155	30	105	30
Analyte	Hexachloroethane	67-72-1	10	330	50	140	55	120	30
Analyte	Hexachloropropene	1888-71-7	10	330	60	140	60	140	30
Analyte	Indeno[1,2,3-cd]pyrene	193-39-5	10	330	45	150	55	135	30
Analyte	Isodrin	465-73-6	10	330	60	140	60	140	30
Analyte	Isophorone	78-59-1	10	330	45	135	50	115	30
Analyte	Isosafrole	120-58-1	10	330	60	140	60	140	30
Analyte	Kepone	143-50-0	40	830	60	140	60	140	30
Analyte	Methapyrilene	91-80-5	10	830	60	140	60	140	30
Analyte	Methyl methanesulfonate	66-27-3	10	330	60	140	60	140	30
Analyte	Methyl parathion	298-00-0	10	330	60	140	60	140	30
Analyte	Naphthalene	91-20-3	10	330	65	135	55	120	30

Attachment 1: SW846 8270D Method Information (BR-MS-001)

Type	Analytes	CAS #	RL		Control Limits (%R)				Precision
			Water	Soil	Water		Soil		RPD
			(ug/L)	ug/Kg)	Lower	Upper	Lower	Upper	≤
Analyte	Nitrobenzene	98-95-3	10	330	60	135	55	120	30
Analyte	N-Nitro-o-toluidine	99-55-8	10	330	35	120	20	85	30
Analyte	N-Nitrosodiethylamine	55-18-5	10	330	60	140	60	140	30
Analyte	N-Nitrosodimethylamine	62-75-9	10	330	45	110	55	125	30
Analyte	N-Nitrosodi-n-butylamine	924-16-3	10	330	60	140	60	140	30
Analyte	N-Nitrosodi-n-propylamine	621-64-7	10	330	60	130	45	125	30
Analyte	N-Nitrosodiphenylamine	86-30-6	10	330	65	125	45	130	30
Analyte	N-Nitrosomethylethylamine	10595-95-6	10	330	60	140	60	140	30
Analyte	N-Nitrosomorpholine	59-89-2	10	330	60	140	60	140	30
Analyte	N-Nitrosopiperidine	100-75-4	10	330	60	140	60	140	30
Analyte	N-Nitrosopyrrolidine	930-55-2	10	330	60	140	60	140	30
Analyte	o,o',o''-Triethylphosphorothioate	126-68-1	10	330	60	140	60	140	30
Analyte	p-Dimethylamino azobenzene	60-11-7	10	330	60	140	60	140	30
Analyte	Pentachlorobenzene	608-93-5	10	330	60	140	60	140	30
Analyte	Pentachloroethane	76-01-7	10	330	60	140	60	140	30
Analyte	Pentachloronitrobenzene	82-68-8	10	330	60	140	60	140	30
Analyte	Pentachlorophenol	87-86-5	25	830	50	165	50	140	30
Analyte	Perylene	198-55-0	10	330	60	140	60	140	30
Analyte	Phenacetin	62-44-2	10	330	60	140	60	140	30
Analyte	Phenanthrene	85-01-8	10	330	70	135	60	125	30
Analyte	Phenol	108-95-2	10	330	25	90	60	140	30
Analyte	Phorate	298-02-2	10	330	60	140	60	140	30
Analyte	p-Phenylene diamine	106-50-3	25	330	60	140	60	140	30
Analyte	Pronamide	23950-58-5	10	330	60	140	60	140	30
Analyte	Pyrene	129-00-0	10	330	70	160	35	175	30
Analyte	Pyridine	110-86-1	10	330	10	105	15	105	30
Analyte	Safrole, Total	94-59-7	10	330	60	140	60	140	30
Analyte	Sulfotepp	3689-24-5	10	330	60	140	60	140	30

Attachment 1: SW846 8270D Method Information (BR-MS-001)

Type	Analytes	CAS #	RL		Control Limits (%R)				Precision
			Water	Soil	Water		Soil		RPD
			(ug/L)	ug/Kg)	Lower	Upper	Lower	Upper	≤
Analyte	Thionazin	297-97-2	10	330	60	140	60	140	30
Surrogate	2,4,6-Tribromophenol	118-79-6	NA	NA	50	135	50	120	NA
Surrogate	2-Fluorobiphenyl	321-60-8	NA	NA	30	95	45	110	NA
Surrogate	2-Fluorophenol	367-12-4	NA	NA	90	95	45	110	NA
Surrogate	Nitrobenzene-d5	4165-60-0	NA	NA	55	125	50	115	NA
Surrogate	Phenol-d5	4165-62-2	NA	NA	10	75	50	120	NA
Surrogate	Terphenyl-d14	1718-51-0	NA	NA	55	145	40	145	NA
Surrogate	2-Chlorophenol-d4	93951-73-6	NA	NA	55	120	50	115	NA
Surrogate	1,2-Dichlorobenzene-d4	2199-69-1	NA	NA	55	120	50	115	NA
ISTD 1	1,4-Dichlorobenzene-d4	3855-82-1	NA	NA	NA	NA	NA	NA	NA
ISTD 2	Naphthalene-d8	1146-65-2	NA	NA	NA	NA	NA	NA	NA
ISTD 3	Acenaphthene-d10	15067-26-2	NA	NA	NA	NA	NA	NA	NA
ISTD 4	Phenanthrene-d10	1517-22-2	NA	NA	NA	NA	NA	NA	NA
ISTD 5	Chrysene-d12	1719-03-5	NA	NA	NA	NA	NA	NA	NA
ISTD 6	Perylene-d12	1520-96-3	NA	NA	NA	NA	NA	NA	NA

Attachment 1: SW846 8270D Method Information (BR-MS-001)

Type	Analytes	CAS #	ICAL Group	Internal Standard Association	Characterisitc Ion			Minimum Response Factor
					Primary	Secondary	Tertiary	
Analyte	1,1'-Biphenyl	92-52-4	A	3	154	153	152	0.010
Analyte	1,2,4,5-Tetrachlorobenzene	95-94-3	A	3	216	214	218	0.010
Analyte	1,2,4-Trichlorobenzene	120-82-1	A	2	180	182	145	
Analyte	1,2-Dichlorobenzene	95-50-1	A	1	146	148	111	
Analyte	1,3,5-Trinitrobenzene	99-35-4	A	4	213	74	91	
Analyte	1,3-Dichlorobenzene	541-73-1	A	1	146	148	111	
Analyte	1,3-Diethyl-1,3-Diphenyl Urea	85-98-3	A	4	148	120	77	
Analyte	1,3-Dinitrobenzene	99-65-0	A	3	168	50	76	
Analyte	1,4-Dichlorobenzene	106-46-7	A	1	146	148	111	
Analyte	1,4-Naphthoquinone	130-15-4	A	3	158	102	76	
Analyte	1-Methylnaphthalene	90-12-0	A	2	142	141	115	
Analyte	1-Naphthylamine	134-32-7	A	3	143	116	115	
Analyte	2,2'-oxybis[1-chloropropane]	108-60-1	A	1	45	121	NA	0.010
Analyte	2,3,4,6-Tetrachlorophenol	58-90-2	A	3	232	231	131	0.010
Analyte	2,4,5-Trichlorophenol	95-95-4	B	3	196	198	97	0.200
Analyte	2,4,6-Trichlorophenol	88-06-2	A	3	196	198	220	0.200
Analyte	2,4-Dichlorophenol	120-83-2	A	2	162	164	98	0.200
Analyte	2,4-Dimethylphenol	105-67-9	A	2	122	107	121	0.200
Analyte	2,4-Dinitrophenol	51-28-5	B	3	187	107	NA	0.010
Analyte	2,4-Dinitrotoluene	121-14-2	A	3	165	89	NA	0.200
Analyte	2,6-Dichlorophenol	87-65-0	A	2	162	164	98	
Analyte	2,6-Dinitrotoluene	606-20-2	A	3	165	89	NA	0.200
Analyte	2-Acetylaminofluorene	53-96-3	A	5	181	223	152	
Analyte	2-Chlorobenzaldehyde	89-98-5	A	2	139	140	141	
Analyte	2-Chlorobenzoic Acid	118-91-2	C	3	141	156	111	
Analyte	2-Chloronaphthalene	91-58-7	A	3	162	127	164	0.800
Analyte	2-Chlorophenol	95-57-8	A	1	128	64	130	0.800
Analyte	2-Methyl-3-Nitroaniline	603-83-8	A	3	135	104	NA	

Attachment 1: SW846 8270D Method Information (BR-MS-001)

Type	Analytes	CAS #	ICAL Group	Internal Standard Association	Characterisitc Ion			Minimum Response Factor
					Primary	Secondary	Tertiary	
Analyte	2-Methylnaphthalene	91-57-6	A	2	115	141	142	0.400
Analyte	2-Methylphenol	95-48-7	A	1	107	108	79	0.800
Analyte	2-Naphthylamine	91-59-8	A	3	143	116	115	
Analyte	2-Nitroaniline	88-74-4	B	3	65	92	138	0.010
Analyte	2-Nitrodiphenylamine	119-75-5	A	4	214	167	180	
Analyte	2-Nitrophenol	88-75-5	A	2	139	109	65	0.100
Analyte	2-Picoline	109-06-8	A	1	93	66	NA	
Analyte	2-Toluidine	95-53-4	A	1	106	107	NA	
Analyte	3 & 4 Methylphenol	15831-10-4	A	1	107	108	79	0.600
Analyte	3,3'-Dichlorobenzidine	91-94-1	A	5	252	254	126	0.010
Analyte	3,3'-Dimethylbenzidine	119-93-7	A	5	212	213	106	
Analyte	3,5-Dinitroaniline	618-87-1	A	4	183	64	67	
Analyte	3-Chlorobenzaldehyde	587-04-2	A	2	139	140	141	
Analyte	3-Methylcholanthrene	56-49-5	A	6	268	253	126	
Analyte	3-Nitroaniline	99-09-2	B	3	138	65	92	0.010
Analyte	4,6-Dinitro-2-methylphenol	534-52-1	B	4	198	121	106	0.010
Analyte	4-Aminobiphenyl	92-67-1	A	4	169	168	115	
Analyte	4-Bromophenyl phenyl ether	101-55-3	A	4	248	250	141	0.100
Analyte	4-Chloro-3-methylphenol	59-50-7	A	2	107	44	NA	0.200
Analyte	4-Chloroaniline	106-47-8	A	2	127	129	NA	0.010
Analyte	4-Chlorobenzaldehyde	104-88-1	A	2	139	140	141	
Analyte	4-Chlorophenyl phenyl ether	7005-72-3	A	3	204	206	141	0.400
Analyte	4-Nitroaniline	100-01-6	B	3	139	92	108	0.010
Analyte	4-Nitrophenol	100-02-7	B	3	109	81	65	0.010
Analyte	4-Nitroquinoline-1-oxide	56-57-5	A	4	101	128	174	
Analyte	7,12-Dimethylbenz(a)anthracene	57-97-6	A	6	256	241	128	
Analyte	Acenaphthene	83-32-9	A	3	154	153	152	0.900
Analyte	Acenaphthylene	208-96-8	A	3	152	153	NA	0.900

Attachment 1: SW846 8270D Method Information (BR-MS-001)

Type	Analytes	CAS #	ICAL Group	Internal Standard Association	Characterisitc Ion			Minimum Response Factor
					Primary	Secondary	Tertiary	
Analyte	Acetophenone	98-86-2	A	1	105	77	51	0.010
Analyte	alpha,alpha-Dimethyl phenethylamine	122-09-8	A	2	58	91	NA	
Analyte	Aniline	62-53-3	B	1	93	66	65	
Analyte	Anthracene	120-12-7	A	4	178	176	179	0.700
Analyte	Aramite, Total	140-57-8	A	5	185	191	319	
Analyte	Atrazine	1912-24-9	A	4	200	173	215	0.010
Analyte	Azobenzene	103-33-3	A	4	182	77	NA	
Analyte	Benzaldehyde	100-52-7	A	1	77	105	106	0.010
Analyte	Benzidine	92-87-5	B	5	184	92	185	
Analyte	Benzo[a]anthracene	56-55-3	A	5	228	229	226	0.800
Analyte	Benzo[a]pyrene	50-32-8	A	6	252	253	125	0.700
Analyte	Benzo[b]fluoranthene	205-99-2	A	6	252	253	125	0.700
Analyte	Benzo[e]pyrene	192-97-2	A	6	252	253	125	
Analyte	Benzo[g,h,i]perylene	191-24-2	A	6	276	138	277	0.500
Analyte	Benzo[k]fluoranthene	207-08-9	A	6	252	253	125	0.700
Analyte	Benzoic acid	65-85-0	A	2	93	95	123	
Analyte	Benzyl alcohol	100-51-6	A	1	108	79	77	0.300
Analyte	Bis(2-chloroethoxy)methane	111-91-1	C	2	93	95	123	
Analyte	Bis(2-chloroethyl)ether	111-44-4	A	1	93	95	NA	0.700
Analyte	Bis(2-ethylhexyl) phthalate	117-81-7	A	5	149	167	NA	0.010
Analyte	Butyl benzyl phthalate	85-68-7	A	5	149	91	206	0.010
Analyte	Caprolactam	105-60-2	A	2	113	55	56	0.010
Analyte	Carbazole	86-74-8	A	4	167	139	NA	0.010
Analyte	Chlorobenzilate	510-15-6	A	5	251	139	111	
Analyte	Chrysene	218-01-9	A	5	228	226	229	0.700
Analyte	Diallate	2303-16-4	A	4	86	234	43	
Analyte	Dibenz(a,h)anthracene	53-70-3	A	6	278	139	279	0.400
Analyte	Dibenzofuran	132-64-9	A	3	168	139	NA	0.800

Attachment 1: SW846 8270D Method Information (BR-MS-001)

Type	Analytes	CAS #	ICAL Group	Internal Standard Association	Characterisitc Ion			Minimum Response Factor
					Primary	Secondary	Tertiary	
Analyte	Diethyl phthalate	84-66-2	A	3	149	177	150	0.010
Analyte	Dimethoate	60-51-5	A	4	87	125	93	
Analyte	Dimethyl phthalate	131-11-3	A	3	330	163	194	0.010
Analyte	Di-n-butyl phthalate	84-74-2	A	4	149	150	104	0.010
Analyte	Di-n-octyl phthalate	117-84-0	A	6	149	NA	NA	0.010
Analyte	Dinoseb	88-85-7	A	4	211	163	147	
Analyte	Di-n-propyl Adipate	106-19-4	A	3	171	129	111	
Analyte	Disulfoton	298-04-4	A	4	97	142	NA	
Analyte	Ethyl methacrylate	97-63-2	A	1	69	99	41	
Analyte	Ethyl methanesulfonate	62-50-0	A	1	79	109	97	
Analyte	Ethyl Parathion	56-38-2	A	4	109	97	291	
Analyte	Famphur	52-85-7	A	5	218	125	93	
Analyte	Fluoranthene	206-44-0	A	4	202	203	101	0.600
Analyte	Fluorene	86-73-7	A	3	166	165	NA	0.900
Analyte	Hexachlorobenzene	118-74-1	A	4	284	142	249	0.100
Analyte	Hexachlorobutadiene	87-68-3	A	2	225	223	227	0.010
Analyte	Hexachlorocyclopentadiene	77-47-4	A	3	237	235	272	0.050
Analyte	Hexachloroethane	67-72-1	A	1	117	201	199	0.300
Analyte	Hexachloropropene	1888-71-7	A	2	213	211	106	
Analyte	Indeno[1,2,3-cd]pyrene	193-39-5	A	6	276	138	277	0.500
Analyte	Isodrin	465-73-6	A	4	193	147	66	
Analyte	Isophorone	78-59-1	A	2	82	95	138	0.400
Analyte	Isosafrole	120-58-1	A	2	162	104	131	
Analyte	Kepone	143-50-0	A	5	272	237	143	
Analyte	Methapyrilene	91-80-5	A	4	58	97	71	
Analyte	Methyl methanesulfonate	66-27-3	A	1	80	79	65	
Analyte	Methyl parathion	298-00-0	A	4	109	125	263	
Analyte	Naphthalene	91-20-3	A	2	128	129	NA	0.700

Attachment 1: SW846 8270D Method Information (BR-MS-001)

Type	Analytes	CAS #	ICAL Group	Internal Standard Association	Characterisitc Ion			Minimum Response Factor
					Primary	Secondary	Tertiary	
Analyte	Nitrobenzene	98-95-3	A	2	77	123	65	0.200
Analyte	N-Nitro-o-toluidine	99-55-8	A	3	152	106	79	
Analyte	N-Nitrosodiethylamine	55-18-5	A	1	102	57	44	
Analyte	N-Nitrosodimethylamine	62-75-9	A	1	74	42	NA	
Analyte	N-Nitrosodi-n-butylamine	924-16-3	A	2	84	116	158	
Analyte	N-Nitrosodi-n-propylamine	621-64-7	A	1	70	42	130	0.500
Analyte	N-Nitrosodiphenylamine	86-30-6	A	4	169	168	167	0.010
Analyte	N-Nitrosomethylethylamine	10595-95-6	A	1	88	42	43	
Analyte	N-Nitrosomorpholine	59-89-2	A	1	56	86	116	
Analyte	N-Nitrosopiperidine	100-75-4	A	2	114	42	55	
Analyte	N-Nitrosopyrrolidine	930-55-2	A	1	100	68	42	
Analyte	o,o',o''-Triethylphosphorothioate	126-68-1	A	2	198	121	97	
Analyte	p-Dimethylamino azobenzene	60-11-7	A	5	120	225	77	
Analyte	Pentachlorobenzene	608-93-5	A	3	250	248	215	
Analyte	Pentachloroethane	76-01-7	A	1	117	167	83	
Analyte	Pentachloronitrobenzene	82-68-8	A	4	237	214	142	
Analyte	Pentachlorophenol	87-86-5	B	4	266	268	204	0.050
Analyte	Perylene	198-55-0	A	6	252	253	125	
Analyte	Phenacetin	62-44-2	A	4	108	179	137	
Analyte	Phenanthrene	85-01-8	A	4	178	179	176	0.700
Analyte	Phenol	108-95-2	A	1	94	65	66	0.800
Analyte	Phorate	298-02-2	A	4	121	260	NA	
Analyte	p-Phenylene diamine	106-50-3	A	2	108	80	53	
Analyte	Pronamide	23950-58-5	A	4	173	175	145	
Analyte	Pyrene	129-00-0	A	5	202	203	101	0.600
Analyte	Pyridine	110-86-1	A	1	79	52	NA	
Analyte	Safrole, Total	94-59-7	A	3	104	103	131	
Analyte	Sulfotepp	3689-24-5	A	4	322	202	266	

Attachment 1: SW846 8270D Method Information (BR-MS-001)

Type	Analytes	CAS #	ICAL Group	Internal Standard Association	Characterisitc Ion			Minimum Response Factor
					Primary	Secondary	Tertiary	
Analyte	Thionazin	297-97-2	A	3	97	107	143	
Surrogate	2,4,6-Tribromophenol	118-79-6	NA	4	330	332	141	
Surrogate	2-Fluorobiphenyl	321-60-8	NA	3	172	171	NA	
Surrogate	2-Fluorophenol	367-12-4	NA	1	112	64	NA	
Surrogate	Nitrobenzene-d5	4165-60-0	NA	2	82	54	128	
Surrogate	Phenol-d5	4165-62-2	NA	1	99	71	42	
Surrogate	Terphenyl-d14	1718-51-0	NA	5	244	122	212	
Surrogate	2-Chlorophenol-d4	93951-73-6	NA	1	132	68	134	
Surrogate	1,2-Dichlorobenzene-d4	2199-69-1	NA	1	152	150	115	
ISTD 1	1,4-Dichlorobenzene-d4	3855-82-1	NA	NA	152	115	150	
ISTD 2	Naphthalene-d8	1146-65-2	NA	NA	136	68	NA	
ISTD 3	Acenaphthene-d10	15067-26-2	NA	NA	164	162	160	
ISTD 4	Phenanthrene-d10	1517-22-2	NA	NA	188	94	80	
ISTD 5	Chrysene-d12	1719-03-5	NA	NA	240	120	236	
ISTD 6	Perylene-d12	1520-96-3	NA	NA	264	260	265	

Chain of Custody Record

Connecticut
128 Long Hill Cross Road
Shelton, CT 06484
Tel: 203-929-8140
Fax: 203-929-8142

TAL-0015 (0508)

151209

Client	Project Manager	Date	Chain of Custody Number
GEI Consultants Inc.	Melissa Felter / Kim Bradley		18896

Address	Telephone Number (Area Code)/Fax Number/e-mail address	Field Telephone Number	Page / of
455 Winding Brook Dr.	860-368-5300 / mfelter@geiconsultants.com / kbradley@geiconsultants.com	860-917-0670	Page 1 of

City	State	Zip Code	Site Contact	Lab Contact
Glastonbury	CT	06033	Kim Bradley / Kari Weber	Pat McIssac

Project Name and Location (State)	Sample Disposal	Analysis (Attach list if more space is needed)
Gowanus Canal Brooklyn, NY	☐ Return To Client ☐ Disposal By Lab ☐ Archive For ____ Months (A fee may be assessed if samples are retained longer than 1 month)	

Contract/Purchase Order/Project No.
093010-2-1202

Field Sample I.D. (Containers for each sample may be combined on one line)	Collection Date	Collection Time	Matrix: Aqueous	Solid	Other	Containers & Preservatives: Unpres.	H_2SO_4	HNO_3	HCl	NaOH	ZnAc/NaOH	Other	SEE ATTACHED SLW Analytical	SEE ATTACHED SEDIMENT ANALYTICAL	SEE ATTACHED Pace Analytical	Comments
													X			
														X		
															X	

Turn Around Time Required (business days) Report / EDD Requirements

☐ 24 Hours ☐ 48 Hours ☐ 5 Days ☐ 10 Days ☐ 15 Days ☒ Other SEE Contact

State Regulatory QC Requirements

1. Relinquished By	Date	Time	1. Received By	Date	Time
2. Relinquished By	Date	Time	2. Received By	Date	Time
3. Received By	Date	Time	Cooler Temps	Passed Rad. Screen (Lab Use Only)	☐ Yes ☐ No

Comments

DISTRIBUTION: *WHITE - Stays with the Samples; CANARY - Returned to Client with Report; PINK - Field Copy*

Note: Deliverable Type → NYS ASP Cat B + EPA II EDD

EDD to datagroup@geiconsultants.com
Hard Copy to Lori Macinnon

CMS

Gowanus Canal Sediment Sampling - Chain of Custody TestAmerica Analytical Chemistry List

Analyze **bulk surficial sediment** samples from 38 locations for bulk sediment chemistry and characteristics:

- VOCs according to USEPA Method 8260B
- SVOCs according to USEPA Method 8270C
- PCBs according to USEPA Method 8082A
- PCB congeners according to USEPA Method 1668A Only locations: 301, 305, 307A, 308A, 308B, 312, 320, and 325)
- Pesticides according to USEPA Method 8081A
- Herbicides according to USEPA Method 8151A
- Target Analyte List (TAL) metals according to USEPA Method 6020
- Mercury according to USEPA 7471
- Methyl Mercury according to USEPA 1630
- Total cyanide according to USEPA Method 9012
- Ammonia according to USEPA Method 350.1
- Total organic carbon (TOC) according to USEPA Method Lloyd Kahn
- Nitrate/nitrite according to USEPA Method 300.0
- Sulfate according to USEPA Method 300.0
- Sulfide according to USEPA 9030B/9034
- SEM/AVS TAL metals according to USEPA Method 200 series
- Biological Oxygen Demand (BOD) according to SM 5210B
- Black Carbon
- Bulk Density according to ASTM D2937
- Percent Water Content according to ASTM D2216
- Grain Size according to ASTM D422
- pH according to EPA Method 9045C
- Hardness according to ASTM 2340B
- Salinity according to ASTM 2520B

Analyze **interstitial water samples** from 38 locations for:

- 34 PAHs according to USEPA Method 8272-SPME GC/MS
- TAL metals according to USEPA Method 6020
- Mercury according to USEPA Method 7470
- Methyl Mercury according to USEPA 1630
- Free cyanide according to USEPA Method 9016
- Total cyanide according to USEPA Method 9012
- Ammonia according to USEAP Method 350.1
- TOC according to USEPA Method 9060
- Nitrate/nitrite according to USEPA Method 300.0
- Sulfate according to USEPA Method 300.0
- Dissolved Organic Carbon (DOC) according to SM5310C
- pH according to EPA Method 9045C
- Hardness according to ASTM 2340B
- Salinity according to ASTM 2520B

Analyze depth-integrated **surface water samples** from 38 locations for:

- VOCs according to USEPA Method 8260B
- SVOCs according to USEPA Method 8270C
- PCBs according to USEPA Method 8082A
- Pesticides according to USEPA Method 8081A
- Herbicides according to USEPA Method 8151A
- TAL metals according to USEPA Method 6020
- Mercury according to USEPA Method 7470
- Methyl Mercury according to USEPA 1630
- Free cyanide according to USEPA Method 9016
- Total cyanide according to USEPA Method 9012
- Ammonia according to USEAP Method 350.1
- Nitrate/nitrite according to USEPA Method 300.0
- Sulfate according to USEPA Method 300.0
- TOC according to USEPA Method Lloyd Kahn
- Biological Oxygen Demand (BOD) according to SM 5210B
- Chemical Oxygen Demand (COD) according to USEPA Method 410.4
- Dissolved Organic Carbon (DOC) according to SM5310C
- Total Suspended Solids (TSS) according to ASTM 2540D
- pH according to EPA Method 9045C
- Hardness according to ASTM 2340B
- Salinity according to ASTM 2520B

Attachment D

Example Chain of Custody

Columbia Analytical Services[INC]

CHAIN OF CUSTODY

SR#: ______

1317 South 13th Ave, Kelso, WA 98626 | 360.577.7222 | 800.695.7222 | 360.636.1068 (fax)

PAGE ____ OF ____ COC #

PROJECT NAME: Gowanus Canal Sediment

PROJECT NUMBER: ~~093010-3-B07~~ 2-1202

PROJECT MANAGER: Dave Terry / Melissa Felter

COMPANY/ADDRESS: 455 Winding Brook Dr, Suite 201

CITY/STATE/ZIP: Glastonbury, CT 06033

E-MAIL ADDRESS: mfelter@geiconsultants.com

PHONE #: 860 368 5300 FAX #: 860 368 5307

SAMPLER'S SIGNATURE:

SAMPLE I.D.	DATE	TIME	LAB I.D.	MATRIX	NUMBER OF CONTAINERS	Semivolatile Organics by GC/MS 625☐ 8270☐ 8270LL☐	Volatile Organics 624☐ 8260☐	Hydrocarbons (*see below) Gas☐ 8021☐ BTEX☐	Fuel Fingerprint (FIQ) ☐ Diesel☐ Oil☐	Oil & Grease/TRPH NW-HCID Screen ☐	PCB's 1664 HEM☐ 1664 SGT☐	Aroclors☐ Congeners☐	Pesticides/Herbicides 608☐ 8081A☐ 8141A☐ 8151A☐	Chlorophenolics - 8151M Tri☐ Tetra☐ PCP☐	PAHS 8310☐ SIM☐	Metals, Total or Dissolved (See list below)	Cyanide☐	Hex-Chrom☐	pH, Cond., Cl, SO4, PO4, F, NO_2, NO_3, BOD, TSS, TDS (circle)	NH_3-N, COD, Total-P, TKN, TOC, DOC (circle) NO_2+NO_3	TOX 9020☐ AOX 1650☐ 506☐	Group I & II EDL/PEC P1691	Group 5 Alkylphenols D7065-06			REMARKS
				SED	1-8 oz																	X	X			
				SW	4-1L Amber																	X	X			

REPORT REQUIREMENTS

___ I. Routine Report: Method Blank, Surrogate, as required

___ II. Report Dup., MS, MSD as required

___ III. Data Validation Report (includes all raw data)

X IV. CLP Deliverable Report

X V. EDD EPA Region II

INVOICE INFORMATION

P.O. # ______

Bill To: ______

TURNAROUND REQUIREMENTS

___ 24 hr. ___ 48 hr.

___ 5 Day

X Standard (10-15 working days)

___ Provide FAX Results

______ Requested Report Date

Circle which metals are to be analyzed:

Total Metals: Al As Sb Ba Be B Ca Cd Co Cr Cu Fe Pb Mg Mn Mo Ni K Ag Na Se Sr Tl Sn V Zn Hg

Dissolved Metals: Al As Sb Ba Be B Ca Cd Co Cr Cu Fe Pb Mg Mn Mo Ni K Ag Na Se Sr Tl Sn V Zn Hg

*INDICATE STATE HYDROCARBON PROCEDURE: AK CA WI NORTHWEST OTHER: ____ (CIRCLE ONE)

SPECIAL INSTRUCTIONS/COMMENTS:

Please send EDD to datagroup@geiconsultants.com

Please send hard copy to Lorie MacKinnon, 113 Odell Rd, Sandown, NH 03873

☐ Sample Shipment contains USDA regulated soil samples (check box if applicable)

RELINQUISHED BY:	RECEIVED BY:	RELINQUISHED BY:	RECEIVED BY:
Signature / Date/Time	Signature / Date/Time	Signature / Date/Time	Signature / Date/Time
Printed Name / Firm	Printed Name / Firm	Printed Name / Firm	Printed Name / Firm

Attachment E

Field Change Order Form

Field Modification Form
for
Paerdegat Basin , Sediment, Tissue and Porous Surface Sampling
GEI Consultants, Inc.

Date:

Document:

Activity:

Requested Modification:

Rationale:

Attachments:

Project Manager: