JOHNSON LAKE FISH TISSUE MONITORING STUDY

SAMPLING AND ANALYSIS PLAN

PREPARED FOR:

OWENS-BROCKWAY GLASS CONTAINER INC.
5850 NE 92ND DRIVE
PORTLAND, OR, 97222

PREPARED BY:

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APRIL 28, 2022

Grette Associates LLC
ENVIRONMENTAL CONSULTANTS

TABLE OF CONTENTS

1 INTRODUCTION 1
1.1 Project Site Description and Background 1
1.2 Remediation Activities 1
1.3 Sampling and Analysis Plan (SAP) 2
1.3.1 Purpose 2
1.3.2 Scope 2
1.3.3 Organization 2

2 FISH SAMPLING 5
2.1 Fish Collection 6
2.2 Tissue Sample Collection, Decontamination, and Packaging 8
2.3 Sample Transport and Chain of Custody Procedures 9

3 LABORATORY ANALYSIS 10
3.1 Testing Facilities 10
3.2 Laboratory Chain of Custody 10
3.3 Chemicals of Concern (Chemicals of Interest) Screening 10
3.4 Testing Methods 10

4 QUALITY ASSURANCE 11

5 REFERENCES 12

LIST OF TABLES

Table 1. Sample Holding Time and Storage Criteria.....10

LIST OF FIGURES

Figure 1: Site Map of Johnson Lake.....13

LIST OF ATTACHMENTS

Attachment A. ODFW Scientific Taking Permit
Attachment B. ODEQ May 25, 2017 SAP approval and comments
Attachment C. ALS Standard Operating Procedure MET-TISP (5/29/16) – Fish Tissue Sample Preparation
Attachment D. ODEQ January 29, 2018 Letter and Comments
Attachment E. Johnson Lake Quality Assurance Project Plan

1 INTRODUCTION

This Sampling and Analysis Plan (SAP) has been prepared to describe the Johnson Lake fish tissue monitoring study, consistent with the *Remediation Operations & Maintenance Plan* (O&M Plan)*, Sediment Remedial Action* (DOF 2012b), hereafter "O&M Plan." In support of this SAP, the approved Scientific Take Permit from the Oregon Department of Fish and Wildlife (ODFW) is included as Attachment A. Approval of the 2017 SAP by comment letter from Oregon Department of Environmental Quality (DEQ), comments which are incorporated herein, are included as Attachment B. The analytical laboratories (ALS Global) standard operating procedures for fish tissue sample preparation are attached as Attachment C. DEQ comments from the 2017 collection and sampling effort are included as Attachment D.

The 2022 Johnson Lake fish tissue monitoring study is a requirement of the O&M Plan in response to the results of the 2017 monitoring effort showing contaminant levels in fish samples exceeding the levels specified in the 2007 Record of Decision for the site cleanup.

1.1 PROJECT SITE DESCRIPTION AND BACKGROUND

Owens-Brockway Glass Containers Inc. (Owens) owns and operates a glass manufacturing plant located at 5850 NE 92nd Drive, Portland, Oregon (the "Property"). Owens' Property is approximately 43 acres in size and is located on the south shore of Johnson Lake. Johnson Lake extends over 18 acres and is directly connected to the Whitaker Slough, which in turn flows to the Columbia Slough. Johnson Lake is bounded to the south by the Owens Property and to the west and east by other industrial facilities and highways. The Owens Property and Johnson Lake are collectively referred to as the "Site."

Sediments in Johnson Lake were found to contain polychlorinated biphenyls (PCBs), as described in the *Johnson Lake Investigation Work Plan* (ARCADIS 2004a), *Site Investigation Report* (ARCADIS 2004b), and *Johnson Lake Risk Assessment* (Environ and ARCADIS 2004). Based on the previous investigations and a Feasibility study prepared in 2006, DEQ selected a final remedy for the site in the Record of Decision (October 2007) and Record of Decision Amendment (July 2009). The 2007 Record of Decision and the 2009 Amendment are referred to collectively in this document as the ROD.

1.2 REMEDIATION ACTIVITIES

As described in the ROD, the final remedy for cleanup of the PCBs and other contaminants at the site consisted of the following tasks.

1. Surface soils from the low-lying area were excavated.
2. Excavated soils were disposed of at an off-site landfill.
3. The excavated area was covered with clean soil and revegetated.
4. A stormwater bio-swale (swale) was constructed.
5. A thin layer cap was placed over Johnson Lake sediments.

All of the above-listed tasks have been completed and are addressed in detail in the two Site completion reports:

- *Project Completion Report, Soil Remediation Action*, March 22, 2011 (DOF and Environ 2011) (Tasks 1 through 4)

- *Project Completion Report, Sediment Remedial Action,* September 24, 2012 (DOF 2012a) (Task 5).

1.3 SAMPLING AND ANALYSIS PLAN (SAP)

1.3.1 Purpose

The purpose of this SAP is to describe the Johnson Lake fish tissue monitoring study and build upon previous study efforts. The objective of this study is to evaluate the level of PCBs in fish tissue sampled from Johnson Lake, consistent with the O&M Plan (DOF 2012b). The ROD included the following objective with respect to PCBs in fish tissue, "Prevent human consumption of fish tissue with tissue concentrations of greater than 0.003 ug/kg PCB congener 126". Fish tissue results will be compared with this concentration.

In 2017, a collection and sampling field effort was completed, and all field and lab data were included in a monitoring report submitted to DEQ in January of 2018. As PCBs were still present in fish tissue at levels exceeding the ROD standard, further efforts to collect fish tissue in 2022 will be conducted and will include information provided in the 2018 report as well as a summary of results from the 2017 and 2004 events for comparison (DEQ 2018). This SAP addresses comments from DEQ regarding the 2017 sampling efforts.

1.3.2 Scope

The SAP describes the procedures for the following:

- Description of the lake area as a whole;

- Fish collection (including environmental data collection);

- Tissue sampling (for fish categories with fillet samples), decontamination, and packaging;

- Laboratory Analysis (EPA Method 1668C; NOAA or Bligh and Dyer (1959) for lipids), and;

- Quality Assurance.

1.3.3 Organization

The sampling and analysis for the Johnson Lake fish tissue monitoring study is based on the monitoring report requirements as described in the O&M Plan (DOF 2012b), with additional input as provided during discussions between Rob Webb of DOF ("Project Engineer"), Susan Sholl of Owens, Scott Maharry of Grette Associates, and DEQ personnel in early 2017. Further monitoring

report requirements will be incorporated based on comments from DEQ in the 2018 comment letter. The additional field sampling requirements specified by DEQ in the 2018 comment letter (Attachment D) include:

- level of sampling effort must be sufficient to ensure the required number of fish specified in the OMP (and herein) are collected and that fish are collected throughout the lake;
- fish in each category must adequately represent that category;
- use of a full range of available gear types to collect the targeted fish species in each sample category.

The comments from the 2018 DEQ letter related to laboratory procedures and reporting include:

- the evaluation of results should consider total PCB, PCB TEQ, and lipid normalized total PCB concentrations in addition to PCB congener 126 concentrations to support evaluation of data trends over time;
- laboratory protocols for sample preparation should specify that composite homogenates of fish tissue are created using equal mass from each individually homogenized fish sample;
- composite samples of each fish should be of the same type (species) and size (age) class, and;
- consistent with EPA Guidelines (EPA 2000), the differences between lengths of individuals in a composite sample and the average of the lengths should not exceed 10 percent and at least one replicate should also be analyzed.

The monitoring report for the 2022 fish tissue sampling effort will include, at a minimum, the following:

- the date of the fish tissue collection;
- location of fish collection;
- fish tissue PCB concentrations;
    - reported both as total PCB and by congener;
    - reported both on the basis of mass and lipid content;
    - reported as total PCB, PCB TEQ and, lipid normalized total PCB concentrations in addition to PCB congener 126 concentrations
- a summary of the results from the 2017 and 2004 sampling events;
- descriptions of the fish collected (number, species, length, mass) and which fish constituted which composite, and;

- results of cap inspection with photos (2017, to be provided by the Project Engineer).

2 DESCRIPTION OF JOHNSON LAKE

2.1 INSPECTION OF AREAS NEAR OUTFALLS TO JOHNSON LAKE

During the course of the fish sampling program, inspection of the areas near the outfalls to Johnson Lake will occur. Inspection of the outfalls will include recording of observations related to increased turbidity, discoloration, or algae growth. Photographs of the outfalls and immediately surrounding areas will be taken and included in the monitoring report.

2.2 INSPECTION OF WARNING SIGNS

In addition to the outfall inspections above, the condition and effectiveness of the fish warning signs installed around the perimeter of Johnson Lake will be assessed. For safety, multiple monitoring staff will participate in this assessment. In addition to recording observations of the signs and surrounding areas, photographs of the signs will be taken and included in the monitoring report.

3 FISH SAMPLING

3.1 FISH COLLECTION

The following fish collection program is consistent with the O&M Plan (DOF 2012b) as well as phone and email communication between the Project Engineer and DEQ personnel in early 2017 and the 2018 DEQ comment letter.

The fish collection will be conducted on foot or from a small vessel(s) using a waveform electro-shocker, traditional hook and line methods, and nets or seining. Fish will be collected from a variety of locations around and within the lake, as practicable, and where fish can be collected to represent general fish tissue concentrations lake-wide. Composite samples of each fish collected shall adequately represent the sampling category and be of the same type (species) and size (age) class. The collection will be consistent with EPA Guidelines (Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume 1 Fish Sampling and Analysis; EPA 2000), in a manner such that individual fish comprising a single composite sample should be of similar size (the smallest individual in a single composite should be no less than 75% of the total length of the largest individual).

The lake has been remediated with a thin-layer cap, as described in Section 1.2. Fish collection methods must not damage the cap. The Project Engineer advises that the cap is resilient enough for sampling methods which could require biologists to walk on it, including seining (R. Webb, personal communication).

Fish collection will target the following categories of fish. The goal for each category is to collect enough fish for the lab to composite five samples of the same fish type (fillets) and size range (whole fish) into a single composite sample for each category.

- Game fish fillets: One composite sample will target fish typically consumed by humans (e.g., largemouth bass).
- Panfish fillets: One composite sample will target panfish (e.g., bluegills); panfish tend to be smaller than game fish and may be consumed by humans.
- Rough fish fillets: One composite sample will target rough fish that are not typically targeted by humans but may be consumed by humans (e.g., suckers, carp).
- Large whole-fish: One composite sample will target fish greater than 350 mm in length that may be consumed by humans and large wildlife species[1].
- Small whole-fish: One composite sample will target fish <125 mm in length that may be consumed by smaller wildlife species.

[1] The Large Whole-Fish category in the O&M Plan references target fish between 125 and 300 mm. In a comment letter dated March 29, 2022 (DEQ ECSI # 2086), DEQ stated that "the large whole-fish tissue type in the O&M Plan should be updated to reflect fish >350 mm in length consistent with the RI and Baseline Risk Assessment."

All game and pan fish collected that are greater than 125 mm will be retained for potential analysis. Due to the scarcity of these fish in Johnson Lake, the analysis of individual fish may be required.

During the 2017 collection effort, a single white sturgeon (*Acipenser transmontanus*) approximately six feet in length was observed in Johnson Lake. The sturgeon was not harmed during the field collection and may still be present in the lake. If this is the case, interactions with the sturgeon will be avoided to the extent practicable. The sturgeon will be included in the list of potential bycatch species in the application for a Scientific Taking Permit obtained from ODFW for the project. The ODFW Scientific Taking Permit is included as Attachment A.

For replicate samples (minimum one replicate sample), the relative difference between the average length of individuals within any replicate sample and the average of the average lengths of individuals in all replicate samples should not exceed 10 percent. Replicate composite samples should be as similar to each other as possible. In addition to being members of the same species, individuals within each composite should be of similar length (size). The relative difference between the average length (size) of individuals within any composite sample from a given site and the average of the average lengths (sizes) of individuals in all composite samples from that site should not exceed 10 percent.

Where possible, fish should be of a harvestable size and weight. Samples collected will be representative of fish likely to be consumed by humans and large enough that this exposure would be reasonable.

Due to habitat limitations and low oxygen conditions previously documented in Johnson Lake, it may not be practicable to collect and analyze the numbers and types of fish samples described above as there may not be a significant fish population in the lake. A good faith effort (four days moving about the lake on foot or by boat, attempting to electroshock or otherwise collect fish that are present) will be made to characterize fish PCB concentrations that are representative of those existing in Johnson Lake. In the event that adequate fish are not collected from each category, a second 4-day collection and sampling effort will be made over the same period at a later date during 2022. In the event a second 4-day collection and sampling effort is required, DEQ will be provided advance notice. This approach is consistent with the O&M Plan (DOF 2012b).

The previous sampling indicates that small fish do not inhabit the center of the lake (ARCADIS 2004b). As such, the collection of small whole-fish composites (<125 mm) will prioritize perimeter sampling zones 6, 7, and 8 to evaluate small home range species closest to the Owens facility.

Sampling methods will include the use of a backpack electro shocker with dip nets, passive nets (e.g., gill net, Fyke net), beach seine, and hook and line angling.

Fish collected at each sample location will be enumerated by species, photographed, categorized (game fish, panfish, rough fish), and measured/weighed. A record will be kept of all fish collected, including species type, length, weight, method of capture, and whether selected for sampling or released. Fish collected during sampling from a specific location within the lake will be temporarily held in a decontaminated cooler filled with water from Johnson Lake. Once sampling activities from that area are complete, fish targeted for tissue analysis will be euthanized via CO_2

immersion (dry ice added to holding water), measured/weighed, wrapped in aluminum foil, placed in individually-labeled plastic bags, and stored on ice in collection bags for transport to ALS Global laboratories in Kelso, Washington for analysis. In the event that multiple areas of the lake are sampled concurrently (e.g., angling in one area while a gill net is deployed in another), fish from different areas of the lake will be kept in separate coolers and not mixed. Fish that are kept for analysis will be held on ice for the duration of the 4-day sampling event, and delivered to ALS Laboratories after the completion of sampling activities on the fourth day.

In an effort to implement humane practices, larger fish will be dispatched by stunning the fish with an appropriately sized blunt tool just above the eyes on the back of the skull followed by pithing using a sharp, decontaminated knife. Any fish not required for tissue analysis will be returned to the location in the lake from which they were collected prior to euthanasia.

If fish in any category continue to be collected after the goal of five individuals and replicate samples has been met, they may be used to replace previously collected fish to get a more spatially-representative sample of fish tissue in each category. This will be noted on field forms.

Five specimens of the same fish species shall be collected to form into a single whole-body composite sample. An additional five specimens of the same species should be collected to form a single fillet composite sample. This allows for more accurate whole-body estimates and allows for correct implementation of Section 11.2.4.1 Whole Fish Tissue in the attached ALS Standard Operating Procedure. If there are not enough fish collected to develop both whole body and fillet composites, whole body composites shall be the priority. This is consistent with the goals of both DEQ and the Oregon Health Authority to ensure risk is not underestimated for people that typically eat the whole fish.

While five fish per composite is the goal, if fewer than five fish of a species are collected, potential compositing with a smaller number of fish or the analysis of individual fish will be considered. In this case, decisions regarding compositing of type and sizes of fish will be made with DEQ coordination.

Fish will not be composited across species, in accordance with the EPA Guidance for Fish Sampling (EPA 2000).

Each sampling location will be recorded using a dGPS. This will include locations where any fish collection was not successful. Water temperature, turbidity, and dissolved oxygen levels will be collected in the middle water column at each sample location, along with weather conditions.

3.2 TISSUE SAMPLE COLLECTION, DECONTAMINATION, AND PACKAGING

Filleting of fish and compositing of samples will be done in the analytical laboratory, not in the field. When preparing euthanized fish for transport to the lab, care will be taken to avoid cross-contamination. Nitrile gloves and aluminum foil work surfaces will be changed regularly to avoid contamination.

Individual fish will be rinsed with analyte-free-ionized water, wrapped in aluminum foil, bagged, labeled, and placed in an ice cooler for shipment to the analytical laboratory.

The analytical laboratory will composite the fillet and whole-fish samples into the single composite samples for each category (Section 3). Each fish tissue sample composite will be created using equal mass from each individually homogenized fish aliquot, in accordance with the analytical laboratory's Tissue Sample Preparation Standard Operating Procedure (MET-TISP, dated 5/29/16; Attachment C). Filleting of fish will be done in accordance with the procedures in Attachment C as well as the procedures in Section 7.2.2.7 in EPA Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories: Volume 1 - Fish Sampling and Analysis (EPA 2000).

3.3 SAMPLE TRANSPORT AND CHAIN OF CUSTODY PROCEDURES

After collected and bagged/labeled, fish will be placed in the ice coolers and packed for shipping. The field representative will log each sample sent for analysis on a chain of custody (COC) form, noting sample identification code, date and time of collection, requested testing, and comments as appropriate.

All fish captured during the sampling effort(s) will be given a unique identifier. The sample code will allow individual fish to be correlated to the data collected, as well as determine which individual fish are included in composited samples in the lab. The sample identification scheme is described below.

- The first two letters of the sample identification code will identify the category of fish sampled or if the fish was returned to the lake, i.e., fish returned (FR), game fish (GF), pan fish (PF), rough fish (RF), large whole fish (LF), and small whole fish (SF);
- The first set of numbers in the sample code will be; the area of the lake from which the fish was captured;
- The second set of numbers in the sample code will be a sequential count of the fish captured
- If the fish is to be used as a replicate, a letter code (R) will follow the sequential number.

Two example fish sample codes are as follows:

GF-05-09-R;

where, "GF" = Game Fish, "05" = Area 5 within Johnson Lake, "09" = the ninth fish captured during the sampling effort, "R" = this is a replicate sample.

LF-02-15;

where, "LF" = Large Whole Fish, "02" = Area 2 within Johnson Lake, "15" = the fifteenth fish captured during the sampling effort.

Chain-of-custody procedures will commence in the field and will track the delivery of the samples to the laboratory. Upon transfer of possession to the testing laboratory, the chain-of-custody forms will be signed by the persons transferring custody of the samples.

4 LABORATORY ANALYSIS

4.1 TESTING FACILITIES

The collected fish will be delivered to and processed at ALS Global in Kelso, WA. The processed samples will then be shipped to ALS's laboratory in Houston, Texas, for analysis.

4.2 LABORATORY CHAIN OF CUSTODY

A laboratory chain-of-custody record for each set of samples will be maintained throughout all sampling, processing, and analytical activities. Information tracked by the laboratory chain-of-custody records include a sample identification number, date and time of sample receipt, analytical parameters required, location and conditions of storage, date and time of removal from and return to storage, the signature of the person removing and returning the sample, the reason for removing from storage, and final disposition of the sample.

4.3 CHEMICALS OF CONCERN (CHEMICALS OF INTEREST) SCREENING

The evaluation of results shall consider total PCB, PCB TEQ, and lipid normalized total PCB concentrations in addition to PCB congener 126 concentrations to support evaluation of data trends over time (DEQ 2018). They will also be reported relative to tissue mass and tissue lipid content.

4.4 TESTING METHODS

PCB congeners will be analyzed using EPA method 1668C. The storage temperature for this analysis is 4°C, holding time is 14 days.

Table 1. Sample holding time and storage criteria

Sample Type	EPA Method	Holding Time	Temperature	Sample condition
Fish tissue	1668C	14 days until extraction	4° C	fillets (with belly flap attached); whole fish
Fish tissue (lipids)	NOAA or Bligh and Dyer (1959)	14 days until extraction	4° C	fillets (with belly flap attached); whole fish

5 QUALITY ASSURANCE AND DATA VALIDATION

All sampling and analysis will be done in accordance with the existing DEQ-approved Quality Assurance Project Plan (QAPP) for the Johnson Lake Remediation Project (Arcadis 2004, Johnson Lake Investigation Work Plan, Appendix B, Quality Assurance Project Plan), as well as comments provided in the DEQ 2018 response letter to DOF. The Project QAPP is included as Attachment E.

All field data forms and chain-of-custody forms will be reviewed by the field lead prior to demobilization from the site to ensure completeness and correctness. The field lead will also be responsible for all fish species identification and for ensuring that each fish receives the proper sample identification code.

The field sampling team will attempt to collect replicate and duplicate samples concurrent with sampling activities for laboratory QA/QC. These attempts will be made to an extent practicable during the field efforts and through the course of sampling.

All laboratory QA/QC forms will be included as an appendix to the final report including method blanks, laboratory control samples, and calibration data.

6 REFERENCES

ARCADIS. 2004a. Johnson Lake Investigation Work Plan, Revision 2. Prepared for Owens-Brockway Glass Container, Inc. Portland Oregon, by ARCADIS G&M, Inc., Cleveland, Ohio. January 29, 2004.

ARCADIS. 2004b. Johnson Lake Site Investigation Report. Prepared for Owens-Brockway Glass Container, Inc. Portland Oregon, by ARCADIS G&M, Inc., Cleveland, Ohio. July 15, 2004.

ARCADIS. 2004c. Johnson Lake Investigation Work Plan, Revision 2, Appendix B, Quality Assurance Project Plan. Prepared for Owens-Brockway Glass Container, Inc. Portland Oregon, by ARCADIS G&M, Inc., Cleveland, Ohio. January 29, 2004.

Dalton, Olmsted & Fuglevand, Inc. (DOF) and Environ. 2011. Project Completion Report – Soil Remediation Action. Johnson Lake, Portland, Oregon. Prepared for Owens-Brockway Glass Container Inc. March 22, 2011.

Dalton, Olmsted & Fuglevand, Inc. (DOF). 2012a. Project Completion Report – Sediment Remedial Action. Johnson Lake, Portland, Oregon. Prepared for Owens-Brockway Glass Container Inc. September 24, 2012.

Dalton, Olmsted & Fuglevand, Inc. (DOF). 2012b. Remediation Operations & Maintenance Plan (O & M Plan): Sediment Remedial Action. Johnson Lake, Portland, Oregon. Prepared for Owens-Brockway Glass Container Inc. December 20, 2012.

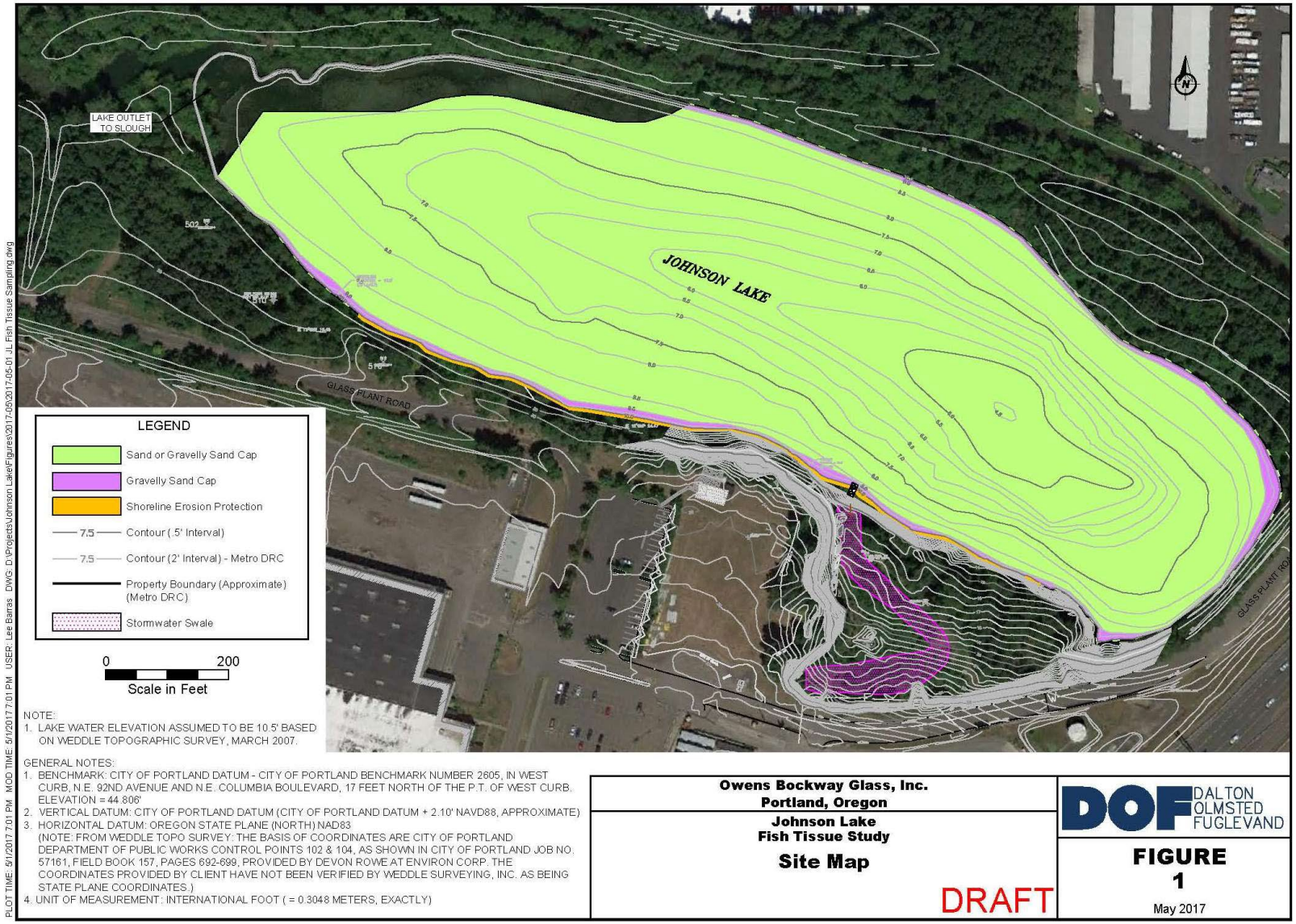
Grette Assoc. 2018. Revised Johnson Lake Fish Tissue Monitoring Study – Monitoring Report

Oregon Department of Environmental Quality. (DEQ). 2018. Johnson Lake Fish Monitoring Study Comment Response Letter. Prepared for Dalton Olmsted Fuglevand

United States Environmental Protection Agency. (EPA). 2000. EPA Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories. Vol. 1 Fish Sampling and Analysis. Third edition. Office of Water. EPA 823-B-00-007.

Webb, R. personal communication. Phone call with M. Shelton, April 26, 2017.

Figure 1: Site Map of Johnson Lake



JOHNSON LAKE FISH TISSUE MONITORING STUDY

SAMPLING AND ANALYSIS PLAN
ATTACHMENT A: ODFW SCIENTIFIC TAKING PERMIT

Oregon
Kate Brown, Governor

Department of Fish and Wildlife
Fish Division
4034 Fairview Industrial Drive SE
Salem, OR 97302
(503) 947-6201
FAX (503) 947-6202
www.dfw.state.or.us/

2022 - SCIENTIFIC TAKING PERMIT - FISH

Permit Applicant: **Scott Maharry**
Organization: **Grette Associates, LLC**

Permit Number: **26458**
Project Title: **Johnson Lake Sediment Remedial Action: Fish Tissue Monitoring Study**
Dates Valid: **04/05/2022 – 10/05/2022**

Federal Authorization: **This permit does not provide any federal authorization for ESA listed fish species.**

Principal Investigator: **Scott Maharry**

Co-Investigator(s): **Janae Dinkins, Donny Neel, Chad Wallin**

PI Signature:_____ CI Signature(s): _____

Terms and Conditions of This Permit:

1. The Permit Holder, Principal Investigator, Co-Investigator, and all people working under their supervision shall at all times observe and comply with all federal and state laws, including the Endangered Species Act of 1973, as amended, and lawful regulations issued thereunder, which relate to threatened or endangered plant or animal species while performing activities described in the permit application. ODFW's approval of this permit does not certify that Permittee's activities described in its application are lawful under the federal ESA. Permittee's compliance with permit conditions is not in lieu of compliance with any federal requirements related to the federal Endangered Species Act. General conditions of Oregon Revised Statutes and Oregon Administrative Rules apply to this permit that cannot be used in lieu of any permit required by federal law or regulation. Permission to sample in areas where federally protected fish may occur is contingent upon the permittee obtaining necessary authorization from the appropriate federal agency and acting in accordance with the conditions established by the federal government. If a condition on this permit conflicts with a condition on a federal permit or authorization, then the permittee must comply with the more restrictive condition.
2. This permit is not transferable and must be carried while collecting. The Principal Investigator and all Co-Investigators must sign their own copy of the permit.
3. Persons not named on the permit may assist in collecting only while accompanied by the Principal Investigator or Co-investigator(s) listed above.
4. Access to private property is contingent on landowner permission. This permit does not authorize trespassing.
5. This permit is not valid in any refuge, park, city, wildlife area, or area closed to collection without written approval of manager or administrator. When collecting from state parks, please contact Oregon Parks and Recreation Department to inquire about the need for a scientific research collection permit from them - https://oprdpermits.org/
6. Sampling must be coordinated with local Oregon Department of Fish and Wildlife District Fish Biologists (contact list attached in permit). All requirements contained in the district biologist comments section of the application must be followed and you must notify them prior to sampling in their districts. Oregon State Police must also be notified prior to sampling, preferably by contacting local offices. http://www.oregon.gov/osp/Pages/contact_us.aspx or else the Northern (503-375-3555) or Southern

JOHNSON LAKE FISH TISSUE MONITORING STUDY

SAMPLING AND ANALYSIS PLAN

ATTACHMENT B: ODEQ MAY 25, 2017, SAP APPROVAL AND COMMENTS

Oregon
Kate Brown, Governor

Department of Environmental Quality
Northwest Region Portland Office
700 NE Multnomah St., Suite 600
Portland, OR 97232
(503) 229-5263
FAX (503) 229-5471
TTY 711

May 25, 2017

Mr. Rob Webb, Principal Engineer
Dalton Olmsted Fuglevand
1236 Finn Hill Road
Poulsbo, WA 98370

RE: 2016 Johnson Lake Fish Tissue Monitoring Study
ECSI # 2086

Dear Rob:

Thank you for providing the *Sampling and Analysis Plan* (dated May 9, 2016) for fish tissue monitoring in Johnson Lake, located west of I205 adjacent to the Owens Brockway Glass Container, Inc. facility (5850 NE 92nd Drive in Portland, Oregon). This plan was submitted as part of the long-term monitoring of the remedial action implemented by Owens Brockway Glass Container, Inc as described in the *Remediation Operations and Maintenance Plan-Sediment Remedial Action* (December 20, 2012). DEQ approves the plan assuming the following:

1. A Scientific Taking Permit for fish is obtained from Oregon Department of Fish and Wildlife and included as an appendix to the fish tissue sampling results report.

2. All methods indicated in Section 2.1 will be utilized throughout the lake including off-shore areas of deeper water unless sufficient samples are obtained with a subset of the methods. This includes boat electro-shocking, deeper water angling, and off-shore netting with gill and Fyke nets. We expect these methods will be necessary to obtain representative fish, particularly those fish large enough to support determinations on fish advisories.

3. Fish sampling, processing and analysis procedures will follow EPA guidance provided in *Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories Volume 1 Fish Sampling and Analysis, 2000* (https://www.epa.gov/fish-tech/epa-guidance-developing-fish-advisories).

4. A minimum of three fish will be included in each composite consistent with EPA guidance (five is preferred). Individual fish comprising a composite should be of similar size (smallest individual in a composite should be no less than 75% of the total length of the largest individual). For evaluation of impacts to human health and larger ecological receptors, fish should be of harvestable size or weight. Please coordinate with DEQ

during sampling so that we can participate in reviewing collected fish and determining an appropriate compositing plan.

5. Processing of fillets will be conducted in the laboratory rather than in the field. Laboratory clean room conditions are required to properly contain and control fish fluids during resection. Laboratory standard operating procedures for fish processing, resection, and homogenate preparation should be provided to DEQ in advance of fish collection. Fillets should not be rinsed as a standard practice as suggested in the work plan. Rinsing is only a contingency if there is rupture of organs during filleting which should be avoided consistent with standard laboratory practices. If rupture occurs, please attempt to consult with DEQ prior to selecting fish for analysis.

Please call me if you have any questions at (503) 229-6148.

Sincerely,



Jennifer Sutter, Project Manager
NWR Cleanup and Tanks

Cc: Sue Sholl, Owens Illinois
Susan Smith, Owens Illinois
Rebecca Hillwig, Oregon Health Authority
Tim Dean, City of Portland
Jennifer Peterson, DEQ NWR Cleanup and Tanks
ECSI File #2086

DEQ-DC1

JOHNSON LAKE FISH TISSUE MONITORING STUDY

SAMPLING AND ANALYSIS PLAN

ATTACHMENT C: ALS STANDARD OPERATING PROCEDURE MET-TISP (5/29/16) – TISSUE SAMPLE PREPARATION

ALS Standard Operating Procedure

DOCUMENT TITLE:	TISSUE SAMPLE PREPARATION
REFERENCED METHOD:	N/A
SOP ID:	MET-TISP
REVISION NUMBER:	10
EFFECTIVE DATE:	5/29/2016



TISSUE SAMPLE PREPARATION

ALS-KELSO

SOP ID:	MET-TISP	Rev. Number:	10	Effective Date:	5/29/16

Approved By: _____ Date: 5/11/16
Department Manager/Technical Director – Jeff Coronado

Approved By: _____ Date: 5/11/16
QA Manager – Carl Degner

Approved By: _____ Date: 5/12/16
Laboratory Director – Jeff Grindstaff

Issue Date: _____	Doc Control ID#: _____	Issued To: _____

ANNUAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR TWELVE ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

TABLE OF CONTENTS

1.SCOPE AND APPLICATION 3
2.METHOD SUMMARY 3
3.DEFINITIONS 3
4.INTERFERENCES 4
5.SAFETY 4
6.SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE 4
7.STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS 4
8.APPARATUS AND EQUIPMENT 4
9.PREVENTIVE MAINTENANCE 5
10.RESPONSIBILITIES 5
11.PROCEDURE 5
12.QA/QC REQUIREMENTS 13
13.DATA REDUCTION AND REPORTING 13
14.CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA 13
15.METHOD PERFORMANCE 14
16.POLLUTION PREVENTION AND WASTE MANAGEMENT 14
17.TRAINING 14
18.METHOD MODIFICATIONS 15
19.REFERENCES 15
20.CHANGES SINCE THE LAST REVISION 15

TISSUE SAMPLE PREPARATION

1. SCOPE AND APPLICATION

1.1. This standard operating procedure describes procedures for the initial preparation of tissue samples prior to sample analysis. Customer-specific contracts or statement of works (SOWs) with alternate procedures will take precedence over this SOP.

1.2. This SOP is intended to provide guidance for the preliminary preparation of tissue samples prior to the sample aliquoting and analytical preparation described in individual analytical SOPs. The procedures described in this SOP also apply to compositing and subsampling of tissue samples for analyses to be subcontracted.

2. METHOD SUMMARY

2.1. Tissue samples are inherently heterogeneous requiring special considerations in order to obtain a truly representative sample aliquot for analysis. This SOP provides guidance for handling tissue samples prior to the sample preparation steps described in analytical SOPs. This SOP applies to samples delivered to the lab in whole body form or in the form of pre-dissected tissues.

2.2. The sample handling strategy must consider:

- what analyses are to be performed (metals, organics, or both, and VOC or non-VOC),
- how much sample is available
- are the analyses to be performed on individual samples or composite homogenates,
- are the analyses to be performed on whole body, edible portions or specific organs, and
- are any of the analyses going to be subcontracted which may require subsampling.

2.3. Proper preparation and handling of tissue samples is required to obtain a representative sample, avoid contamination, and to ensure loss of sample and target constituents is minimized.

3. DEFINITIONS

3.1. Sample: The material presented to the laboratory for analysis or testing.

3.2. Sample Aliquot: A representative part or portion of a sample for analysis which is a fraction of the whole sample. See subsampling also.

3.3. Compositing: The process by which sample aliquots from two or more samples are united to form a combined sample which is subsequently analyzed.

3.4. Subsampling: The process by which a representative portion is obtained from a whole sample.

3.5. Service Request: The service request (SR) is a document prepared at the time of sample receipt and summarizes sample analysis and reporting instructions about a customer's sample(s).

3.6. QAPP: Quality Assurance Project Plan document provided by the client specifically written for their project.

3.7. VOC Analyses: Volatile organic compounds (VOC) analyses, including halogenated and aromatic volatile organic compounds and gasoline range organics (GRO) analyses.

3.8. Non-VOC Analyses: Any analysis other than a VOC analysis.

4. INTERFERENCES

4.1. If precautions are not taken, cross-contamination can occur when handling tissue samples in large quantities. Equipment must be thoroughly cleaned as described in this SOP and related SOPs. Also, the SOP describes the use of homogenization and rinsate blanks to monitor any possible contamination.

4.2. For organics samples, polypropylene and polyethylene (plastic) surfaces, implements, and containers are a potential source of adsorption and contamination and should not be used. Gloves should be talc free and of non-contaminating materials.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in MSDSs where available. Refer to the ALS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

6.1. Refer to the determinative method.

7. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

7.1. Not applicable to this procedure.

8. APPARATUS AND EQUIPMENT

Note: Refer to the Procedure section for specific equipment used based on the determinative analysis to be performed. The use of implements and surfaces may vary depending on the analyses to be performed.

8.1. Hobart Food Chopper, or comparable device.

8.2. Tissumizer.

8.3. Waring blender, or similar device.

8.4. Freeze-drier, Labonco or equivalent.

8.5. Glass or PTFE cutting boards.

8.6. Knives and cutting implements – refer to Procedure section.

8.7. Standard laboratory glassware (beakers, scintillation vials, etc.)

8.8. VOA vial – pre-cleaned, 40ml with Teflon-lined cap.

8.9. Pre-cleaned glass jars with PTFE lined lids, various sizes.

8.10. Gloves: Should be talc free and of non-contaminating materials.

8.11. Heavy duty aluminum foil.

9. PREVENTIVE MAINTENANCE

9.1. No specific maintenance steps are needed other than normal cleaning and inspection of apparatus.

9.2. For organics samples, polypropylene and polyethylene (plastic) surfaces, implements, and containers are a potential source of adsorption and contamination and should not be used. Gloves should be talc free and of non-contaminating materials.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. Sample custodians, together with project chemists and department supervisors, are responsible for documenting any required sample preparation (including the percent solids or percent lipids determination if required) on the service request. All personnel preparing tissue samples should be familiar with the contents of this document prior to commencing work.

10.3. Tissue sample preparation is to be performed only by lab analysts instructed in the proper handling techniques outlined in this SOP. It is the responsibility of the analyst to perform this procedure to complete all documentation required for data review.

11. PROCEDURE

11.1. Sample Login

Any special sample handling must be noted on the service request and on a label attached to the sample itself. During sample receiving, a sample custodian must follow the procedures described in Section 11.1.1.

11.1.1. Tissue Samples with Limited Quantity

An assessment of the required sample quantity should be made by the project chemist when taking delivery of the sample. This assessment must take into consideration the tests, the required detection limits and the necessary quality assurance samples. If the quantity of sample given to the laboratory is insufficient for the analyses requested, the sample custodian will, along with the project chemist estimate the total amount of sample available. A "LIMITED SAMPLE VOLUME" tag is attached to the sample on which is recorded the estimated sample quantity.

In some cases it may be beneficial to perform sample preparations as described in this SOP prior to estimating the sample amount. In this case, the analyst preparing the sample will provide the project chemist with an estimate of the amount available.

The project chemist must determine if limited sample quantity exists and set the priorities for the analyses and, if possible, estimate the quantity of sample to be *used for each test*. This information is to be documented and placed in the project file and on the service request to communicate to the laboratory staff. For example, 8081 use 10 g; metals use 1g.

NOTE. Samples that are quantity limited and require multiple analyses must be identified as soon as possible. Optimally, this should happen during sample login; however, discovery at any time should trigger appropriate actions as described in Section 11.1.1.

11.2. Sample Homogenization

This section outlines the steps for preparing homogenous samples of whole fish, edible fish (fillets), edible shellfish, worm composite homogenates, eggs, and plant tissues.

11.2.1. Samples for Organics Analyses:

Equipment used for the processing of tissue samples for organics analyses should be of stainless steel, anodized aluminum, glass or polytetrafluorethylene (PTFE). Polypropylene and polyethylene (plastic) surfaces, implements, and containers are a potential source of adsorption and contamination and should not be used. Gloves should be talc free and of non-contaminating materials. Filleting should be done on glass or PTFE cutting boards that are cleaned properly between samples or on cutting boards that are covered with heavy duty aluminum foil (hexane rinsed) that is changed between samples. Tissue should be handled with precleaned, high quality, corrosion-resistant stainless steel instruments. Fillets or homogenate should be stored in cleaned glass jars of suitable dimensions with PTFE lined lids. If the sample is to be analyzed for VOCs, the homogenization steps should be performed on sample tissue that is partially frozen or chilled. An aliquot of the homogenate should be placed in a clean 40ml voa vial and libeled "FOR VOA ANALYSIS ONLY".

Prior to handling each sample, utensils, cutting boards and containers should be washed in a detergent hot water solution and rinsed with tap water, hexane, and DI water. Pre-cleaned, certified sample containers may be used without further cleaning. If the sample is to be analyzed for VOCs, methanol is substituted for the rinsing of implements with acetone and the hexane rinsing of the aluminum foil. Exposure to solvent vapors must be minimized.

11.2.2. Samples for Metals Analyses:

Equipment used in the processing of samples for metals analyses should be of PTFE, ceramic, polypropylene or polyethylene. Filleting should be performed on PTFE cutting boards which are cleaned after each sample. Knives with titanium or high quality stainless steel blades may be used for tissue resections. Tissue should be stored in glass jars with PTFE lined lids.

Prior to sample handling, utensils, cutting boards and containers should be washed in a detergent hot water solution, rinsed with tap water, 25% HCL (except metal utensils), and DI water. Pre-cleaned, certified sample containers may be used without further cleaning.

11.2.3. Samples for both Metals and Organics Analyses:

If the sample is to be prepared for both organics and metals, care must be taken to use equipment and cleaning procedures that are non-contaminating for both. Quartz, ceramic, glass and PTFE are recommended materials for sample processing equipment. Knives with titanium or high quality stainless steel blades may be used for tissue resections. Glass or PTFE cutting boards should be used. If the sample is to be analyzed for VOC's, the homogenization steps should be performed on sample tissue that is partially frozen or chilled. An aliquot of the homogenate should be placed in a clean 40ml voa vial and labeled "FOR VOA ANALYSIS ONLY".

Prior to handling each sample, utensils, cutting boards and containers should be washed in a detergent hot water solution and rinsed with tap water, acetone, methanol, or hexane (as appropriate), and DI water. Precleaned, certified sample containers may be used without further cleaning. Non-metallic surfaces and utensils should also be rinsed with 25% HCL followed by DI water. If the sample is to be analyzed for VOCs, methanol is substituted for the rinsing of implements with acetone. Exposure to solvent vapors must be minimized.

11.2.4. Sample Preparation

Each tissue sample may be homogenized in the original glass bottle container if there is sufficient space to allow thorough mixing. If homogenization is not achievable in the original container, place the entire sample contents into a clean glass jar. Generally, liquids contained in the container are to be considered part of the sample. If the sample requires size reduction prior to homogenization, chop the sample into the 1-2" chunks using a titanium or stainless steel bladed knife. Large samples may require the use of industrial food processors such as a Hobart Food Chopper, or comparable device. Size-reduced chunks of tissue are thoroughly homogenized to a paste-like consistency using a Tissumizer, Waring blender, or similar device until it reaches a paste-like consistency. Transfer the sample paste to a glass jar for storage and freeze until ready for sample extraction. The new sample

container is labeled with the sample I.D., the word "homogenized", initialed, and dated.

11.2.4.1.Whole Fish Tissue

Samples may be frozen in the field or in the laboratory. While still partially frozen, rinse the fish with DI water to remove extraneous materials and liquids. Cut the fish into appropriate size chunks and mechanically macerate the sample using cutting tools appropriate for the size of the sample and the analysis type. If necessary, process fish tissue chunks through the Hobart Food Chopper. To ensure thorough mixing, divide the ground sample into quarters, mix opposite quarters and then mix halves. Homogenize sample using a Tissumizer or Waring blender until it reaches a paste-like consistency. Transfer the sample paste to a glass jar for storage and freeze until ready for sample extraction. The new sample container is labeled with the sample I.D., the word "homogenized", initialed, and dated.

11.2.4.2.Edible Fish Tissue

If the client or QAPP indicates that only edible tissue be analyzed, the fish must be filleted. If the sample arrives pre-filleted, the sample tissue may be frozen before processing. If the sample is not yet filleted, the sample should remain chilled until the filleting is completed. Freezing can result in the contamination of edible tissues from the bursting of internal organs. Fish having ruptured internal organs should be noted on the prep benchsheet and the Project Chemist consulted. Rinse the fish with DI water to remove extraneous materials and liquids. Remove scales from scaled fish or skin from non-scaled fish. Rinse the fish again prior to filleting. A separate or clean cutting board should be used for filleting. Gloves should be changed between samples. Carefully remove the fillets from the carcass by following the steps outlined in Appendix A. Care should be taken to avoid contaminating fillet with inadvertent puncture of internal organs. Cut the fillet tissue into appropriate size chunks and mechanically macerate the sample using cutting and grinding tools appropriate for the size of the sample and the analysis type. Proper selection of maceration equipment must consider the potential contaminants, sample size/volume and amount of tissue likely to be lost in using the equipment.

Divide the ground sample into quarters, mix opposite quarters and then mix halves. Again homogenize the sample using an appropriate blending mixer. Continue repeating this process until the sample is truly homogenous and no chunks of tissue remain. Freeze sample until ready for extraction.

11.2.4.3.Shellfish Tissue

Shellfish should be frozen as soon as possible after receipt by the laboratory unless samples can be prepared within 48 hours of sampling. Edible portions of various shellfish are described below and resection described in Appendix B. Thawing of frozen shellfish samples should be kept to a minimum during tissue removal to avoid loss of liquids. Shellfish should be rinsed with DI water prior to tissue removal to dislodge external debris. When multiple

organisms constitute a single sample, the edible tissues are collected, composited and homogenized.

11.2.4.3.1.Bivalve mollusks (oysters, clams, mussels, and scallops).

Bivalves are typically prepared by severing the adductor muscle, prying open the shell, and removing all of the soft tissue. The soft tissue includes viscera, meat, and body fluids.

11.2.4.3.2.Crabs

Edible tissue includes all leg and claw meat, back shell meat and body cavity meat. Internal organs generally are removed. If the crab is soft shelled, the entire crab is used in the sample.

11.2.4.3.3.Shrimp and Crayfish - Edible tissue includes the tail meat.

11.2.4.3.4.Lobster - Edible tissue includes the tail and claw meat.

11.2.4.4.Worms

Samples are typically supplied to the lab in sample jars containing multiple organisms. Liquid and specimens constitute the entire sample and are blended together typically in the sample container. When a worm sample containing dirt particles or significant amounts of water is encountered, the technician should contact the project chemist to seek guidance from the client.

11.2.4.5.Eggs

Avian eggs are typically removed from the shell and blended. Aquatic eggs are blended including the soft shell.

11.2.4.6.Internal Organs Extraction

Organs such as livers or kidneys must be identified and removed by an experienced sample technician following clear written resection procedures or other guidance provided by the client.

11.2.4.7.Plant Tissue

Plant tissue should be handled using the size reduction, homogenization and implement cleaning steps outlined in Sections 11.2.1, 11.2.2, 11.2.3, and 11.2.4. Where these procedures are inappropriate, specific written procedures or guidance from the client is recommended.

If drying is requested by the client or is project-specified, a subsample for mercury analysis is taken from the wet sample, and then the plant tissue is dried at 60˚C prior to homogenization.

11.2.4.8.Small Mammals and Rodents

11.2.4.8.1. There are two primary concerns in working with small mammals and rodents: safety and sample homogenization.

11.2.4.8.2. Small mammals are potential carriers of lethal viruses, such as hantavirus and rabies, and bacteria that can be contracted through inhalation or direct contact. Typically, these organisms are excreted in the feces and distributed on the air as the fecal matter dries. During the sample preparation process, tissue is typically freeze-dried in order to calculate a percent solids value and to analyze for metals. As such, it is possible to increase the potential for dispersion of the bacteria or viruses after the sample is homogenized and processed. Prior to processing, all samples should be stored frozen.

11.2.4.8.3. Prior to sample homogenization, instructions should be received from the client regarding the processing of the hide. For organics, it is recommended that the hides be left on the carcass and the entire sample be homogenized. For metals, there is a potential for accumulation in the hair. As a non-digestible portion of the rodent, inclusion of the hair may result in a high bias if the data is to be used in estimating bioaccumulation up the food chain. Skinning may be a preferred alternative when metals are the primary chemicals of concern.

11.2.4.8.4. Homogenization should be done while the carcass is still partially frozen.

11.2.4.8.5. If the hide is to be included in the homogenization, snip the feet from the animal using stainless steel scissors.

The tail should be removed if it will prevent complete homogenization of the sample (e.g., the tail of a mouse or rat may result in incomplete homogenization and should not be included with the sample). Remove seeds, grasses, and mud from the hide.

11.2.4.8.6. If the hide is to be removed from the carcass, make an incision through the skin on the back of the neck (do not cut into the muscle). In most cases, the hide can be removed by pulling the incision horizontally along the back in one direction, and over the ears, head and snout in the opposite direction. The eyes are usually lost during this procedure. Continue to skin the animal by peeling the hide over the hind legs, off the underside of the animal, and around the front legs. The hide is removed at the hind legs and the snout. Care should be taken not to tear the connective tissue under the hide. Fat should be scraped from the hide when possible and included with the sample. Rinse the skinned carcass with DI water to remove any hair or dirt that has accumulated during the skinning procedure.

11.2.4.8.7. Homogenize the sample using a stainless steel Waring blender. Select a blender cup that is sized in accordance with the amount of sample to be homogenized. That is, small samples should be homogenized using small blender cups. This will improve the overall homogenization and recovery of the sample. Continue to mix the

sample into a paste like consistency. Make sure no chunks of muscle, hide, or bone are distributed in the sample. Transfer the sample paste to a glass jar for storage and freeze. The new sample container is labeled "homogenized", initialed, and dated.

11.2.5. General Provision for Handling Large Sample Mass

In some cases, large specimens will be received by the laboratory for homogenization prior to chemical analysis. For the purpose of this SOP, 'large' is defined as requiring preliminary size reduction to allow sequential processing of the sample. Sub-samples of the whole specimen should be cut to a size appropriate for the blender, mixer, or grinder that will be used. After each individual fraction is processed, the homogenized material is added to a reservoir large enough to hold all fractions as they accumulate. The reservoir will be constructed of a material suitable for the analytical application as defined under Section 10.2.3. For very large specimens (i.e. >20 pounds), high grade stainless steel containers are used (large bowls or small drums).

Blending of the combined fractions to achieve a whole homogenous material is achieved via manual mixing. In general, this is accomplished using a high grade stainless steel paddle or spoon of appropriate size (i.e. relative to the whole homogenate). Very large specimens (i.e. >20 pounds) generally require secondary processing through the grinder, particularly when large amounts of skin, bone, and/or cartilage is present. In these cases, the Hobart grinder is generally used.

11.3. Compositing

Each sample is to be logged in and receive a lab code. Additionally, the sample composite also is assigned a lab code. The compositing process is to be performed by trained staff. It is to be performed in an area free of contamination. It is imperative that the samples are treated in a manner consistent with the requirements of the tests to be performed on the composited sample. Compositing of homogenates should be performed according to this SOP or specific instructions provided by the client.

11.3.1. Documentation (use applicable bench sheet)

The analyst preparing the composite will document

- that homogenization was done before removing an aliquot,
- the quantity of each (field or discrete) sample used for the composite,
- the date and time of compositing, and
- any other pertinent observations.

11.3.2. Tissue Samples with Limited Quantity

Samples and sample composites that are quantity limited will be handled by the same procedure as described in Section 10.1.1

11.3.3. Compositing Procedure

11.3.3.1.Each tissue sample is first homogenized as per instructions in Section 11.2.

11.3.3.2. An equal weight of sample aliquot from each of the homogenized samples is weighed into a clean glass sample bottle. The amount to be weighed of each sample will depend upon the number of analyses to be performed on the composite and if the quantity of any individual sample is limited.

11.3.3.3. The mixture of the individual sample aliquots is thoroughly homogenized in the glass container. The composite sample bottle is labeled with:

- the name of the composite,
- the lab code of the composite,
- the analyst's initials
- the date of composite preparation
- The composite sample and the remaining (individual, discrete) samples are stored frozen until analysis.

11.3.4. Tissue Samples Requiring VOC Analyses

A separate aliquot of the composite homogenate should be placed in 40ml voa vial container for later analysis by the VOA department. Each container should be labeled with the lab identifier, date, initials, and "FOR VOA ANALYSIS ONLY". To minimize losses of volatile constituents, the sample should be kept as cold as possible, the work should be completed as quickly as possible, and the VOA vial filled to the top to minimize head space.

11.4. Sub-sampling

The sample is first thoroughly homogenized as per Section 11.2. A sample aliquot is removed and placed into a clean glass container of appropriate size and labeled as follows:

- the name of the sample,
- the lab code of the sample,
- "homogenized" written on the label,
- the purpose of the sub-sample (e.g. "dioxin subsample")
- the analyst's initials
- the date.

11.5. Freeze-Drying

11.5.1. Depending on project specifications, samples may require freeze-drying. Freeze-drying may be performed on a separate portion of sample to determine % Freeze-Dried Solids, or may be done on the analytical subsample for certain tests. The analyst should obtain direction from the supervisor and/or Project Chemist.

11.5.2. Weigh 5-8 g of sample (wet weight) into a scintillation vial. Freeze the sample for at least 2 hours.

11.5.3. Remove the sample from the freezer and place in the freeze drier for at least 24 hours or longer if necessary for the particular sample matrix.

11.5.4. Record the measurements on the applicable bench sheet.

11.5.5. When freeze drying samples that require PFOA/PFOA, aluminum foil must be placed on the container before putting the lid on, after the freeze drying process and before freezing the sample.

12. QA/QC REQUIREMENTS

12.1. A rinsate blank should be prepared to accompany each batch of tissue samples. The blank is comprised of a collection of DI water rinses of cleaned equipment (knives, cutting boards and mixers/grinders) ***prior*** to the commencement of sample batch preparation. If contamination of the samples is suspected, the rinsate blank is extracted and analyzed for contaminants. The rinsate blank should be labeled with the extraction date and the associated SR numbers and stored at 4˚ C. In the event that contamination is suspected, the rinsate blank can be analyzed to confirm the presence of contaminants in the tissue preparation process.

12.2. A homogenization blank is prepared to determine if the homogenization equipment was effectively cleaned between samples. Unless a project plan specifies otherwise, the laboratory prepares two homogenization blanks with each shift of sample preparation. One is a 500 mL aliquot for Metals testing and the other is a 1000 mL aliquot for Organics testing. Any requirements other than the labs default procedure must be defined in the project plan and communicated to the laboratory.

12.2.1. Some project quality plans may require homogenization blanks between each sample. Following the blending of a tissue sample decontaminate the Hobart mixer (model HCM62) by following these steps:

- Wash the bowl, blade assembly, and lid with soap and hot water.
- Rinse all parts with deionized water.
- Move to fume hood and hexane rinse all parts.
- Allow excess hexane to evaporate.

12.2.2. Reassemble the mixer and make ready for the next sample.

12.2.3. Fill the bowl with deionized water and turn the mixer on for the approximately average time used for the type of samples being processed.

12.2.4. Aliquot the deionized water to bottles appropriate for the testing being conducted and preserve accordingly. If insufficient sample volume is produced for the required testing, repeat the procedure after the next tissue sample is homogenized.

13. DATA REDUCTION AND REPORTING

13.1. Sample handling documentation must include information about sample homogenization (was it done or not), compositing, and sub-sampling. The established and applicable data bench sheets provide a means for recording this information. Completed bench sheets listing the sample handling information are filed in the project file with the raw data.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1. Refer to the SOP for *Non Conformance and Corrective Action (CE-QA008)* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels
- Sample holding time missed due to laboratory error or operations
- Deviations from SOPs or project requirements
- Laboratory analysis errors impacting sample or QC results
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc)
- Sample preservation or handling discrepancies due to laboratory or operations error

15. METHOD PERFORMANCE

15.1. Refer to determinative methods.

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

16.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Environmental Health and Safety Manual.

16.3. This method uses non-halogenated solvents and any waste generated from this solvent must be collected. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

17. TRAINING

17.1. Training outline – Training Plan

17.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

17.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst until the supervisor feels the new employee can work independently. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.2. Training is documented following ADM-TRAIN, ALS-Kelso Training Procedure.

When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. METHOD MODIFICATIONS

18.1. This section is not applicable because this procedure is a laboratory developed method.

19. REFERENCES

19.1. Kateman and L. Buydens, *Quality Control in Analytical Chemistry*, Second Edition, John Wiley & Sons, Inc., New York, NY, 1993: Chapter 2 on Sampling and especially sections 2.5 (Sample Quality) and 2.7 (Handling of Samples).

19.2. *Guidance For Assessing Chemical Contaminant Data For Use In Fish Advisories;* Volume 1; Fish Sampling and Analysis, 3rd Edition; USEPA Office of Water; EPA 823-B-00-007; Nov 2000.

19.3. *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*; Tetra Tech, Inc.; final report TC-3991-04 Recommended Guidelines for Measuring Organic Compounds in Puget Sound Sediment and Tissue Samples Revision April 1996.

19.4. *PCB's and Mirex In Fish Tissue and Clams* New York State Department of Health Wadsworth Center For Laboratories and Research; Albany, N.Y. 10/6/81

19.5. *Draft Method 1613-Tissue*; Determination of PCDDs and PCDFs in Fish and Other Tissue Using Method 1613; USEPA Office of Water June 1993.

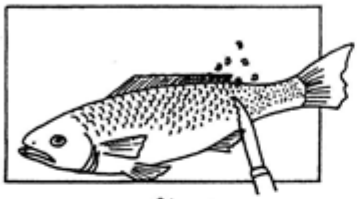
20. CHANGES SINCE THE LAST REVISION

20.1. Signature page: Updated Quality Assurance manager.
20.2. Added the sentence in Section 11.5.5.

APPENDIX A
Fish Filleting Procedure

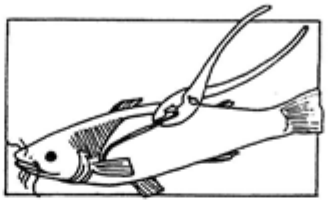
1 Scaled Fish

After removing the scales (by scraping with the edge of a knife) and rinsing the fish:



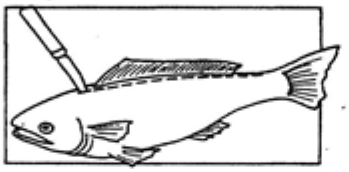
1b Scaleless Fish

Grasp the skin at the base of the head (preferably with pliers) and pull toward the tail.



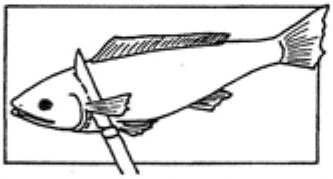
Note: This step applies only for catfish and other scaleless species.

2



Make a shallow cut through the skin (on either side of the dorsal fin) from the top of the head to the base of the tail.

3



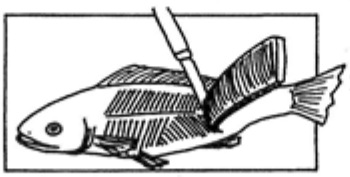
Make a cut behind the entire length of the gill cover, cutting through the skin and flesh to the bone.

4



Make a shallow cut along the belly from the base of the pectoral fin to the tail. A single cut is made from behind the gill cover to the anus and then a cut is made on both sides of the anal fin. Do not cut into the gut cavity as this may contaminate fillet tissues.

5



Remove the fillet.

Source: U.S. EPA, 1991d.

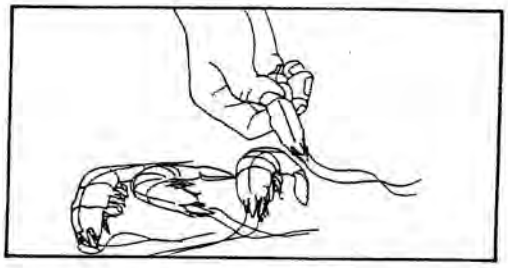
Figure 7-3. Illustration of basic fish filleting procedure.

APPENDIX B
General Procedure for Removing Edible Tissues from Shellfish

Heading, peeling and deveining shrimp

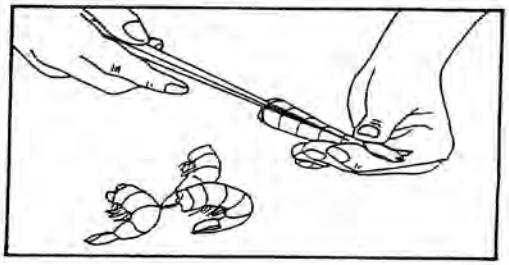
1

To head a shrimp, hold it in one hand. With your thumb behind shrimp head, push head off. Be sure to push just the head off so that you do not lose any meat.



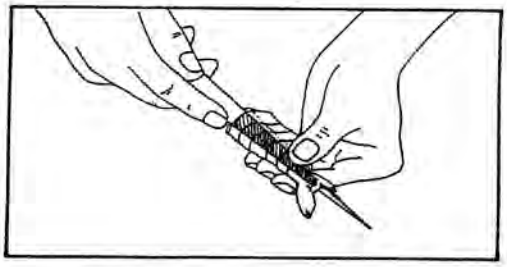
2

If using a deveiner, insert it at head end, just above the vein.



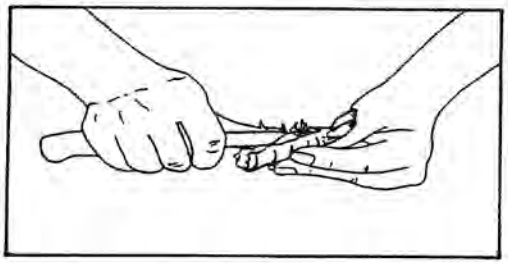
3

Push through shrimp to the tail and split and remove shell. This removes vein at the same time.



4

If you prefer to use a paring knife, shell shrimp with your fingers or knife. Then use knife to gently remove vein.



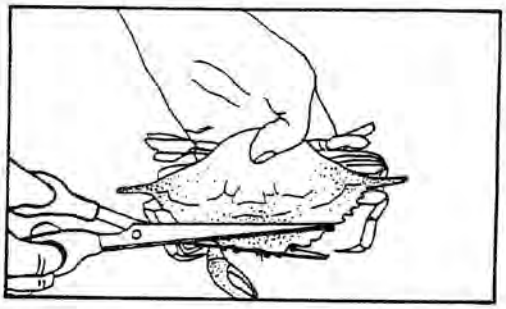
Source: UNC Sea Grant Publication UNC-SG-88-02

G-3

Cleaning soft-shell crabs

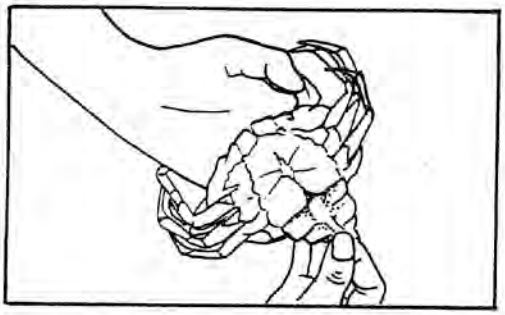
1

Hold crab in one hand and cut across body just behind eyes to remove eyes and mouth.



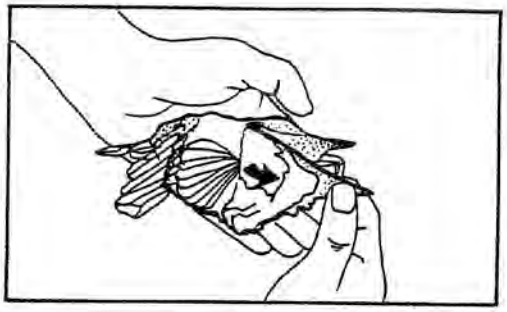
2

Turn crab on its back. Lift and remove apron and vein attached to it.



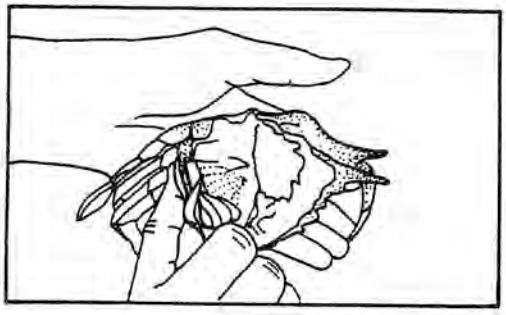
3

Turn crab over and lift one side of top shell.



4

With a small knife, scrape off grayish-feathery gills. Repeat procedure on other side.



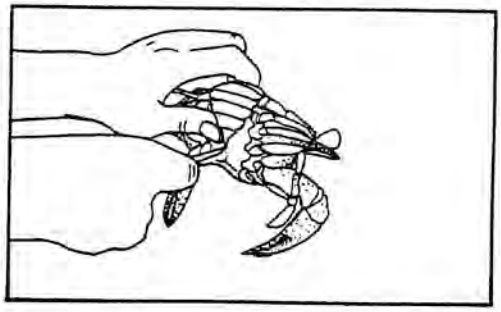
Source: UNC Sea Grant Publication UNC-SG-88-02

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Cleaning hard-shell crabs

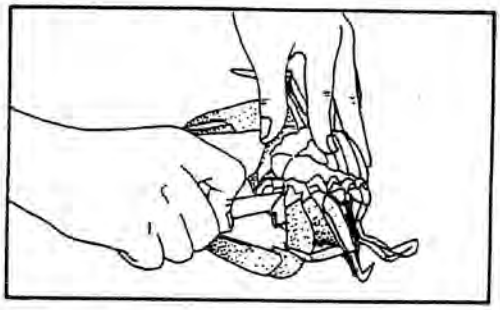
1

Hold crab in one hand. Turn crab over and stab straight down at point of apron with a knife.



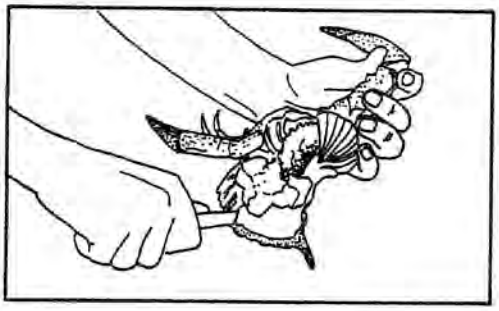
2

Make two cuts from this point to form a V-pattern that will remove mouth.



3

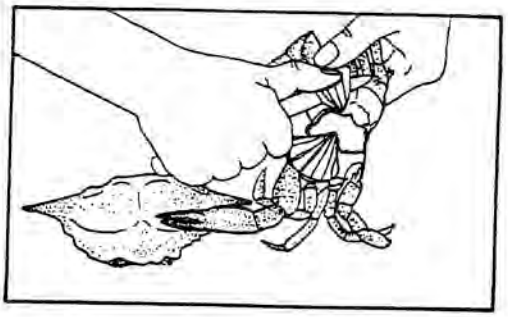
Do not remove knife after making second cut. Firmly press crab shell to cutting surface without breaking back shell. With other hand, grasp crab by legs and claws on the side where you are holding knife, and pull up. This should pull crab body free from back shell.



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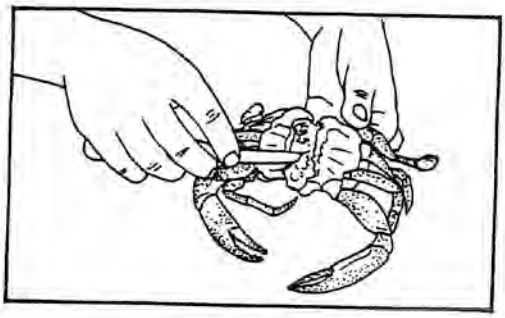
4

Remove gray, feathery gills, which are attached just above legs. Cut and scrape upward to remove gills.



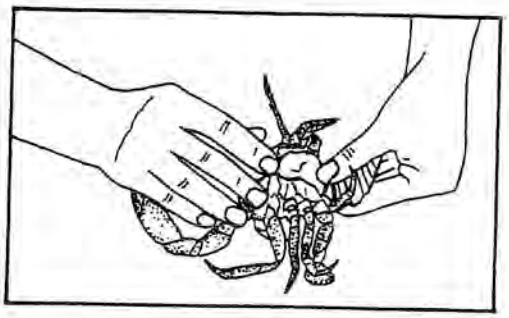
5

Remove all loose material—viscera and eggs—from body cavity.



6

If apron did not come loose with shell, remove it.



Source: UNC Sea Grant Publication UNC-SG-88-02

Shucking oysters

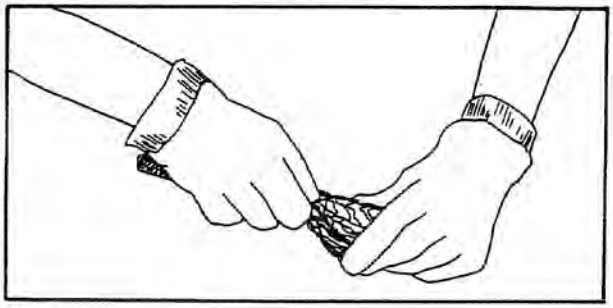
1

Oyster shells are especially sharp; be sure to wear gloves to protect your hands. Chip off a small piece of shell from the thin lip of the oyster until there is a small opening.



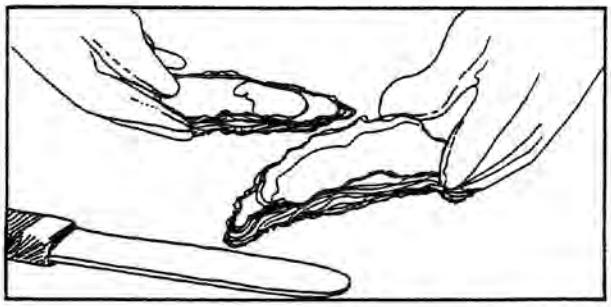
2

Insert knife blade into the opening and cut muscle free from top and bottom shells.



3

Remove oyster meat from the shell.



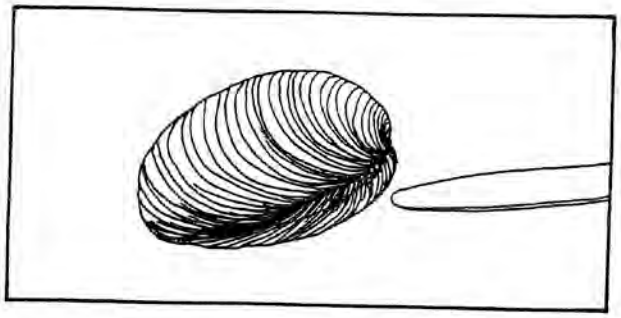
Source: UNC Sea Grant Publication UNC-SG-88-02

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Shucking clams

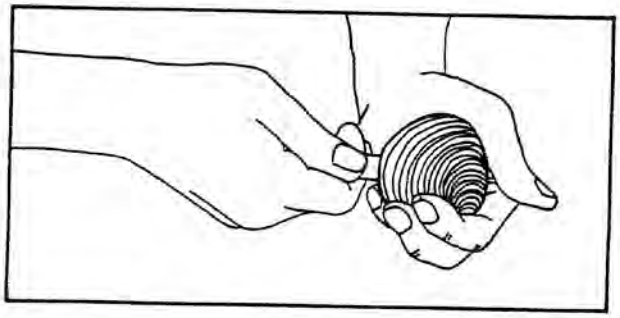
1

In the back of clam near the hinge is a black ligament. Toward the front where ligament ends is a weak spot. Insert your knife at this spot.



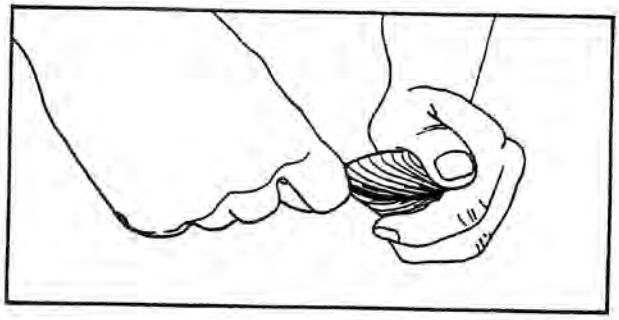
2

Inside are two muscles. Run the knife around the shell to sever both muscles.



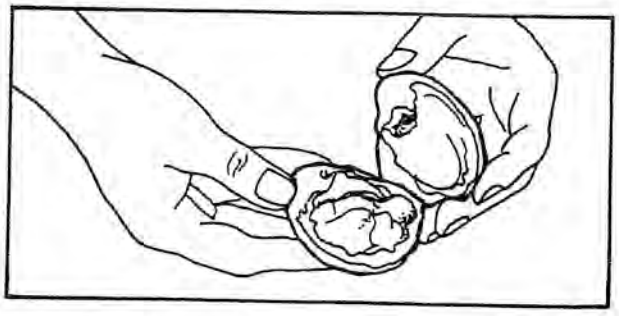
3

Now insert the knife blade into the front of the shell and separate the two shells.



4

Scrape the meat free from the top and bottom shell.



Source: UNC Sea Grant Publication UNC-SG-88-02

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JOHNSON LAKE FISH TISSUE MONITORING STUDY

SAMPLING AND ANALYSIS PLAN

ATTACHMENT D: ODEQ JANUARY 29, 2018, LETTER AND COMMENTS

Oregon
Kate Brown, Governor

Department of Environmental Quality
Northwest Region Portland Office
700 NE Multnomah St., Suite 600
Portland, OR 97232
(503) 229-5263
FAX (503) 229-5471
TTY 711

January 29, 2018

Mr. Rob Webb, Principal Engineer
Dalton Olmsted Fuglevand
1236 Finn Hill Road
Poulsbo, WA 98370

RE: 2016 Johnson Lake Fish Tissue Monitoring Study
ECSI # 2086

Dear Rob:

Thank you for providing the *Revised Johnson Lake Fish Tissue Monitoring Study – Monitoring Report* (dated January 19, 2018) documenting the results of fish tissue monitoring conducted in Johnson Lake, located west of I205 adjacent to the Owens Brockway Glass Container, Inc. facility (5850 NE 92nd Drive in Portland, Oregon). This report was submitted as part of the long-term monitoring of the remedial action implemented by Owens Brockway Glass Container, Inc. as described in the *Remediation Operations and Maintenance Plan-Sediment Remedial Action (OMP)* (December 20, 2012). The revised version addresses comments provided by DEQ (letter dated December 18, 2017) and DEQ approves the revised report.

As noted in our previous letter, because PCBs are still present in fish tissue in Johnson Lake at levels that exceed protective concentrations for human consumption, warning signs must be maintained and fish must be sampled again in 2022. Documentation of the 2022 fish tissue sampling event should include the information provided in the 2018 revised report as well as a summary of the results from the 2017 and 2004 events for comparison. In addition, the 2022 sampling effort and evaluation should incorporate the following:

1. The level of effort detailed in the 2018 report was not sufficient to meet data quality objectives specified in the OMP. The level of sample collection effort at the 2022 sampling event should be sufficient to ensure that the required number of fish specified in the OMP are collected and that fish are collected throughout the lake. Fish in each category must adequately represent that category; e.g., samples representing fish likely to be consumed by humans should be large enough that this exposure would be reasonable. This may require sampling with different gear types at multiple times during 2022 to collect fish when lake conditions support capture. The use of a full range of available gear types (e.g. beach seining and gill nets) is not precluded by the presence of algae and vegetation, but rather is used to ensure success in these conditions. Gear can be modified to exclude non-target species such as sturgeon and turtles.

2. The evaluation of results should consider total PCB, PCB TEQ, and lipid normalized total PCB concentrations in addition to PCB congener 126 concentrations to support evaluation of data trends over time.

3. The revised report did not clearly indicate that composite homogenates of fish tissue were created using equal mass from each individually homogenized fish sample. Lab protocols for sample preparation should specify this procedure will be followed.

4. Composite samples of each fish should be of the same type (species) and size (age) class. Consistent with EPA Guidelines (Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume 1 Fish Sampling and Analysis) the differences between lengths of individuals in a composite sample and the average of the lengths should not exceed 10 percent and at least one replicate should also be analyzed.

I am retiring at the end of this month. The new project manager for this site is Heidi Nelson. Please notify Heidi at (503) 229-6802 of any activities that have potential to expose underlying contaminated sediment in the lake and any necessary maintenance that is warranted. While it is a long way out, a draft sampling plan for the 2022 fish sampling event should be submitted to Heidi no later than 60 days in advance of the planned sampling.

Sincerely,



Jennifer Sutter, Project Manager
NWR Cleanup and Tanks

Cc: Sue Sholl, Owens Illinois
Susan Smith, Owens Illinois
Rebecca Hillwig, Oregon Health Authority
Tim Dean, City of Portland
Heidi Nelson, DEQ NWR Cleanup
Jennifer Peterson, DEQ NWR Cleanup
ECSI File #2086

DEQ-DC1

JOHNSON LAKE FISH TISSUE MONITORING STUDY

SAMPLING AND ANALYSIS PLAN

ATTACHMENT E: QUALITY ASSURANCE PROJECT PLAN

January 30, 2004

By FedEx

Ms. Jennifer L. Sutter
Project Manager
Oregon Department of Environmental Quality
Site Response Section
Northwest Region
2020 SW Fourth Avenue, Suite 400
Portland, OR 97201-4987

RE: Submittal of Revision 2 of the Johnson Lake Investigation Work Plan

Dear Ms. Sutter:

Enclosed please find Revision 2 of the Johnson Lake Investigation Work Plan ("IWP"), approved by the Oregon Department of Environmental Quality ("DEQ") on January 12, 2004. Revision 2 of the IWP includes:

- June 5, 2003 comments from the Oregon DEQ on the April 30, 2003 Johnson Lake IWP; and
- August 20, 2003 and December 1, 2003 comments from the Oregon DEQ on Revision 1 of the Johnson Lake IWP.

Oregon DEQ's comments and ARCADIS' responses are attached to the enclosed Revision 2 of the IWP. I also have included a CD to comply with your request to have the documents in electronic format.

A summary of the significant revisions to the April 30, 2003 IWP are as follows:

- For screening purposes, surface water samples were collected from Johnson Lake in July 2003. No chemical concentrations detected in Johnson Lake surface water exceed any of the ecological criteria or screening values. As such, no chemicals of interest are identified in surface water, and toxicity testing of surface water was removed from the IWP.
- Revision 2 of the IWP includes a comparison of chemical concentrations in soil adjacent to Johnson Lake with screening values developed for sediment. This comparison was conducted for the purpose of identifying chemicals of interest in the IWP, because the

Heidi.Goldstein@ThompsonHine.com Phone 216.566.5559 Fax 216.566.5800 rds 10960509.1

existing sediment data set for Johnson Lake provides limited spatial coverage. The revised list of chemicals of interest following this screening includes polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), two pesticides (DDT and endosulfan) and their metabolites and eight metals (arsenic, cadmium, copper, chromium, lead, nickel, selenium, and zinc).

- At the request of the Oregon DEQ, total petroleum hydrocarbons (TPH) were added as chemicals of interest in sediment.
- At the request of the Oregon DEQ, lead was added as a chemical of interest in biota tissue.
- At the request of Oregon DEQ, PCB congeners will be analyzed in a subset of sediment and biota samples, for comparison purposes only.
- A conceptual site model for human health and ecological exposure pathways was included in Revision 2 of the IWP. The conceptual site model includes written descriptions, identifying human health exposure scenarios and ecological receptors, as well as the basis for their selection. Ecological assessment and measurement endpoints are identified in the conceptual site model.
- A hypothetical recreator (wader) scenario was added to the Revision 2 of the IWP to account for exposure to sediment in a possible future public recreation area.

Owens-Illinois, Inc. will submit a Risk Assessment Work Plan after ARCADIS has completed the proposed sampling and a preliminary review of the analytical results. A land use evaluation will be included as part of the Risk Assessment Work Plan.

The revised implementation schedule is as follows:

Initiation of Field Activities	60 days from DEQ approval of Final IWP, weather permitting
Laboratory Analysis and Data Validation	8 weeks from laboratory receipt of samples
Site Investigation Report and Risk Assessment Work Plan	45 days from completion of data validation

Field activities are contingent upon weather conditions. ARCADIS will provide notification to Oregon DEQ at least two weeks prior to initiation of field activities.

I look forward to receiving a draft of the Fact Sheet for Johnson Lake. If you have any questions or require additional information, please do not hesitate to contact me.

Very truly yours,

Heidi B. Goldstein

Heidi B. Goldstein

cc: Susan L. Smith, Esq.
Timothy R. Barber, Ph.D.
Mark J. Morford, Esq.

Johnson Lake Investigation Work Plan

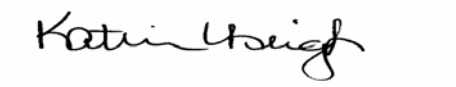
Owens-Brockway Glass Container, Inc.,
Portland, Oregon

Revision: 2

ARCADIS

Infrastructure, buildings, environment, communications

ARCADIS



Katrina Leigh
Environmental Scientist



Phyllis C. Fuchsman
Environmental Scientist



Timothy R. Barber, PhD
Project Manager/Principal Scientist

Johnson Lake Investigation Work Plan

Owens-Brockway Glass Container, Inc.,
Portland, Oregon

Revision: 2

Prepared for:
Owens-Brockway Glass Container, Inc.

Prepared by:
ARCADIS G&M, Inc.
26451 Curtiss Wright Parkway
Suite 100
Cleveland
Ohio 44143
Tel 216 261 7373
Fax 216 261 7225

Our Ref.:
CL000233.0003

Date:
January 29, 2004

Table of Contents

1. Introduction **1**

1.1 Purpose and Objectives 1

2. Site Characteristics **2**

2.1 Site Description 2

2.2 Summary of Previous Investigations 3

3. Johnson Lake Investigation **5**

3.1 Sediment Screening Evaluation 6

3.1.1 Comparison to Screening Values 7

3.1.2 Refinement of Chemicals of Interest 8

3.1.3 Summary of Chemicals of Interest 15

3.1.4 Tetrachloroethene (PCE) and Toluene 16

3.2 Additional Investigation of Johnson Lake 17

3.2.1 Toxicity Testing 18

3.2.2 Sediment Chemistry 19

3.2.3 Biota Tissue Analyses 22

4. Risk Assessment **25**

4.1 Human Health Risk Assessment 25

4.2 Ecological Risk Assessment 28

4.2.1 Ecological Risk Assessment Approach 28

4.2.2 Ecological Conceptual Site Model 30

5. Reporting **34**

6. Summary **34**

7. References **34**

Tables

3-1 Summary of Chemical Concentrations Detected in Sediment, Johnson Lake, Portland, Oregon

3-2 Summary of Chemical Concentrations Detected in On-Site Soil, Johnson Lake, Portland, Oregon

3-3 Maximum Sediment and Soil Concentrations Compared to Screening and Local Baseline Values

Figures

1-1 Site Location Map, Johnson Lake, Portland, Oregon

2-1 Aerial Site Map, Johnson Lake, Portland, Oregon

2-2 Sediment Sampling Locations, Johnson Lake, Portland, Oregon

3-1 Sediment and On-Site Soil Sampling Locations, Johnson Lake, Portland, Oregon

3-2 Proposed Sampling Zones, Johnson Lake, Portland, Oregon

4-1 Current Land Use, Johnson Lake, Portland, Oregon

4-2 Conceptual Site Model for Human Health Exposures

4-3 Conceptual Site Model for Ecological Exposures

Appendices

A Sampling and Analysis Plan (SAP)

B Quality Assurance Project Plan (QAPP)

C Health and Safety Plan (HASP)

D Threatened and Endangered Species Review

E Project Management Plan (PMP)

Attachments

1 Oregon DEQ's Comments on the Johnson Lake Investigation Work Plan, dated June 5, 2003

2 ARCADIS' Response to the Oregon DEQ's June 2003 Comments on the Johnson Lake Investigation Work Plan

3 Oregon DEQ's Comments on Revision 1 of the Johnson Lake Investigation Work Plan, dated August 20, 2003

4 ARCADIS' Response to the Oregon DEQ's August 2003 Comments on Revision 1 of the Johnson Lake Investigation Work Plan

5 Oregon DEQ's Comments on Revision 1 of the Johnson Lake Investigation Work Plan, dated December 1, 2003

6 ARCADIS' Response to the Oregon DEQ's December 2003 Comments on Revision 1 of the Johnson Lake Investigation Work Plan

1. Introduction

At the request of Thompson Hine LLP, on behalf of Owens-Brockway Glass Container, Inc. (Owens-Brockway), ARCADIS prepared this Johnson Lake Investigation Work Plan (IWP) (Revision 2) for the Owens-Brockway facility (site). The site is located at 5850 NE 92nd Drive, Multnomah County, in Portland, Oregon (Figure 1-1). This investigation will be conducted under the Oregon Voluntary Cleanup Program. This Johnson Lake IWP follows applicable Oregon Department of Environmental Quality (DEQ) guidance, Oregon Administrative Rules (OAR 340-122-0010 through –0115), and, as appropriate, the *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (United States Environmental Protection Agency [U.S. EPA] 1988). This work plan addresses comments from the Oregon DEQ, dated June 5, 2003, on the Johnson Lake IWP (ARCADIS 2003a) and Oregon DEQ's comments, dated August 20 and December 1, 2003, on Revision 1 of the Johnson Lake IWP (ARCADIS 2003b). Oregon DEQ's comments and ARCADIS' responses are presented in Attachments 1 through 6.

1.1 Purpose and Objectives

The purpose of this work plan is to develop and articulate methods that will be used to address chemical constituents in the locality of the site (i.e., Johnson Lake). The investigations described in this work plan will provide sufficient information to support site characterization, risk assessment, and the subsequent analysis and selection of remedial alternatives. The specific objectives of the work plan are as follows:

- Summarize site characteristics relevant to the Johnson Lake investigation;
- Identify chemicals of interest in Johnson Lake sediment, based on existing data;
- Describe additional investigations to address chemicals of interest in Johnson Lake;
- Describe human health and ecological risk assessment methodologies to be employed for Johnson Lake; and
- Provide necessary information on data reporting and project schedule.

A separate work plan addressing on-site, upland areas that might act as a source of chemicals of interest to Johnson Lake will be completed based on the results of the Johnson Lake investigation, as appropriate[1].

2. Site Characteristics

Pertinent site characteristics are described below, followed by a summary of previous investigations conducted in and near Johnson Lake.

2.1 Site Description

The Owens-Brockway facility is a glass manufacturing plant occupying approximately 43 acres on the south shore of Johnson Lake. For information on site history and operations, refer to Anderson (1998). Johnson Lake is a six-acre lake which is part of the Whitaker Slough / Columbia Slough drainage system. The Whitaker Slough runs immediately to the north of Johnson Lake, and the Columbia Slough is approximately 500 feet north of the Whitaker Slough (Figure 2-1). The Portland International Airport is located north of the Columbia Slough, between the slough and the Columbia River. Johnson Lake is bounded to the south by the Owens-Brockway facility and to the west and east by other industrial areas (Figure 2-1).

The Columbia and Whitaker Slough system is a naturally occurring system of lakes, wetlands, and channels that once dominated the floodplain of the Columbia River. The slough system runs nearly 18 miles, originating in Fairview Lake and discharging to the Willamette River just upstream of its confluence with the Columbia River. The Columbia Slough consists of two stretches: the lower slough, which extends to the Peninsula Canal, and the upper slough, which extends east to Fairview Lake and consists of long, narrow pond-like segments separated by road crossings and culverts. Whitaker Slough and Johnson Lake are located south of the upper reach of the Columbia Slough. Johnson Lake is fed by storm runoff and groundwater discharge. The lake drains via a short connecting stream (Johnson Lake Slough) into Whitaker Slough.

Stormwater runoff from the Owens-Brockway facility enters Johnson Lake through several outfalls (Outfalls 1 through 6). The City of Portland has one operational and

[1] See Comment #1 in Attachment 2 and Comment #1 in Attachment 4.

one former municipal stormwater outlet to Johnson Lake. The operating outfall (ID #AAP247), located on the western side of Johnson Lake, discharges into Johnson Lake Slough. The former outfall (ID #AAP198) discharged to the western portion of Johnson Lake. This outfall was reportedly removed from service (personal communication, Michael Murray, Multnomah County Drainage District, February 2003). It is unclear whether the former outfall was tied into another existing outfall, rerouted, or continues to discharge to Johnson Lake. Stormwater runoff originating from the industrial areas east of the facility also discharges into the eastern portion of Johnson Lake via Outfall #AAP235. Myers Container Corporation, located at 10103 NE Marx Street, Portland, Oregon, is an identified discharger to Johnson Lake through this eastern outfall. This outfall is located on property owned by the City of Portland.

2.2 Summary of Previous Investigations

Previous sampling investigations are described below.

- In 1994, Parametrix, Inc. collected sediment from target and random locations along Columbia and Whitaker Sloughs, as well as downstream areas (i.e., the Peninsula Drainage Canal and Buffalo Slough). In addition, three sediment locations in Johnson Lake were sampled. All samples were analyzed for polychlorinated biphenyls (PCBs), semi-volatile organic compounds (SVOCs), volatile organic compounds (VOCs), pesticides, and metals. PCBs were detected below reporting limits in two sediment samples from Johnson Lake and were estimated as "J" values. PCBs were identified by Oregon DEQ as a potential chemical of concern in Johnson Lake. Other organic and inorganic chemicals were also detected (Parametrix, Inc. 1995).

- In September 1998, Kennedy/Jenks Consultants, on behalf of Owens-Brockway, collected eight surface sediment samples and one subsurface sediment sample from Johnson Lake, adjacent to the drainage way associated with Outfalls 1 and 2 (Kennedy/Jenks Consultants 1998). PCBs and total organic carbon (TOC) were analyzed. PCB Aroclor 1260 was detected in three samples.

- In September 1998, TRC, on behalf of Myers Container Corporation, collected and analyzed one subsurface sediment sample from the West Pond, situated near the western perimeter of the Myers property (TRC 1999a). The sample was collected to determine the presence of PCBs, cadmium, chromium, and lead in the pond sediment. Only chromium was detected. An

additional, deeper sediment sample was also collected but was never analyzed.

- In 1999, TRC, on behalf of Myers Container Corporation, collected two sediment samples from the West Pond (TRC 1999b). The samples were analyzed for PCBs. Aroclor 1260 was detected in both samples.

- In October/November 2000, Kennedy/Jenks Consultants, on behalf of Owens-Brockway, collected seven surface soil samples in the drainage way associated with Outfalls 1 and 2 (Kennedy/Jenks Consultants 2000). Additionally, seven subsurface soil samples were collected from two locations, at Outfalls 1 and 2. Subsurface sampling depths ranged from 1.0 to 5.5 feet below ground surface. All samples were analyzed for PCBs. Aroclor 1260 was detected in five of seven surface soil samples and two of seven subsurface soil samples.

- In November 2000, Kennedy/Jenks Consultants, on behalf of Owens Brockway, collected three sediment samples from the catch basin on Owens Brockway property (Kennedy/Jenks Consultants 2000). Samples were analyzed for PCBs; all were non-detect.

- In 2001, TRC, on behalf of Myers Container Corporation, collected twenty-nine surface soil samples, one sediment sample, and one sludge sample from Myers property and adjacent to Johnson Lake (TRC 2002). Samples were analyzed for PCBs, polycyclic aromatic hydrocarbons (PAHs), phthalates, chromium, lead, and zinc. Aroclor 1254 and1260 were detected in samples collected from around the eastern pond and the West Pond on Myers property. PAHs, phthalates and metals were also detected.

- In February 2003, ARCADIS, on behalf of Owens-Brockway, collected nine sediment samples from the Columbia Slough, Whitaker Slough, Prison Pond (a part of the Whitaker Slough located upstream of Johnson Lake), and Johnson Lake (Figure 2-2[2]). Samples were analyzed for PCBs, SVOCs, VOCs, pesticides, herbicides, and metals. PCBs and other constituents were detected at locations upgradient, cross-gradient, and downgradient from

[2] See Comment #2 in Attachment 4.

Johnson Lake, as well as within Johnson Lake near an outfall from the Meyers facility on the eastern side of the lake[3]. Aroclor 1242 was only detected in sample SD-07 collected in the Whitaker Slough north of Johnson Lake. Sample SD-07 also contained the highest total PCB concentration (6,210 µg/kg) reported for any location in this area. (see Appendix A in ARCADIS 2003a).

- In February 2003, ARCADIS, on behalf of Owens-Brockway, collected ten on-site surface soil samples from each of the on-site outfalls discharging to Johnson Lake and from the drainage way associated with Outfalls 1 and 2 (ARCADIS 2003c). Samples were analyzed for PCBs, VOCs, SVOCs, pesticides, herbicides, and metals. Various organic and inorganic constituents were detected.

- In July 2003, ARCADIS, on behalf of Owens-Brockway, collected eight surface water samples from Johnson Lake [4]. Sampling was conducted according to the methods described in the Johnson Lake IWP, dated April 10, 2003 (ARCADIS 2003a). Samples were analyzed for PCBs, VOCs, SVOCs, pesticides, and dissolved metals. Dissolved metals, tetrachloroethene (PCE), and toluene were detected in Johnson Lake surface water.

3. Johnson Lake Investigation

The nature of additional investigations needed in Johnson Lake is determined based on a screening-level evaluation of existing data (Section 3.1). No chemicals of interest were identified in Johnson Lake surface water, following a preliminary screening evaluation (see Attachment 4). Chemical exposures via surface water are considered negligible relative to other exposure pathways. Therefore, chemicals of interest in Johnson Lake are based on the screening evaluation of sediment and surface soil adjacent to Johnson Lake. Based on this screening-level assessment, targeted sampling and analyses are proposed to support human health and ecological risk assessments of the lake (Section 3.2).

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[3] See Comment #2 in Attachment 4.

[4] See Comment #10 in Attachment 4.

3.1 Sediment Screening Evaluation

Chemicals of interest for further evaluation in Johnson Lake are identified based on a screening-level evaluation of existing data. This screening evaluation considers sediment samples collected from Johnson Lake in 1994, 1998, and 2003. Because only a limited number of samples from Johnson Lake have been analyzed for constituents other than PCBs, the screening evaluation also considers analytical results for surface soil samples collected from the Owens-Brockway property near Johnson Lake in 2000 and 2003[5]. Soil data do not represent chemical concentrations to which sediment-dwelling organisms are exposed, but they provide a conservative indication of concentrations that might be detected in sediment upon further sampling. In actuality, chemical concentrations are likely to be lower in sediment than in soil (for any constituents originating from the site), because the soil sampling effort was designed to target potential source areas, and chemical concentrations typically decline with distance from a source. Sediment and surface soil sample locations are shown in Figure 3-1. Chemical concentrations detected in Johnson Lake sediment and surface soil are summarized in Tables 3-1 and 3-2.

The initial step in the screening evaluation consists of comparing the maximum detected concentration of each chemical to screening values identified by the Oregon DEQ (Section 3.1.1). While this process provides a simple means of eliminating some chemicals from further evaluation, it does not provide enough information to identify additional data collection needs. Therefore, the chemicals of interest are refined based on U.S. EPA guidance and other information from the scientific literature (Section 3.1.2). A summary of the chemicals of interest identified through this process is provided in Section 3.1.3.

The screening process is based on ecological considerations, because sediment screening values for the protection of human health are not currently available. Chemicals of interest identified for further evaluation are assessed for their bioaccumulation potential, and those chemicals having the potential to biomagnify through the food web are identified for the assessment of hypothetical risks to human consumers of fish from Johnson Lake (Section 3.1.3).

[5] See Comments #2 and #3 in Attachment 2.

3.1.1 Comparison to Screening Values

At the request of Oregon DEQ, the following screening values are used for the first step in the screening evaluation:

- Level II screening level values for freshwater and marine sediment, taken from the Oregon DEQ's (1998a) ecological risk assessment guidance; and
- Columbia Slough sediment contaminant baseline maxima, generated through graphical analysis methods by Oregon DEQ (Oregon DEQ 2002).

The Level II screening values include up to three values per chemical, termed "freshwater," "marine," and "bioaccumulation" values. The freshwater and marine values are intended to be protective of aquatic life, whereas the bioaccumulation values are intended to protect wildlife that feed on aquatic life. For initial screening purposes, the lower of the applicable aquatic life and bioaccumulation values are used. Freshwater values are used to represent applicable aquatic life values if available; marine values are substituted only if freshwater values are not available. It is important to note that the aquatic life values recommended by Oregon DEQ are based on associations between chemical concentrations and various measures of adverse effects, in field-collected sediments containing complex chemical mixtures. They do not represent cause-effect, concentration-response relationships for individual chemicals or site-specific chemical mixtures. Rather, each aquatic life value serves as an indicator of an overall level of contamination (by chemical mixtures) that is intended to be generally predictive of a lack of toxicity. The bioaccumulation values are derived by estimating the extent to which each chemical may accumulate in prey tissue and the amount of chemical that can be safely ingested over time, together with various wildlife exposure parameters such as ingestion rates and body weights. The chemical-specific and species-specific information used in deriving the bioaccumulation values is not documented[6].

The Columbia Slough baseline values are intended to represent naturally occurring concentrations of metals and/or ubiquitous concentrations of anthropogenic metals and organic chemicals in sediment[7]. Local baseline values were derived by the Oregon

[6] See Comment #4 in Attachment 4.

[7] See Comment #5 in Attachment 4.

DEQ using two methods: normal probability plots and linear ordinal response plots. The two methods generated similar values (Oregon DEQ 2002). For this evaluation, the values derived using normal probability plots are used for initial screening purposes, because the methodology is considered more appropriate (i.e., because chemical concentrations are continuous rather than ordinal). Local baseline values are not available for all chemicals detected in Johnson Lake.

The comparison of maximum detected chemical concentrations to screening values is shown in Table 3-3. Chemicals are eliminated from further consideration if they are below the respective ecological screening value or baseline value. If the maximum concentration exceeds baseline levels and exceeds an ecological screening value, then the chemical is evaluated further (Section 3.1.2) or is identified as a chemical of interest (Section 3.1.3). Additionally, TPH is included as a chemical of interest at Oregon DEQ's request, although it is not included in the chemical-specific screening evaluation[8].

3.1.2 Refinement of Chemicals of Interest

As described above, the initial screening comparisons provided in Table 3-3 do not provide enough information to identify additional data collection needs[9]. Therefore, additional information is considered to identify which chemicals merit further investigation in Johnson Lake. This information includes toxicity data from U.S. EPA guidance and the scientific literature, additional characterization of naturally occurring concentrations of metals, and identification of common laboratory contaminants.

For organic chemicals, the equilibrium partitioning approach (Di Toro, Zarba et al. 1991; Fuchsman 2003; U.S. EPA 2000a,b,c,d) provides an important line of evidence. This approach to developing sediment quality benchmarks for benthic organisms is based on cause-effect, concentration-response data and accounts for site-specific factors that affect chemical bioavailability. The equilibrium partitioning approach has been extensively validated in field and laboratory studies (U.S. EPA 2000a,b,c,d). As described by Di Toro, Zarba et al. (1991), this approach uses the mass fraction of organic carbon in sediment (f_{oc}) and the chemical-specific partition coefficient between water and organic carbon (K_{oc}) to calculate sediment quality benchmarks as follows:

[8] See Comment #6 in Attachment 2 and Comments #3 and #11 in Attachment 4.

[9] See Comment #6 in Attachment 2.

$$Sediment\ quality\ benchmark = Water\ quality\ benchmark \times K_{oc} \times f_{oc}$$

For chemicals having log K_{ow} values below 3.5, however, this equation incorporates a mathematical error, resulting in screening values that are more conservative than intended. Fuchsman (2003) developed a corrected method for calculating sediment screening values for these compounds, as follows:

$$Sediment\ quality\ benchmark = Water\ quality\ benchmark \times \left[\left(K_{oc} \times f_{oc} \right) + \left(\frac{1 - f_{solids}}{f_{solids}} \right) \right]$$

where f_{solids} represents the mass fraction of sediment present as solids. For this evaluation, K_{oc} values are estimated from log K_{ow} values according to the regression equation determined by Di Toro, Zarba et al. (1991):

$$\log K_{oc} = 0.00028 + 0.983 \log K_{ow}$$

Log K_{ow} values are obtained from the U.S. EPA's Log Octanol-Water Partition Coefficient Program (KOWWIN) from EPI Suite v3.10. Organic carbon and solids content are identified from sample-specific data. Water quality benchmarks are identified on a chemical-specific basis (see below).

Several chemicals are eliminated from further evaluation in Johnson Lake based on a refined screening analysis. The rationale for eliminating chemicals is discussed below. Chemicals identified for further investigation are summarized in Section 3.1.3.

- *Acetone:* Acetone was detected in two of five sediment samples, with a maximum reported concentration in sediment (750 µg/kg) that exceeds the Oregon DEQ's recommended screening value (290 µg/kg). The maximum acetone concentration reported in surface soil is below the screening value. Acetone is a common laboratory contaminant and may not actually occur in Johnson Lake. Further, despite exceeding the screening value, the reported concentration is extremely unlikely to cause adverse ecological effects. The Oregon DEQ's screening value is reportedly based on the protection of wildlife from bioaccumulation-related effects, but acetone is strongly hydrophilic and exhibits virtually no bioaccumulation potential. Based on an extensive review of the aquatic toxicity and bioavailability of acetone (Fuchsman 2003; Staples 2000), an appropriate sample-specific sediment quality benchmark for acetone is 20,000 µg/kg (based on a water quality

benchmark of 10,600 µg/L, a log K_{ow} of –0.24, an organic carbon content of 1.9 percent and a solids content of 34.8 percent), far higher than the maximum reported concentration. Therefore, acetone does not merit further evaluation in Johnson Lake sediment.

- *Benzoic Acid:* Benzoic acid was detected in two of five sediment samples, with a maximum sediment concentration (120 µg/kg) that exceeds the Oregon DEQ's recommended screening value (65 µg/kg). The maximum detected concentration is similar to the local baseline concentration (110 µg/kg) identified by the Oregon DEQ. Benzoic acid was not detected in surface soil. Benzoic acid is used in various consumer products (e.g., as a food additive) and industrial processes and is also formed naturally during the degradation of woody plant material. In water, benzoic acid partially dissociates, forming benzoate; under normal pH conditions, the aquatic toxicity of benzoic acid and sodium benzoate is similar (WHO 2000). Based on tests with a variety of fish, amphibian, and invertebrate species, acutely toxic concentrations of benzoic acid and/or sodium benzoate exceed 100,000 µg/L (WHO 2000). Even if the benzoic acid concentrations observed in Johnson Lake sediment were 100 percent bioavailable, the concentrations would be several orders of magnitude below this level. Therefore, benzoic acid does not merit further evaluation in Johnson Lake sediment.

- *Benzyl Alcohol:* Benzyl alcohol was detected in sediment and surface soil at concentrations (up to 590 µg/kg) that exceed the Oregon DEQ's recommended screening value (52-57 µg/kg). The Oregon DEQ did not identify a local background concentration for benzyl alcohol. Similar to benzoic acid, benzyl alcohol is used in foods and various other consumer products and industrial processes, as well as occurring naturally due to degradation of plant material. Benzyl alcohol is metabolized to benzoic acid in mammals (WHO 2000) and potentially in other types of animals. Although it has been studied less extensively, the aquatic toxicity of benzyl alcohol appears to be similar to that of benzoic acid and sodium benzoate, based on data listed in the U.S. EPA's online AQUIRE database. Therefore, the relatively low concentrations of benzyl alcohol found in and near Johnson Lake are very unlikely to pose a risk of toxicity, and benzyl alcohol does not merit further evaluation in sediment.

- *4-Methylphenol:* Methylphenols are another product of the degradation of woody plant material and are also used industrially. Concentrations of 4-methylphenol in Johnson Lake sediment range from 25 to 1,600 µg/kg, exceeding the Oregon DEQ's recommended screening value of 100 µg/kg. In

on-site soil, 4-methylphenol was detected in only 2 of 10 samples, with a maximum concentration of 390 µg/kg. To identify a sediment quality value for 4-methylphenol based on cause-effect, concentration-response data, the U.S. EPA's (2000c) equilibrium partitioning approach is applied, as modified by Fuchsman (2003) for less hydrophobic chemicals. It is well established that the aquatic toxicity of 4-methylphenol is less than that of 2,4-dimethylphenol (e.g., Feng, Shuokui et al. 1996; Karabunarliev, Mekeyan et al. 1996; Schultz, Holcombe et al. 1986); therefore, the U.S. EPA's (1993a) chronic water quality criterion for 2,4-dimethylphenol (530 µg/L) is used as a surrogate for 4-methylphenol, which has been less extensively studied. Together with a log K_{ow} of 2.06 for 4-methylphenol and sample-specific organic carbon and solids contents of 1.9 percent and 34.8 percent, respectively, this results in a sediment quality benchmark of 2,100 µg/kg. Because the maximum detected 4-methylphenol concentration in Johnson Lake is below this value, 4-methylphenol will not be evaluated further in sediment.

- *2-Methylphenol:* 2-Methylphenol was not detected in Johnson Lake sediment but was detected in 1 of 10 surface soil samples at a concentration of 10 µg/kg. Although the Oregon DEQ's recommended screening value for 2-methylphenol (8 µg/kg) is an order of magnitude lower than the 4-methylphenol screening value, the two methylphenol isomers are actually expected to exhibit very similar bioavailability and toxicity (Feng, Shuokui et al. 1996; Karabunarliev, Mekeyan et al. 1996; Schultz, Holcombe et al. 1986). Thus, 2-methylphenol would be very unlikely to cause toxicity in sediment at concentrations similar to that found in soil, and 2-methylphenol will not be evaluated further in Johnson Lake.

- *Phenol:* Similar to the methylphenols discussed above, phenol is used in industrial processes and can also occur naturally in sediment due to degradation of woody plant material. Phenol was detected in three of five sediment samples, with a maximum concentration (130 µg/kg) that exceeds the Oregon DEQ's recommended screening value (48 µg/kg). The maximum phenol concentration detected in surface soil (32 µ/kg) does not exceed the screening value. To identify a sediment quality value based on cause-effect, concentration-response data for phenol, the U.S. EPA's (2000c) equilibrium partitioning approach is applied, as modified by Fuchsman (2003) for less hydrophobic chemicals. Based on a water quality benchmark of 110 µg/L (U.S. EPA 1993b) and a log K_{ow} of 1.51 for phenol, together with a sample-specific organic carbon content of 1.9 percent and a solids content of 34.8 percent, this approach results in a sediment quality benchmark of 270 µg/kg

(Fuchsman 2003). Because the maximum detected phenol concentration in Johnson Lake is below this value, phenol will not be evaluated further in sediment.

- *Butylbenzyl phthalate:* The maximum detected concentration of butylbenzyl phthalate in surface soil (340 µg/kg) exceeds the Oregon DEQ's recommended screening value (63 µg/kg) and local baseline concentration (151 µg/kg), although no detected concentrations in sediment exceeded the screening or baseline values[10]. Call, Markee et al. (2001) evaluated the toxicity of butylbenzyl phthalate to sediment-dwelling organisms. Based on these authors' results, the equilibrium partitioning approach predicts a 10-day LC50 for the amphipod *Hyalella azteca* of 150,000 µg/kg for sediment containing 1 percent organic carbon, with even higher subchronic LC50s predicted for the midge *Chironomus tentans* and the worm *Lumbriculus variegatus.* In spiked sediment toxicity tests with other phthalate compounds (Call, Cox et al. 2001), the observed test results agreed well with the predictions of the equilibrium partitioning approach. Thus, butylbenzyl phthalate would not be expected to cause toxicity in sediment at the relatively low concentrations detected in soil, and butylbenzyl phthalate will not be evaluated further in Johnson Lake.

- *Chlordanes:* The maximum concentrations of alpha- and gamma-chlordane in surface soil (47 and 57 µg/kg, respectively) exceed the Oregon DEQ's recommended sediment screening values (10 µg/kg for alpha-chlordane and 4.5 µg/kg for "chlordane"). These screening values are intended to protect aquatic life; the detected chlordane concentrations do not exceed the Oregon DEQ's bioaccumulation screening value (420 µg/kg) for protection of wildlife. Detected concentrations of chlordanes in sediment are below all of the recommended screening values. Using the equilibrium partitioning approach, a sediment quality benchmark for the protection of aquatic life is calculated as 2,400 µg/kg for sediment containing 1 percent organic carbon. This value is based on the U.S. EPA's (1980) Final Chronic Value of 0.17 µg/L[11] and a log K_{ow} of 6.26. The chlordane concentrations detected in soil are well below this

[10] See Comment #7 in Attachment 4.

[11] The Final Chronic Value for chlordane is used because it is based on the protection of aquatic life, whereas the U.S. EPA's Ambient Water Quality Criterion for chlordane is based on the protection of wildlife.

sediment quality benchmark, and therefore chlordanes will not be evaluated further in Johnson Lake.

- *Endrin ketone:* Endrin ketone (a breakdown product of the pesticide endrin) was detected in two of five sediment samples, with a maximum detected concentration of 36 µg/kg dry weight. Endrin ketone was not detected in surface soil. A screening value is not available for endrin ketone, and the Oregon DEQ also did not identify a local background concentration for endrin ketone. Therefore, for screening purposes, endrin is used as a surrogate. The maximum reported concentration of endrin ketone exceeds the Oregon DEQ's recommended screening value for endrin of 3 µg/kg. However, the U.S. EPA has developed a sediment quality guideline for endrin based on cause-effect, concentration-response data. Specifically, the guideline of 5.4 micrograms per gram organic carbon (µg/gOC) is based on extensive investigations of cause-effect, concentration-response data and reflects site-specific conditions (i.e., sediment organic carbon) that affect chemical bioavailability (U.S. EPA 2000d). Based on a sample-specific organic carbon concentration of 5 percent, the maximum detected endrin ketone concentration is equivalent to 0.72 µg/gOC. Because this concentration is well below the U.S. EPA guideline for endrin, endrin ketone will not be evaluated further in Johnson Lake sediment.

- *Barium:* The maximum concentrations of barium reported for Johnson Lake sediment (83 mg/kg) and surface soil (145 mg/kg) exceed the screening value of 48 mg/kg. The Oregon DEQ did not identify a local background concentration for barium. However, background concentrations of barium in sediment at sites throughout the United States are generally higher than the levels observed in and adjacent to Johnson Lake (Mason and Dragun 1996). Benthic organisms have evolved to tolerate naturally occurring concentrations of metals in sediment. Thus, it is implausible that the low levels of barium found in Johnson Lake pose a risk of toxicity. Furthermore, barium forms various insoluble salts and is expected to show limited bioavailability in sediment. Barium also is not highly toxic to aquatic organisms (Warrington 2000). Adverse effects due to barium are thus considered very unlikely at the reported concentrations.

- *Cyanide:* Cyanide was detected in one of three sediment samples at a concentration of 0.56 mg/kg. Cyanide was not detected in surface soil. A screening value is not available for cyanide in sediment. The detected concentration is similar to the local background concentration of 0.5 mg/kg.

On this basis, cyanide does not merit further evaluation in Johnson Lake sediment.

- *Molybdenum:* Molybdenum was detected in one of three sediment samples at a concentration of 2 mg/kg. Molybdenum was not detected in surface soil. The Oregon DEQ identified a local background concentration for molybdenum of <1 mg/kg. However, the molybdenum concentration observed in Johnson Lake is within the range of background concentrations found at other sites in the western United States (Mason and Dragun 1996). A screening value is not available for molybdenum in sediment. Because the observed concentration is similar to naturally occurring background levels, molybdenum does not merit further evaluation in Johnson Lake sediment.

- *Thallium:* Thallium was detected in 3 of 10 surface soil samples, with a maximum concentration (7 mg/kg) that exceeds the Oregon DEQ's recommended screening value (0.7 mg/kg) and local baseline value (0.58 mg/kg)[12]. Thallium concentrations in detected sediment do not exceed the screening or baseline values. Borgmann, Cheam et al. (1998) investigated toxicity and bioaccumulation in amphipods (*Hyalella azteca*) exposed to thallium in Great Lakes sediment. The amphipod tissue concentration associated with a 25 percent reduction in survival was calculated as approximately 60 mg/kg, corresponding to a sediment LC25 of approximately 40 to 120 mg/kg. Thallium concentrations in the amphipods were approximately equal to the concentrations in sediment, indicating a low potential for biomagnification through the food web. The thallium concentrations reported in soil are also within the range of background concentrations reported for sediment, although only limited data area available (Mason and Dragun 1996). Because the reported thallium concentrations are below a level expected to cause toxicity and are similar to naturally occurring levels, thallium will not be evaluated further in Johnson Lake.

- *Vanadium:* The maximum concentrations of vanadium reported for Johnson Lake sediment (82 mg/kg) and surface soil (78 mg/kg) exceed the screening value of 57 mg/kg. The Oregon DEQ did not identify a local background concentration for vanadium. The vanadium concentrations observed in Johnson Lake sediment are within the range of naturally occurring background

[12] See Comment #7 in Attachment 4.

concentrations at sites in the western United States (Mason and Dragun 1996). On this basis, it is very unlikely that the low concentrations of vanadium found in Johnson Lake sediment pose a risk of toxicity. Vanadium thus does not merit further evaluation in sediment.

3.1.3 Summary of Chemicals of Interest

Chemicals of interest in Johnson Lake sediment include[13]:

- PCBs;
- PAHs;
- Two groups of pesticides (DDT and its metabolites, DDE and DDD; and endosulfans, including endosulfan I and II and endosulfan sulfate);
- Eight metals (arsenic, cadmium, chromium, copper, lead, nickel, selenium, and zinc); and
- Total petroleum hydrocarbons (TPH), at the request of Oregon DEQ[14].

Chemicals of interest in Johnson Lake biota include:

- PCBs;
- Two groups of pesticides (DDT and its metabolites, DDE and DDD; and endosulfans, including endosulfan I and II and endosulfan sulfate);
- Selenium; and
- Lead, at the request of Oregon DEQ[15].

[13] See Comment #6 in Attachment 2.

[14] See Comment #6 in Attachment 2 and Comments #3 and #11 in Attachment 4.

[15] See Comment #13 in Attachment 4.

All chemicals of interest will be investigated further with respect to potential toxicity to aquatic life. Additionally, PCBs, pesticides, and selenium have the potential to biomagnify through the food web, potentially affecting wildlife and/or human consumers of aquatic organisms. Thus, these chemicals will also be investigated to determine the site-specific extent of bioaccumulation in aquatic organisms.

The remaining chemicals of interest may occur at detectable concentrations in biota, but they do not biomagnify through the food web[16]. For instance, fish rapidly metabolize PAHs, and the potential for adverse effects on fish consumers due to PAHs is very low (Fuchsman, Leigh et al. 2001). Further, PAHs generally have not been detected in fish collected from the Columbia Slough system[17]. Similarly, with the exception of selenium, the metals identified as chemicals of interest for Johnson Lake have a relatively low bioaccumulation potential. However, at the request of Oregon DEQ, both selenium and lead will be evaluated to assess site-specific bioaccumulation. For the remaining metals and for PAHs, the evaluation of risks to aquatic life is expected to be protective of consumers of aquatic organisms for these chemicals.

3.1.4 Tetrachloroethene (PCE) and Toluene

In addition to the identification of chemicals of interest, the sediment data for Johnson Lake also provide information on potential ongoing sources of chemicals to the lake. Specifically, the detection of PCE and toluene is of note, because these compounds are volatile and are not persistent in sediment. Although the maximum detected concentrations of PCE and toluene in sediment do not exceed the screening or baseline values for each chemical, the detection of PCE and toluene in Johnson Lake may indicate one or more off-site sources of contaminated groundwater impacting Johnson Lake (see Attachment 4). In addition to their detection in sediment, PCE and toluene were the only organic chemicals detected in recent analyses of surface water collected from Johnson Lake[18]. Concentrations of PCE in surface water were similar throughout the lake. Given that Owens-Brockway did not use PCE in its operations, it is likely that the PCE in Johnson Lake is related to off-site sources. Similarly, the spatial distribution of toluene concentrations suggests that toluene is entering the lake from the

[16] See Comment #8 in Attachment 2 and Comment #9 in Attachment 4.

[17] See Comment #9 in Attachment 4.

[18] See Comment #8 in Attachment 4.

north, rather than from the Owens-Brockway site (see Attachment 4). Although toluene is a component of petroleum hydrocarbons, other characteristic petroleum hydrocarbon chemicals (e.g., benzene, ethylbenzene, xylenes) are not present at elevated concentrations in Johnson Lake sediment or surface water. This further suggests an off-site source related specifically to toluene use rather than petroleum hydrocarbons.

3.2 Additional Investigation of Johnson Lake

The sediment screening evaluation for Johnson Lake identified chemicals of interest for further evaluation with regard to aquatic life exposures and exposures via consumption of aquatic life. The study design for investigation of these chemicals of interest is based on the human health and ecological conceptual site model described in Section 4. Potential risks to benthic organisms will be evaluated primarily based on toxicity testing (Section 3.2.1). Concurrent analyses of sediment chemistry will also be conducted to facilitate the interpretation of toxicity test results and to support the human health risk assessment (Section 3.2.2). Potential risks to consumers of aquatic life, including wildlife and humans, will be investigated through analyses of chemicals of interest in biota tissue (Section 3.2.3). Following a preliminary screening evaluation and at the recommendation of the Oregon DEQ, chemicals of interest are not identified in surface water, and analyses of surface water are not included as part of the proposed investigation[19].

Samples will be collected from eight zones designated in Johnson Lake, as shown in Figure 3-3. The sampling and analytical study design is described below. Detailed sampling and analytical methods are provided in the Sampling and Analysis Plan (SAP) (Appendix A) and the Quality Assurance Project Plan (QAPP) (Appendix B)[20]. Health and safety information is provided in Appendix C[21].

The investigation described in this work plan will provide sufficient information to support site characterization, human health and ecological risk assessments, and the

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[19] See Comment #12 in Attachment 2.

[20] See Comments #13, #16, and #20 in Attachment 2.

[21] See Comment #20 in Attachment 2.

calculation of site-specific remediation goals. Additional sampling may be required in the future to delineate hot spots, if necessary.

3.2.1 Toxicity Testing

Composite sediment samples will be collected for toxicity testing. Multiple grab samples of surface sediment (top 0 to 6 inches) will be collected throughout each of the eight sampling zones and will be composited for toxicity testing and chemistry analyses[22]. Kennedy/Jenks Consultants (2000) reported that the upper 6 inches of Johnson Lake sediment consisted of loose sand or silt, which was underlain by dense clay[23]. Thus, surface sediment is expected to represent the biologically active zone of Johnson Lake sediment.

Toxicity tests will be conducted using the following standard methods[24]:

- Ten-day, whole-sediment toxicity tests with the amphipod *Hyalella azteca*, measuring survival (U.S. EPA 2000e); and
- Ten-day, whole-sediment toxicity tests with the midge *Chironomus tentans*, measuring survival and growth (U.S. EPA 2000e).

Toxicity test results will be interpreted using criteria established by the U.S. Army Corps of Engineers (U.S. ACOE), U.S. EPA et al. (1998)[25]. Test organism survival or growth will be considered significantly impaired if both a statistically significant difference and a minimum significant difference are observed, based on comparisons to control organisms. Minimum significant differences are defined as a decrease relative to the control of 15 percent for amphipod mortality, 20 percent for midge mortality, and 40 percent for midge growth. The use of a minimum significant difference is implemented because low variance among replicates (e.g., due to 100 percent survival of control organisms) can sometimes result in statistically significant differences that

[22] See Comment #9 in Attachment 2.

[23] See Comment #13 in Attachment 2.

[24] See Comment #10 in Attachment 2.

[25] See Comment #11 in Attachment 2.

are not ecologically significant (Phillips, Hunt et al. 2001; Thursby, Heltshe et al. 1997).

Toxicity tests have the advantage of integrating the effects (if any) of all chemicals occurring in the samples. However, toxicity test organisms can also be affected by factors unrelated to chemical contamination, such as the nutritional content of the sample. Therefore, toxicity test results will be compared to sediment chemistry data and physical characteristics (see below) to help determine whether any observed impairment of the test organisms represents chemical toxicity. In the event that significantly reduced survival or growth are observed but do not appear to be due to measured chemical or physical characteristics of the sediment, it may be necessary to conduct a comparative evaluation using a reference sediment. In that event, an appropriate reference area will be selected in consultation with the Oregon DEQ.

3.2.2 Sediment Chemistry

Sediment for chemical analyses will be collected concurrently with the collection of sediment for toxicity testing[26]. Each of the sediment analyses will provide information on the site-specific bioavailability and potential toxicity of chemicals of interest or will facilitate the interpretation of toxicity test results. In addition, sediment analyses will provide information relevant to the exposure assessment for hypothetical recreators who may wade in Johnson Lake. The rationale for each analysis is provided below.

- *PCB homologues:* Homologue analysis (U.S. EPA Method 680) measures PCBs based on their level of chlorination. Results are reported as total monochlorobiphenyls through total decachlorobiphenyls. This analysis provides site-specific information on the composition of PCB mixtures, which can be used together with TOC content to estimate PCB bioavailability in sediment. Homologue methods provide definitive identification of PCBs, better quantification, and lower detection limits than Aroclor-based methods (Valoppi, Petreas et al. 1999).

- *PCB congeners:* U.S. EPA Method 8082 measures individual PCB congeners, of which there are 209 possible. Target analytes will include the following twelve congeners: PCBs 77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169,

[26] See Comment #15 in Attachment 2.

and 189. At the request of Oregon DEQ, PCB congeners will be analyzed in a subset of sediment samples for comparison purposes[27].

- *Alkylated PAHs[28]:* The U.S. EPA (2000a) has developed sediment quality guidelines for mixtures of approximately forty PAHs, including substituted and unsubstituted PAH compounds. Measured concentrations of alkylated PAHs will be comparable to the U.S. EPA guidelines, eliminating the need for default adjustment factors to account for unmeasured PAHs. The U.S. EPA guidelines are designed to account for chemical-specific and sediment-specific factors affecting the bioavailability and toxicity of PAH mixtures. Alkylated PAHs will be measured using a modification of U.S. EPA Method 8270.

- *Pesticides:* Standard pesticide analyses, together with TOC, provide sufficient information to estimate the site-specific bioavailability of DDT, endosulfan, and their degradation products in sediment. Pesticides will be analyzed using U.S. EPA Method 8081, which provides data for a suite of pesticides. ARCADIS will report analytical results for all pesticides and will update the screening evaluation for the pesticides not currently identified as chemicals of interest.

- *Acid volatile sulfide (AVS) and simultaneously extracted metals (SEM):* The U.S. EPA (2000b) has developed sediment quality guidelines for mixtures of six cationic metals (cadmium, copper, lead, nickel, silver, and zinc) based on AVS and SEM concentrations. Analysis of AVS and SEM (Allen, Fu et al. 1993) is required to estimate the site-specific bioavailability of these metals in sediment. Sediment TOC is used to further refine the evaluation of AVS and SEM results (U.S. EPA 2000b). Silver is not a chemical of interest for Johnson Lake, but it will be analyzed to assess potential cumulative effects of all six metals on benthic organisms (U.S. EPA 2000b).

  Although chromium does not form insoluble sulfide complexes, the likelihood of sediment toxicity due to chromium can also be predicted based on AVS data (Boothman, Berry et al. 1999). The toxicity and bioavailability of chromium

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[27] See Comment #14 in Attachment 2, Comment #12 in Attachment 4, and Comments #1, #2, and #3 in Attachment 6.

[28] See Comment #9 in Attachment 4.

depend on whether it is present as trivalent or hexavalent chromium. Hexavalent chromium reacts with AVS, such that AVS and hexavalent chromium will not coexist in sediments. Trivalent chromium is highly insoluble and relatively non-toxic, and as a result, chromium toxicity is not expected in sediments which contain AVS (Boothman, Berry et al. 1999). Indeed, amphipods that constructed burrows in chromium hydroxide precipitate exhibited no adverse effects in laboratory tests (Warren Boothman, U.S. EPA, personal communication). Consequently, for the ecological risk assessment, chromium-related toxicity to benthic invertebrates will be considered unlikely if AVS is consistently detected in sediment samples.

- *Porewater metals:* Sediment porewater will be extracted by centrifugation under an inert atmosphere for analysis of dissolved arsenic and selenium. While extensive research has demonstrated that chemical concentrations in porewater are a much better predictor of toxicity than whole-sediment concentrations, the direct measurement of metals in porewater is subject to conservative bias (U.S. EPA 2000b). Specifically, porewater extraction procedures such as centrifugation can result in an overestimation of the metal concentrations occurring in the porewater of undisturbed sediment. Also, characteristics of porewater, such as elevated concentrations of dissolved organic carbon, can reduce the bioavailability of metals in porewater compared with water-only exposures. For these reasons, analyses of chemicals of interest in porewater will be limited to arsenic and selenium, because these are the only chemicals of interest for which methods have not been developed to estimate site-specific bioavailability based on whole-sediment analyses.

- *Total metals:* Total metals will be analyzed using U.S. EPA Method 6010 to support the exposure assessment for hypothetical recreators who may wade in Johnson Lake sediment. As described above, the risk assessment for benthic organisms will rely on analyses of SEM-AVS and porewater metals rather than total metals.

- *Total petroleum hydrocarbons:* C-10 to C-34 of TPH will be analyzed at the request of Oregon DEQ, based on historical evidence of sheens on Johnson Lake.

- *Total organic carbon:* TOC is required to estimate the bioavailability of most chemicals of interest based on whole-sediment analyses.

- *Moisture content:* This is required to determine chemical concentrations on a dry weight basis.

- *Grain size:* Grain size will be compared to sediment toxicity test results, because grain size distribution can affect toxicity test organisms, apart from any effects related to chemicals of interest.

- *Porewater ammonia:* Ammonia occurs naturally in sediment and can influence sediment toxicity test results. Ammonia will be analyzed in the overlying water of laboratory exposure chambers as part of the sediment toxicity test protocol. Analyses of ammonia in porewater will provide additional information to facilitate the interpretation of sediment toxicity test results.

- *Porewater pH:* pH will be measured in porewater to allow interpretation of the porewater ammonia data. The aquatic toxicity of ammonia is pH-dependant.

Sediment will be collected from each of the eight sampling zones for all analyses. Additionally, a subset (i.e., three) of the sediment samples will be analyzed for PCB congeners[28]. The sampling zones in Johnson Lake for PCB congener analysis will be randomly selected.

3.2.3 Biota Tissue Analyses

The primary purpose of analyzing biota tissue is to assess chemical exposures for humans and wildlife that could eat aquatic organisms. In addition, fish tissue analyses will be used as part of the assessment of risks to fish. All biota tissue samples will be collected concurrently with samples for sediment chemistry and toxicity testing[29] and analyzed for PCB homologues, pesticides, selenium, lead, and lipid content. PCB congeners will be analyzed in a limited subset of biota samples for comparison purposes only, at the request of Oregon DEQ[30]. Several types of biota will be sampled,

[29] See Comment #15 in Attachment 2.

[30] See Comment #14 in Attachment 2, Comment #12 in Attachment 4, and Comments #1, #2, and #3 in Attachment 6.

contingent on their occurrence in Johnson Lake[31]. The rationale for each biota type is provided below.

- *Game fish fillets:* Fillet samples represent the portion of fish that is normally consumed by humans. Game fish include large fish that are often the preferred object of recreational fishing, such as bass. Because such species are at the top of the aquatic food web, they tend to contain higher concentrations of bioaccumulative chemicals, compared to other species.

- *Pan fish fillets:* Pan fish, such as bluegills, tend to be smaller than game fish (i.e., pan-sized). Because such species tend not to consume other fish as prey they may contain lower concentrations of bioaccumulative chemicals than do game fish. However, they may be consumed in greater quantities than game fish, if they are more plentiful.

- *Rough fish fillets:* Rough fish, such as carp, generally are not preferred for eating but may occasionally be consumed. Because rough fish tend to forage along the bottom of water bodies, their body burdens may be correlated with sediment concentrations.

- *Large whole fish:* Although large whole fish (>350 mm) are not generally consumed by wildlife or humans, analyses of large whole fish samples are needed to support the risk assessment for fish. Chemical concentrations in large whole fish will be calculated from the concentrations in fillet samples and in the remaining carcass (see below).

- *Small whole fish:* Whole fish samples represent fish as they are consumed by wildlife. Multiple size classes of fish will be analyzed, because chemicals of interest accumulate to different degrees in fish of different sizes. Small fish (up to 125 mm) are the preferred prey of smaller fish-eating (piscivorous) birds, such as kingfishers. Larger piscivores may also consume small fish as a portion of their diet.

- *Medium whole fish:* Larger piscivores, such as mink or herons, consume medium-sized fish (125 to 350 mm).

[31] See Comment #16 in Attachment 2.

- *Benthic invertebrates:* Benthic invertebrates serve as an important component of the diet of various wildlife species, such as sandpipers, ducks, and raccoons, and are also consumed by primarily piscivorous wildlife species, such as kingfishers and herons. Sampling will target large invertebrates, for example, mollusks or crayfish, if available.

- *Aquatic plants:* Aquatic herbivores and omnivores, such as muskrats and ducks, may consume significant quantities of aquatic plants. Aquatic plants may not occur or be accessible to wildlife in all sampling zones, however.

Multiple individual organisms will be composited to satisfy sample volume requirements and to represent average exposure concentrations. Fish collected for fillet analysis and large whole fish analysis will be collected on a lake-wide basis, because these fish are expected to be mobile throughout the lake. Three samples will be collected for each type of fillet (game, pan, and rough fish); one sample of each of the fillet samples will be submitted additionally for PCB congener analysis. Samples will be filleted in the field, and the remaining carcass will be submitted separately for analysis. Whole-body concentrations in large fish will be calculated from the fillet and carcass data. The mass of each fillet and carcass sample will be determined to support this calculation.

Small and medium fish will be collected from each of the eight sampling zones shown in Figure 3-3, because they tend to inhabit smaller home ranges than large fish. Because wildlife generally obtain invertebrate prey from shallow water, invertebrates will be sampled only from the six sampling zones along the shoreline of Johnson Lake and not from the two sampling zones in the center of the lake. Three samples each of medium whole fish and benthic invertebrates collected from Johnson Lake will be submitted additionally for PCB congener analysis. The sampling zones in Johnson Lake for PCB congener analyses will be randomly selected. Similar to invertebrates, aquatic plants will be collected from the two sampling zones in the center of Johnson Lake only if they occur near the water surface. If practical, each biota type will be represented by the same species in all samples, to minimize variation unrelated to the spatial distribution of chemicals of interest. Otherwise, the most available species in each zone will be selected. If a given biota type is absent, then exposure pathways involving consumption of that biota type in the designated sampling zone will be considered incomplete.

4. Risk Assessment

The data collected in accordance with this work plan will be evaluated, together with existing data, in baseline human health and ecological risk assessments. A Risk Assessment Work Plan will be prepared, describing exposure and toxicity factors, equations, and evaluation processes to be used in the risk assessments[32]. Risk assessment methods are summarized below for deterministic risk assessments. Probabilistic risk assessments are also permitted under Oregon DEQ rules. In the event that a probabilistic risk assessment is undertaken for Johnson Lake, a plan addressing specific issues identified in OAR 340-122-0084 will be submitted to the Oregon DEQ for approval prior to initiation of the assessment.

4.1 Human Health Risk Assessment

The baseline human health risk assessment (HHRA) will provide an evaluation of risks to human health based on current conditions. The HHRA will evaluate the potential risk associated with complete exposure pathways and potential receptors identified in Johnson Lake.

The HHRA will follow relevant U.S. EPA guidance, including:

- *Risk Assessment Guidance for Superfund (RAGS) - Parts A, D, and E* (U.S. EPA 1989; 1998a; 2001);
- *Calculating Upper Confidence Limits for Exposure Concentrations at Hazardous Waste Sites* (U.S. EPA 2002);
- *The Lognormal Distribution in Environmental Applications* (U.S. EPA 1997a);
- *Exposure Factors Handbook* (U.S. EPA 1997b); and
- *Dermal Exposure Assessment Guidance* (U.S. EPA 1992a).

[32] See Comment #19 in Attachment 2.

The HHRA will be consistent with Oregon Revised Statutes (ORS 465.315), Oregon Administrative Rules (OAR 340-122), and Oregon DEQ (1998b) guidance.

The HHRA will include the following basic components: hazard identification, exposure assessment, toxicity assessment, and risk characterization. Hazard identification includes the collection and analysis of relevant site data and the identification of chemicals of interest. Exposure assessment identifies exposed populations and potential exposure pathways, estimates exposure point concentrations, and calculates doses for each pathway. Toxicity assessment provides a compilation of quantitative and qualitative toxicity information about each chemical of interest and identifies toxicity values descriptive of the dose-response relationships. Finally, risk characterization estimates and summarizes the cancer risks and noncancer hazards for each exposure pathway and population. In addition, the HHRA will describe the degree of certainty and conservatism associated with each component of the HHRA.

A land use evaluation will be completed as part of the Risk Assessment Work Plan, in accordance with the *Final Guidance - Consideration of Land Use in Environmental Remedial Actions, Oregon Department of Environmental Quality, Waste Management and Cleanup Division, 1998*[33]. At the completion of this evaluation, a land use designation will be made and complete exposure scenarios will be finalized. However, based on current zoning designations within the locality of the facility (i.e., mixed industrial and undeveloped; see Figure 4-1), exposure scenarios are likely to include: site worker, trespasser[34], hypothetical recreator (wader[35]), and hypothetical angler[36]. These exposure scenarios are conservative, and thus are expected to be protective of all potential receptors. Each exposure scenario is depicted in the conceptual site model[37] for human health (Figure 4-2) and is briefly described below.

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[33] See Comment #17 in Attachment 2.

[34] See Comment #17 in Attachment 2.

[35] See Comment #12 in Attachment 2.

[36] See Comment #18 in Attachment 2.

[37] See Comment #7 in Attachment 2.

- *Site Worker Scenario:* Site workers could contact chemicals of interest in soil via dermal contact and incidental ingestion. All site workers are assumed to be adults (i.e., 18 years of age and older).

- *Trespasser Scenario:* Trespassers could contact chemicals of interest in on-site soil via dermal contact and incidental ingestion. Trespassers are assumed to be adolescent (12 to 18 years of age). Adults are also capable of trespassing; however, because adolescents have lower body weights than adults, the adolescent trespasser scenario is more conservative than (and, therefore, protective of) an adult trespasser scenario.

- *Hypothetical Recreator Scenario:* In the event that publicly owned portions of Johnson Lake are opened for recreation, recreators may contact chemicals of interest in sediment *via* dermal contact and incidental ingestion. Recreators are assumed to be either children (6 to 12 years of age) or adolescents (12 to 18 years of age).

- *Hypothetical Angler Scenario:* Johnson Lake is subject to a posted fishing ban, and there is no evidence that anglers use Johnson Lake. A hypothetical angler scenario will be evaluated, in part to determine whether it is necessary to continue to prohibit the consumption of fish from the lake. Anglers will be assumed to experience exposure to chemicals of interest primarily via fish consumption. Incidental exposures to sediment during fishing will also be evaluated.

The site worker and trespasser scenarios, which address potential exposure to on-site soil, are listed here in order to provide a complete conceptual site model for the site. However, these scenarios will not be included in the HHRA for Johnson Lake, which will address hypothetical exposures to sediment and biota. The site worker and trespasser scenarios will be included in any HHRA associated with future investigation(s) of on-site, upland areas, as appropriate.

For each exposure scenario, average and high-end intakes will be calculated for all complete exposure pathways. For each potentially exposed population, the identified risks and hazards will be aggregated across pathways. The estimated exposures will be compared to relevant toxicity benchmarks (i.e., cancer slope factors and noncancer reference doses) from U.S. EPA's Integrated Risk Information System (IRIS). For those chemicals that lack a U.S. EPA-verified toxicity value, data will be obtained from U.S. EPA's (1997c) Health Effects Assessment Summary Tables (HEAST). The results

of the cancer risk assessment will be compared to acceptable risk levels as defined in Oregon Administrative Rules (OAR 340-122-0115). Specifically, cancer risks associated with individual chemicals will be compared to an acceptable risk level of 1×10^{-6}, while cumulative cancer risk estimates will be compared to an acceptable risk level of 1×10^{-5}. The acceptable risk level for noncarcinogenic hazards is defined as a hazard index (HI) less than or equal to one.

4.2 Ecological Risk Assessment

Ecological risk assessment (ERA) is a process that evaluates the likelihood of adverse ecological effects resulting from exposure to one or more stressors. As described by the U.S. EPA (1998b), the process is used to systematically evaluate and organize data, information, assumptions, and uncertainties in order to help understand and predict the relationships between stressors and ecological effects, in a manner that is useful for environmental decision-making. The specific purpose of the ERA described in this work plan will be to identify potential risks associated with exposure of aquatic biota and wildlife receptors to chemicals in water and sediment of Johnson Lake. As such, it will provide an evaluation of the nature and geographical extent of the possible ecological risks, based on current environmental conditions, chemicals of interest, and ecological receptors in the study area.

4.2.1 Ecological Risk Assessment Approach

The ERA will follow relevant U.S. EPA guidance, including:

- *Framework for Ecological Risk Assessment* (U.S. EPA 1992b);
- *Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments* (U.S. EPA 1997d);
- *Guidelines for Ecological Risk Assessment* (U.S. EPA 1998b); and
- *Ecological Risk Assessment and Risk Management Principles for Superfund Sites* (U.S. EPA 1999).

The ERA will be consistent with Oregon Revised Statutes (ORS 465.315), Oregon Administrative Rules (OAR 340-122), and Oregon DEQ (1998a) guidance.

The conceptual approach to the ERA will involve: (1) defining the system to be studied; (2) identifying the study's goals and objectives; (3) providing the details of the analysis; and (4) describing conclusions of the analysis. This approach is consistent with the conceptual framework for ERA developed by the U.S. EPA (1992b), which includes three major components: problem formulation, analysis (exposure characterization and effects characterization), and risk characterization. Using this framework, the ERA will evaluate the nature and spatial extent of possible ecological effects based on current knowledge of environmental conditions in Johnson Lake.

Potential risks to aquatic life will be assessed for the fish and invertebrate communities in Johnson Lake. As described in Section 3, toxicity tests and chemical analyses will provide lines of evidence in the risk evaluation. The outcomes of these lines of evidence will be integrated in a qualitative weight of evidence evaluation, in order to draw conclusions regarding potential risks posed by chemicals of interest. Toxicity test results will receive greater weight than chemistry data, because toxicity tests represent a direct measurement of biological effects, whereas greater uncertainty is associated with biological inferences drawn from chemistry data. The relationship between toxicity and chemistry will be evaluated to identify the apparent causes of observed toxicity, if any. While soil data are used as part of the identification of chemicals of interest in this work plan, soil data will not be included in the ERA for Johnson Lake, because the additional sediment sampling described in this work plan will provide a more accurate characterization of exposure concentrations for aquatic life.

Potential risks to wildlife will be assessed by comparing estimates of average daily doses and toxicity reference values for representative receptors of interest. Selection criteria for receptors include sensitivity, exposure potential, likely presence in the study area, ecological relevance, trophic level, feeding habits, and the availability of life history information. For each receptor of interest, average daily doses will be calculated for each chemical of interest using information compiled in the U.S. EPA's (1993c) *Wildlife Exposure Factors Handbook*. Toxicity reference values will be identified using methods developed by Sample, Opresko et al. (1996).

Consistent with Oregon Administrative Rules, risk estimates will be calculated at the individual level for threatened or endangered species and at the population level for other species. Local populations will be defined spatially as those organisms within each species that inhabit or use Johnson Lake. For individual-level risk estimates (if any), an acceptable level of risk is attained if the probability of important changes in such factors as growth, survival, fecundity, or reproduction related to the health and viability of an individual ecological receptor that are reasonably likely to occur as a

consequence of exposure to hazardous substances is *de minimis*. Acceptable risk at the individual level may be demonstrated by calculating cumulative risks attributable to systemic toxicants with similar endpoints for similarly responding species, where a toxicity index less than or equal to one is defined as acceptable. The acceptable risk level for populations of ecological receptors is defined as (1) a 10 percent chance, or less, that no more than 20 percent of the total local population will be exposed to an exposure point value greater than a chronic LC50 or LD50 for each contaminant of concern, and (2) no other observed significant adverse effects on the health or viability of the local population. Toxicity test results will be related to acceptable risk levels based on the spatial extent and severity of observed toxicity, if any.

4.2.2 Ecological Conceptual Site Model

The ecological conceptual site model for Johnson Lake[38], including chemical sources, exposure pathways, and receptors of interest[39], is illustrated in Figure 4-3. Receptors of interest are selected because it is not feasible to quantitatively evaluate potential risks to all species that may use Johnson Lake. Such an evaluation would also be duplicative, due to similarities in exposures within functional groups (e.g., feeding guilds). Selection criteria for receptors of interest include sensitivity, exposure potential, likely presence in the study area, ecological relevance, trophic level, feeding habits, and the availability of life history and toxicological information. Ecological receptors of interest selected for Johnson Lake include the following:

- *Benthic Invertebrates:* The benthic invertebrate community lives in constant and direct contact with sediment that may be impacted by chemicals of interest. Benthic invertebrates have vital functions within the ecosystem, including serving as a prey base for higher trophic level organisms and cycling of nutrients.

- *Fish:* The fish community lives in constant and direct contact with surface water that may be impacted by chemicals of interest. Exposures are also possible via sediment and the food chain (i.e. secondary consumers), particularly for bioaccumulative chemicals. The fish community often

[38] See Comment #7 in Attachment 2.

[39] See Comment #16 in Attachment 2.

dominates perennial aquatic ecosystems, in terms of biomass, and fish serve as a prey base for piscivorous wildlife.

- *Belted kingfisher (Ceryle alcyon):* The belted kingfisher is selected to represent piscivorous birds in stream and pond systems. Kingfishers may be found in shallow, open water alongside rivers, streams, lakes, and ponds (Hamas 1974, as cited in U.S. EPA 1993c). They prefer shallow open water for foraging (Brewer, McPeek et al. 1991). Kingfishers nest in high vertical cutbanks of friable (sandy-clay) soil. Exposure potential for kingfishers is enhanced by the high proportion of fish in their diet and their limited territory sizes.

- *Great Blue Heron (Ardea herodias):* Great blue herons are evaluated in the ERA as a second representative of the feeding guild of piscivorous birds, in light of substantial differences between herons and kingfishers in feeding preferences and ingestion rates relative to body weight. Fish consumed by great blue herons (up to 300 mm in length) are larger than those consumed by belted kingfishers.

- *Mink (Mustela vison):* Mink are opportunistic feeders on aquatic prey such as fish, crustaceans, and amphibians and represent the piscivorous mammal feeding guild. Mink also feed on terrestrial mammals. Mink were selected because they are more toxicologically sensitive to contaminants, particularly organochlorine compounds, than are most other wildlife species. However, exposure potential is mitigated by their opportunistic feeding habits and large territory sizes. Landform characteristics preferred by mink include irregular shorelines with brushy or wooded cover (Allen 1986).

- *Spotted Sandpiper (Actitis macularia):* Spotted sandpipers represent the insectivorous bird feeding guild. Spotted sandpipers primarily feed on benthic invertebrates picked from the shoreline sediment or from the sediment surface in shallow water. Spotted sandpipers are the most geographically widespread sandpipers, inhabiting pebbly lakeshores, ponds, streamsides, and seashores (Peterson 1980). Exposures of sandpipers to chemicals of interest are substantially augmented by high rates of sediment ingestion, as well as high food ingestion rates.

- *Mallard (Anas platyrhynchos):* Mallards are a receptor of interest representing the omnivorous bird feeding guild for streams and ponds. Although mallards

feed on plants during much of the year, they feed primarily on invertebrates during the breeding season. Mallards are among the more common species in this feeding guild and have been observed on-site. Given their tolerance for a broad range of habitats, either wintering or breeding populations are expected to inhabit the study area. Exposure of mallards to chemicals may be enhanced by their foraging strategy, "dabbling," in which they stir up sediment with their bills in an effort to locate prey. Incidental sediment ingestion may occur as a result.

- *Raccoon (Procyon lotor):* Raccoons represent the feeding guild of invertivorous mammals in aquatic systems. The raccoon has been reported to be the most abundant and widespread medium-sized omnivore in North America (U.S. EPA 1993c). Raccoons also tend to be more closely associated with aquatic systems than other mammalian omnivores, although they feed opportunistically from both aquatic and terrestrial sources. Exposures to raccoons are enhanced by relatively small territory sizes, as well as their potential to ingest sediment that may be attached to invertebrate prey.

- *Muskrat (Ondatra zibethicus):* Muskrats represent the herbivorous mammal feeding guild for wetlands. Compared to other herbivorous mammals, the potential for exposure for muskrats is enhanced by their foraging preferences (i.e., aquatic vegetation), den sites (i.e., along water banks), and limited territory size. Muskrats inhabit the area immediately surrounding their den sites and rarely travel more than 150 meters from their dens (MacArthur 1978, as cited in U.S. EPA 1993c).

In addition, records of rare, threatened, and endangered species occurring within two miles of the site (Appendix D) were obtained from the Oregon Natural Heritage Information Center to support the selection of receptors of interest for the ERA. Most of the species identified are not relevant to the ERA, either because they do not feed on aquatic organisms, or because Johnson Lake does not provide suitable habitat for the species. However, two state-listed turtle species (painted turtle and northwestern pond turtle) could potentially occur in Johnson Lake. Surveys will be conducted to determine whether these species are present in Johnson Lake and should be evaluated as part of the ERA. The ability to quantitatively assess risks to turtles is limited by the lack of toxicity data for reptiles (Sparling, Linder et al. 2000). Therefore, risks to painted turtles and northwestern pond turtles will be inferred from the results of the ERA for fish, birds, and mammals.

In addition to exposure pathways and receptors of interest, assessment and measurement endpoints are identified as part of the conceptual site model, to specify how potential risks will be evaluated for the identified receptors of interest. Assessment endpoints are explicit expressions of the environmental value that is to be protected, operationally defined by an ecological entity (e.g., birds, mammals) and its attributes (e.g., survival, growth, reproduction). Assessment endpoints are selected in this section of the work plan based on ecological relevance, susceptibility (which is a combination of toxicological sensitivity and potential for exposure), and relevance to management goals.

A measurement endpoint is defined as a measurable ecological characteristic that is related to the valued characteristic chosen as the assessment endpoint, and is a measure of biological effects. In some cases, it is possible to measure biological effects (e.g., through toxicity testing), in addition to inferring the potential for effects based on chemistry data. The measurement of biological effects decreases the extent of extrapolation between the measurement and the goal. Comparisons of estimated exposures with toxicological information for each chemical of interest facilitate the interpretation of toxicity test results and serve as the primary measurement endpoint where toxicity test results are not available. Thus, more than one measurement endpoint may be selected for a given assessment endpoint.

The selected assessment endpoints that will be evaluated in the ERA are:

- Benthic macroinvertebrate community structure and function;
- Survival and reproduction of fish populations;
- Survival and reproduction of bird populations;
- Survival and reproduction of mammal populations; and
- If appropriate (pending survey results), survival and reproduction of individual painted turtles and/or northwestern pond turtles.

The corresponding measurement endpoints are:

- For benthic macroinvertebrates: (1) whole-sediment toxicity tests, and (2) comparison of chemical concentrations in sediment to concentrations associated with adverse effects on macroinvertebrates;

- For fish: comparison of chemical concentrations in fish tissue to concentrations associated with adverse effects on fish;
- For birds and mammals: comparison of the predicted intake of chemicals of interest in sediment and biota to toxicity reference values; and
- For turtles (if appropriate): inference from risk estimates for fish, birds, and mammals, calculated at the individual rather than population level.

5. Reporting

Data collected in and adjacent to Johnson Lake will be reported as part of the human health and ecological risk assessment reports. Data will be provided in electronic format. Specific project reporting and schedule information is contained in the Project Management Plan, provided as Appendix E.

6. Summary

A screening evaluation of existing sediment and surface soil data collected in and adjacent to Johnson Lake was completed to identify chemicals of interest for further evaluation. Chemicals of interest include PCBs, PAHs, two groups of pesticides (DDT and endosulfan and their metabolites), eight metals (arsenic, cadmium, chromium, copper, lead, nickel, selenium, and zinc), and TPH. Additional sampling of sediment and biota tissue is planned and will support the completion of human health and ecological risk assessments for the site.

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Table 3-1. Summary of Chemical Concentrations Detected in Sediment, Johnson Lake, Portland, Oregon

Chemical Name	Units	Range of Detects		Arithmetic Mean[a]	Frequency of Detection	Frequency of Detection (%)
		Minimum	Maximum			
VOCs						
Acetone	µg/kg	16	750	188	2 / 5	40%
Ethylbenzene	µg/kg	4	4	7	1 / 5	20%
Methylene chloride	µg/kg	6	200	49	2 / 5	40%
Tetrachloroethene	µg/kg	40	40	14	1 / 5	20%
Toluene	µg/kg	2.2	4,600	924	2 / 5	40%
SVOCs						
2-Methylnaphthalene	µg/kg	14	14	11	1 / 5	20%
4-Methylphenol	µg/kg	25	1,600	548	4 / 5	80%
Acenaphthene	µg/kg	11	11	10	1 / 5	20%
Acenaphthylene	µg/kg	3.3	22	11	2 / 5	40%
Anthracene	µg/kg	6	57	24	4 / 5	80%
Benzo(a)anthracene	µg/kg	19	230	77	5 / 5	100%
Benzo(a)pyrene	µg/kg	36	270	107	5 / 5	100%
Benzo(b)fluoranthene	µg/kg	38	290	113	5 / 5	100%
Benzo(g,h,i)perylene	µg/kg	20	250	99	5 / 5	100%
Benzo(k)fluoranthene	µg/kg	28	200	83	5 / 5	100%
Benzoic acid	µg/kg	60	120	185	2 / 5	40%
Benzyl alcohol	µg/kg	92	92	29	1 / 5	20%
Bis(2-ethylhexyl)phthalate	µg/kg	130	520	546	3 / 5	60%
Butylbenzylphthalate	µg/kg	30	30	23	1 / 5	20%
Chrysene	µg/kg	38	380	140	5 / 5	100%
Dibenz(a,h)anthracene	µg/kg	5.2	12	24	3 / 5	60%
Dibenzofuran	µg/kg	15	15	11	1 / 5	20%
Di-n-octyl phthalate	µg/kg	29	29	23	1 / 5	20%
Fluoranthene	µg/kg	51	240	128	5 / 5	100%
Fluorene	µg/kg	3.1	28	14	2 / 5	40%
Indeno(1,2,3-cd)pyrene	µg/kg	20	280	101	5 / 5	100%
Naphthalene	µg/kg	2.9	2.9	8.6	1 / 5	20%
Phenanthrene	µg/kg	18	140	76	5 / 5	100%
Phenol	µg/kg	36	130	63	3 / 5	60%
Pyrene	µg/kg	45	510	185	5 / 5	100%
Total PAHs	µg/kg	347	3040	1210	5 / 5	100%
PCBs						
Aroclor 1260	µg/kg	31	380	124	7 / 13	54%
Pesticides						
4,4-DDE	µg/kg	1	2.5	0.76	2 / 5	40%
4,4-DDT	µg/kg	3.1	11	6.0	3 / 5	60%
Aldrin	µg/kg	17	17	3.6	1 / 5	20%
alpha-Chlordane	µg/kg	1.2	1.2	0.60	1 / 5	20%
Endrin ketone	µg/kg	1.8	36	8.2	2 / 5	40%
gamma-Chlordane	µg/kg	0.22	0.22	0.35	1 / 5	20%

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Table 3-1. Summary of Chemical Concentrations Detected in Sediment, Johnson Lake, Portland, Oregon

Chemical Name	Units	Range of Detects		Arithmetic Mean[a]	Frequency of Detection	Frequency of Detection (%)
		Minimum	Maximum			
Total Metals						
Aluminum	mg/kg	10,600	19,000	14,520	5 / 5	100%
Antimony	mg/kg	0.2	0.2	3	1 / 5	20%
Arsenic	mg/kg	2	8.5	4.8	5 / 5	100%
Barium	mg/kg	74	83	78	2 / 2	100%
Beryllium	mg/kg	0.3	0.5	0.5	3 / 5	60%
Cadmium	mg/kg	1.4	8	2.8	4 / 5	80%
Calcium	mg/kg	4,290	6,400	5,442	5 / 5	100%
Chromium	mg/kg	18	48	36	5 / 5	100%
Cobalt	mg/kg	6	10	8.7	5 / 5	100%
Copper	mg/kg	14	35	24	5 / 5	100%
Cyanide	mg/kg	0.56	0.56	0.31	1 / 3	33%
Iron	mg/kg	19,300	27,600	22,500	5 / 5	100%
Lead	mg/kg	34	124	65	5 / 5	100%
Magnesium	mg/kg	4,120	4,860	4,374	5 / 5	100%
Manganese	mg/kg	122	259	172	5 / 5	100%
Mercury	mg/kg	0.04	0.11	0.074	5 / 5	100%
Molybdenum	mg/kg	2	2	1	1 / 3	33%
Nickel	mg/kg	12	21	17	5 / 5	100%
Potassium	mg/kg	953	1,410	1,182	2 / 2	100%
Selenium	mg/kg	0.7	3	2	3 / 5	60%
Silver	mg/kg	0.08	0.08	0.6	1 / 5	20%
Sodium	mg/kg	367	776	572	2 / 2	100%
Thallium	mg/kg	0.3	0.5	0.52	3 / 5	60%
Vanadium	mg/kg	69	82	76	2 / 2	100%
Zinc	mg/kg	144	376	248	5 / 5	100%
Other						
Total organic carbon (TOC)	%	0.092	5.6	1.7	13 / 13	100%
Solids	%	19	66	37	5 / 5	100%

a. Average of detected concentrations and one-half the reporting limit for non-detects.

DDE - 1,1-dichloro-2,2-bis(chlorophenyl)ethylene
DDT - 1,1,1-trichloro-2,2-bis(p-chlorophenyl)ethane
mg/kg - milligrams per kilogram
PAHs - polycyclic aromatic hydrocarbons
PCBs - polychlorinated biphyenyls
SVOCs - semi-voltile organic compounds
µg/kg - micrograms per kilogram
VOCs - volatile organic compounds
% - percent

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Table 3-2. Summary of Chemical Concentrations Detected in On-Site Soil, Johnson Lake, Portland, Oregon

Chemical Name	Units	Range of Detects Minimum	Range of Detects Maximum	Arithmetic Mean[a]	Frequency of Detection	Frequency of Detection (%)
VOCs						
2-Butanone (MEK)	µg/kg	17	17	15	1 / 10	10%
Acetone	µg/kg	26	26	37	1 / 10	10%
Methylene chloride	µg/kg	1.6	4.9	5.3	5 / 10	50%
Toluene	µg/kg	1.4	30	5.6	5 / 10	50%
SVOCs						
2-Methylphenol	µg/kg	10	10	234	1 / 10	10%
4-Methylphenol	µg/kg	43	390	274	2 / 10	20%
Anthracene	µg/kg	11	11	255	1 / 10	10%
Benzo(a)anthracene	µg/kg	12	68	254	3 / 10	30%
Benzo(a)pyrene	µg/kg	11	140	272	3 / 10	30%
Benzo(b)fluoranthene	µg/kg	58	160	282	3 / 10	30%
Benzo(g,h,i)perylene	µg/kg	15	250	292	4 / 10	40%
Benzo(k)fluoranthene	µg/kg	73	73	264	1 / 10	10%
Benzyl alcohol	µg/kg	120	590	354	4 / 10	40%
Bis(2-ethylhexyl) phthalate	µg/kg	370	1,400	5,041	3 / 10	30%
Butylbenzylphthalate	µg/kg	89	340	330	5 / 10	50%
Chrysene	µg/kg	61	150	281	5 / 10	50%
Di-n-butylphthalate	µg/kg	44	2,800	675	7 / 10	70%
Fluoranthene	µg/kg	59	170	279	3 / 10	30%
Indeno(1,2,3-cd)pyrene	µg/kg	78	130	270	2 / 10	20%
Phenanthrene	µg/kg	13	39	259	2 / 10	20%
Phenol	µg/kg	32	32	702	1 / 10	10%
Pyrene	µg/kg	91	260	306	5 / 10	50%
PCBs						
Aroclor 1260	µg/kg	30	14,000	2,214	14 / 17	82%
Pesticides						
4,4'-DDD	µg/kg	11	13	6.5	2 / 10	20%
4,4'-DDE	µg/kg	14	14	6.0	1 / 10	10%
4,4'-DDT	µg/kg	18	1,500	305	7 / 10	70%
alpha-Chlordane	µg/kg	13	47	13	4 / 10	40%
Endosulfan I	µg/kg	11	16	7.8	3 / 10	30%
Endosulfan II	µg/kg	24	24	20	1 / 10	10%
Endosulfan sulfate	µg/kg	140	140	19	1 / 10	10%
gamma-Chlordane	µg/kg	22	57	15	3 / 10	30%
Methoxychlor	µg/kg	13	38	15	2 / 10	20%
Total Metals						
Aluminum	mg/kg	482	16,400	8,811	10 / 10	100%
Arsenic	mg/kg	1.7	26	10	10 / 10	100%
Barium	mg/kg	17	145	83	10 / 10	100%
Cadmium	mg/kg	1.9	109	17	10 / 10	100%
Calcium	mg/kg	1,640	8,510	4,467	10 / 10	100%
Chromium	mg/kg	8.4	120	44	10 / 10	100%
Cobalt	mg/kg	3.5	15	8.7	9 / 10	90%
Copper	mg/kg	9.1	248	83	10 / 10	100%
Iron	mg/kg	2,960	34,400	19,511	10 / 10	100%
Lead	mg/kg	26	649	194	10 / 10	100%

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Table 3-2. Summary of Chemical Concentrations Detected in On-Site Soil, Johnson Lake, Portland, Oregon

Chemical Name	Units	Range of Detects Minimum	Range of Detects Maximum	Arithmetic Mean[a]	Frequency of Detection	Frequency of Detection (%)
Total Metals, continued						
Magnesium	mg/kg	208	4,650	2,689	10 / 10	100%
Manganese	mg/kg	22	403	239	10 / 10	100%
Mercury	mg/kg	0.040	0.14	0.084	9 / 10	90%
Nickel	mg/kg	7.3	132	30	9 / 10	90%
Potassium	mg/kg	513	1,080	751	9 / 10	90%
Selenium	mg/kg	1.3	15	5.5	9 / 10	90%
Sodium	mg/kg	418	1,470	784	10 / 10	100%
Thallium	mg/kg	1.3	7.0	1.4	3 / 10	30%
Vanadium	mg/kg	3.3	78	55	10 / 10	100%
Zinc	mg/kg	207	8,590	1,378	10 / 10	100%
Other						
Solids	%	54	88	67	10 / 10	100%
Total organic carbon (TOC)	%	0.25	19	6.2	10 / 10	100%

a. Average of detected concentrations and one-half the reporting limit for non-detects.

DDD - 1,1-dichloro-2,2-bis(p-chlorophenyl)ethane
DDE - 1,1-dichloro-2,2-bis(chlorophenyl)ethylene
DDT - 1,1,1-trichloro-2,2-bis(p-chlorophenyl)ethane
mg/kg - milligrams per kilogram
PCBs - polychlorinated biphyenyls
SVOCs - semi-voltile organic compounds
µg/kg - micrograms per kilogram
VOCs - volatile organic compounds
% - percent

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Table 3-3. Maximum Johnson Lake Sediment and On-Site Soil Concentrations Compared to Screening and Local Baseline Values

Chemical Name	Units	Sediment Maximum	On-Site Soil Maximum	Oregon DEQ Level II Value	Oregon DEQ Level II Basis	DEQ Baseline Norm.-Prob. Plot	Chemical of Interest?
VOCs							
2-Butanone (MEK)	µg/kg	ND	17	1,100,000	B	NA	No (1)
Acetone	µg/kg	750	26	290	B	NA	No (2)
Methylene chloride	µg/kg	200	4.9	930	B	6.4	No (1)
Tetrachloroethene	µg/kg	40	ND	57	M	NA	No (1)
Toluene	µg/kg	4600	30	5,300	B	1.8	No (1)
SVOCs							
2-Methylnaphthalene	µg/kg	14	ND	20	M	<9.5	Yes (2)
2-Methylphenol	µg/kg	ND	10	8	M	NA	No (2)
4-Methylphenol	µg/kg	1,600	390	100	M	16	No (2)
Acenaphthene	µg/kg	11	ND	290	F	<8.7	Yes (2)
Acenaphthylene	µg/kg	22	ND	160	F	11.2	Yes (2)
Anthracene	µg/kg	57	11	57	F	13	Yes (2)
Benzo(a)anthracene	µg/kg	230	68	32	F	87	Yes (1)
Benzo(a)pyrene	µg/kg	270	140	32	F	100	Yes (1)
Benzo(b)fluoranthene	µg/kg	290	160	1,800	M	99	Yes (2)
Benzo(g,h,i)perylene	µg/kg	250	250	300	F	86	Yes (2)
Benzo(k)fluoranthene	µg/kg	200	73	27	F	104	Yes (1)
Benzoic acid	µg/kg	120	ND	65	M	110	No (2)
Benzyl alcohol	µg/kg	92	590	52-57	M	NA	No (2)
Bis(2-ethylhexyl)phthalate	µg/kg	520	1,400	750	F	1930	No (1)
Butylbenzylphthalate	µg/kg	30	340	63	M	151	No (2)
Chrysene	µg/kg	380	150	57	F	103	Yes (1)
Dibenz(a,h)anthracene	µg/kg	12	ND	33	F	27	Yes (2)
Dibenzofuran	µg/kg	15	ND	5,100	F	NA	No (1)
Di-n-butylphthalate	µg/kg	ND	2,800	NA	---	NA	No (1)
Di-n-octyl phthalate	µg/kg	29	ND	61	M	<6.7	No (1)
Fluoranthene	µg/kg	240	170	111	F	132	Yes (1)
Fluorene	µg/kg	28	ND	77	F	8.6	Yes (2)
Indeno(1,2,3-cd)pyrene	µg/kg	280	130	17	F	78	Yes (1)
Naphthalene	µg/kg	2.9	ND	176	F	<10	Yes (2)
Phenanthrene	µg/kg	140	39	42	F	88	Yes (1)
Phenol	µg/kg	130	32	48	F	11	Yes (1)
Pyrene	µg/kg	510	260	53	F	196	Yes (1)
Total PAHs	µg/kg	3,040	1,719	1,610	F	1078	Yes (1)
Total LPAHs	µg/kg	2,619	82	76	F	130	Yes (1)
Total HPAHs	µg/kg	2,170	1,428	193	F	1243	Yes (1)
PCBs							
Aroclor 1260	µg/kg	380	14000	34	F (total PCBs)	NA	Yes (1)
Pesticides							
4,4'-DDD	µg/kg	ND	13	0.3	B	6.7	Yes (1)
4,4-DDE	µg/kg	2.5	14	0.3	B	9.8	Yes (1)
4,4-DDT	µg/kg	11	1,500	0.3	B	1.8	Yes (1)
Aldrin	µg/kg	17	ND	40	F,B	NA	No (1)
alpha-Chlordane	µg/kg	1.2	47	10	M	0.29	No (2)
Endosulfan I	µg/kg	ND	16	110	B (endosulfan)	NA	Yes (2)
Endosulfan II	µg/kg	ND	24	110	B (endosulfan)	NA	Yes (2)
Endosulfan sulfate	µg/kg	ND	140	110	B (endosulfan)	NA	Yes (1)
Endrin ketone	µg/kg	36	ND	3	F (endrin)	NA	No (2)
gamma-Chlordane	µg/kg	0.22	57	4.5	F (chlordane)	2.1	No (2)
Methoxychlor	µg/kg	ND	38	990	B	NA	No (1)

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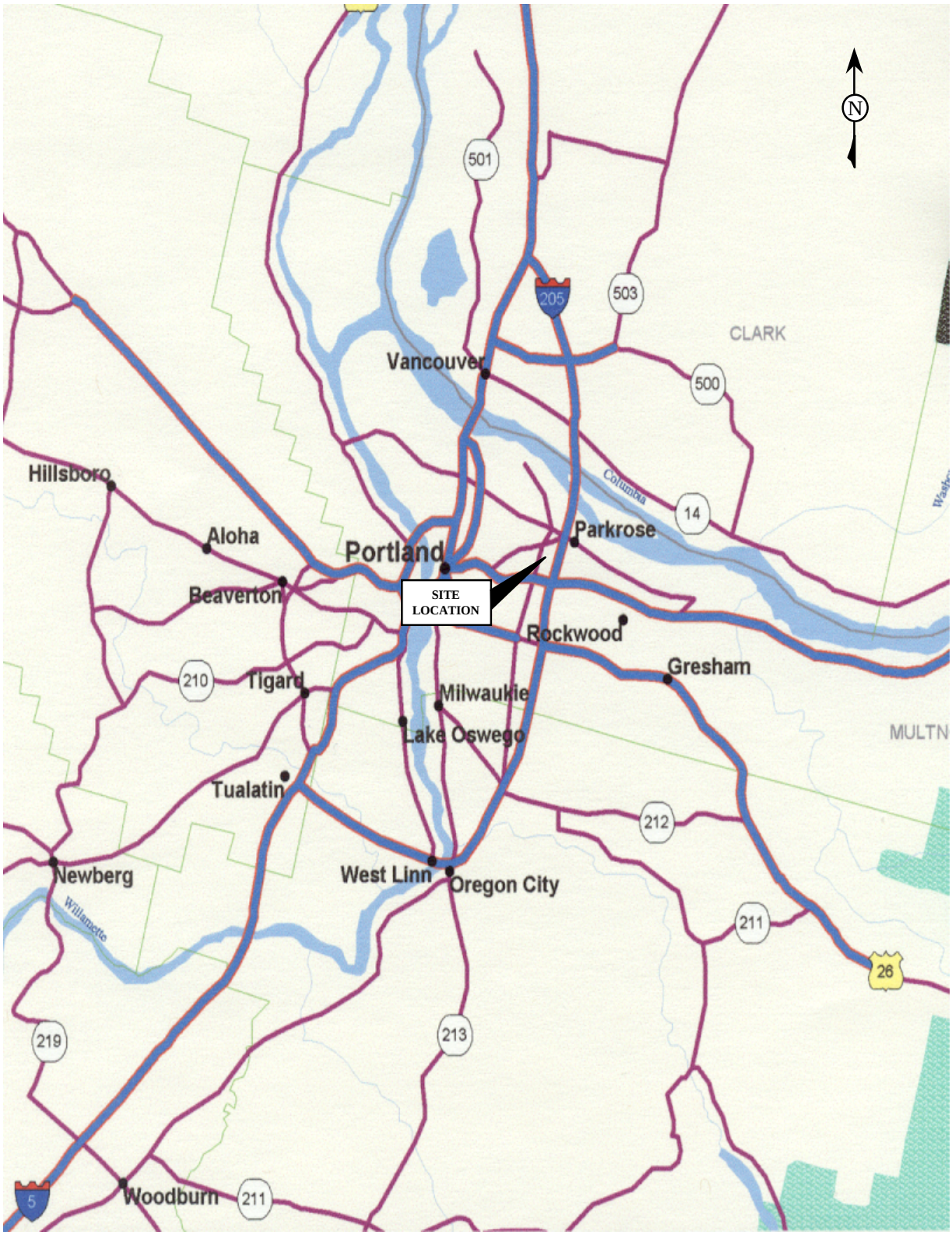
Table 3-3. Maximum Johnson Lake Sediment and On-Site Soil Concentrations Compared to Screening and Local Baseline Values

Chemical Name	Units	Sediment Maximum	On-Site Soil Maximum	Oregon DEQ Level II Value	Oregon DEQ Level II Basis	DEQ Baseline Norm.-Prob. Plot	Chemical of Interest?
Total Metals							
Aluminum	mg/kg	19,000	16,400	NA	---	NA	No (1)
Antimony	mg/kg	0.2	ND	3	F	NA	No (1)
Arsenic	mg/kg	8.5	25.5	4	B	8.5	Yes (1)
Barium	mg/kg	83	145	48	M	NA	No (2)
Beryllium	mg/kg	0.5	ND	122	B	NA	No (1)
Cadmium	mg/kg	8	109	0.003	B	2.3	Yes (1)
Calcium	mg/kg	6,400	8,510	NUT	---	NUT	No (1)
Chromium	mg/kg	48	120	37	F	71	Yes (1)
Cobalt	mg/kg	10	15	NA	---	NA	No (1)
Copper	mg/kg	35	248	10	B	72	Yes (1)
Cyanide	mg/kg	0.56	NA	NA	---	0.5	No (2)
Iron	mg/kg	27,600	34,400	NA	---	NA	No (1)
Lead	mg/kg	124	649	35	F	82	Yes (1)
Magnesium	mg/kg	4,860	4,650	NUT	---	NUT	No (1)
Manganese	mg/kg	259	403	1,100	F	438	No (1)
Mercury	mg/kg	0.11	0.14	0.2	F	0.18	No (1)
Molybdenum	mg/kg	2	NA	NA	---	<1.0	No (2)
Nickel	mg/kg	21	132	18	F	33	Yes (1)
Potassium	mg/kg	1,410	1,080	NUT	---	NUT	No (1)
Selenium	mg/kg	3	14.5	0.1	B	0.44	Yes (1)
Silver	mg/kg	0.08	ND	5	F	0.27	No (1)
Sodium	mg/kg	776	1,470	NUT	---	NUT	No (1)
Sulfide	mg/kg	24	NA	NC	---	NC	No (1)
Thallium	mg/kg	0.5	7.0	0.7	B	0.58	No (2)
Vanadium	mg/kg	82	78	57	F	NA	No (2)
Zinc	mg/kg	376	8,590	3	B	315	Yes (1)

a. Screening value is the lower of bioaccumulation (B) and freshwater (F) values. Marine (M) values are used if no freshwater value is available.

No (1) - Chemical is eliminated from further evaluation based on screening or baseline values or nutrient status.
No (2) - Chemical is eliminated from further evaluation based on additional information (see text).
Yes (1) - Chemical exceeds screening and baseline values.
Yes (2) - Chemical does not exceed screening and baseline values on an individual-chemical basis but will be evaluated to assess potential effects of a chemical class (PAHs or endosulfans).

DEQ - [Oregon] Department of Environmental Quality
DDE - 1,1-dichloro-2,2-bis(chlorophenyl)ethylene
DDT - 1,1,1-trichloro-2,2-bis(p-chlorophenyl)ethane
HPAHs - high molecular weight PAHs
LPAHs - low molecular weight PAHs
mg/kg - milligrams per kilogram
NUT - nutrient, no screening value available
PAHs - polycyclic aromatic hydrocarbons
PCBs - polychlorinated biphyenyls
SVOCs - semi-voltile organic compounds
µg/kg - micrograms per kilogram
VOCs - volatile organic compounds

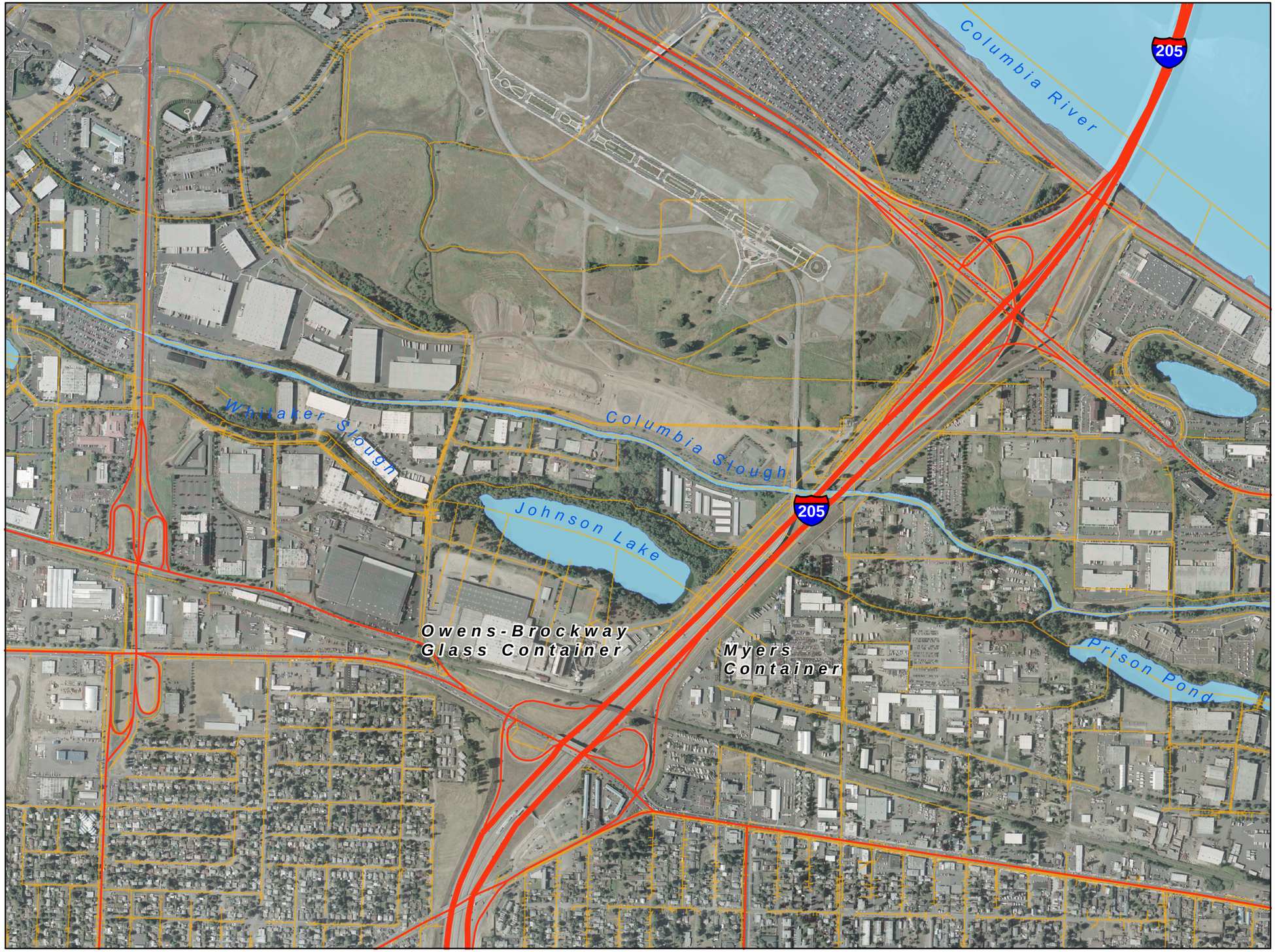


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Site Location Map
Johnson Lake
Portland, Oregon

Date	Project Manager	Drawing Name
8NOV02	T. Barber	Figure 1-1
Drawn By	Lead Design Prof.	Checked
K. Leigh		B. Wiesner
Project Number		Figure Number
CL000233.0001		1-1

Figure 2-1. Aerial Site Map
Johnson Lake
Portland, Oregon



Freeway
Arterial Streets
Sewer/Stormwater Lines
Surface Water

Basedata from METRO DRC 1"=100' orthophotos, City of Portland, and US Census Bureau 2000 Tiger Files. Positions may vary in accuracy due to scale differences between the data sets. Spatial information is meant for general reference only.

Lambert Conformal Conic projection, North American Datum 1983 HARN, Local coordinate grid State Plane Oregon (North) FIPS 3601.

Cartography by ARCADIS - Greenville, SC

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0 300 600 900 1,200 1,500
Feet

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Figure 2-2. Sediment Sampling Locations Johnson Lake Portland, Oregon

2003 Sediment Sampling Locations

Historic Sediment Sampling Locations

Basedata from METRO DRC 1"=100' orthophotos, City of Portland, and US Census Bureau 2000 Tiger Files. Positions may vary in accuracy due to scale differences between the data sets. Spatial information is meant for general reference only.

Lambert Conformal Conic projection, North American Datum 1983 HARN, Local coordinate grid State Plane Oregon (North) FIPS 3601.

Cartography by ARCADIS - Greenville, SC

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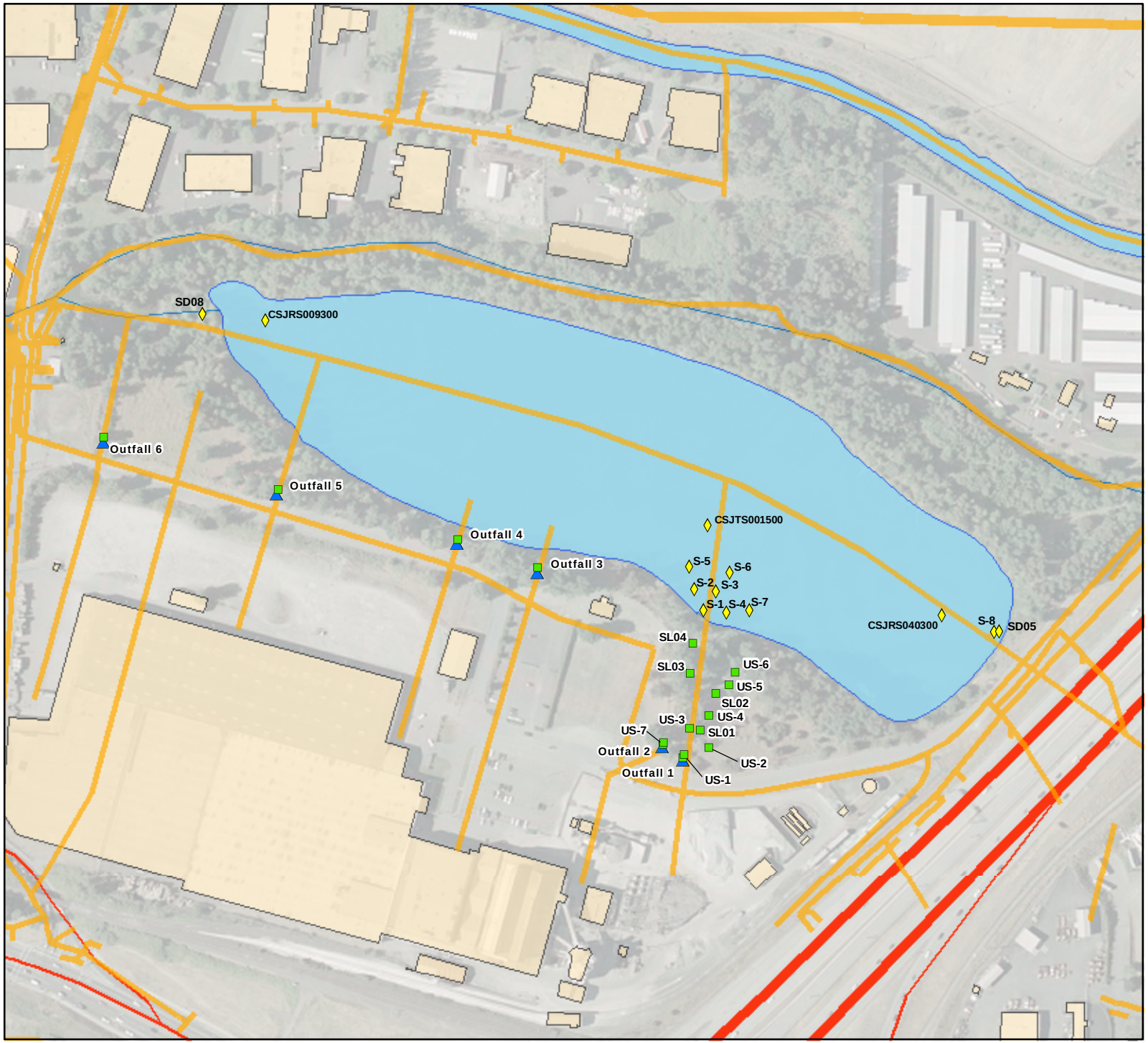


Figure 3-1. Sediment and On-Site Soil Sampling Locations Johnson Lake Portland, Oregon

- Surface Soil Sampling Locations
- Sediment Sampling Locations
- Surface Water
- Building Footprints
- Sewer/Stormwater Lines
- Arterial Streets
- Freeway

Basedata from METRO DRC 1"=100' orthophotos, City of Portland, and US Census Bureau 2000 Tiger Files. Positions may vary in accuracy due to scale differences between the data sets. Spatial information is meant for general reference only.

Lambert Conformal Conic projection, North American Datum 1983 HARN, Local coordinate grid State Plane Oregon (North) FIPS 3601.

Cartography by ARCADIS - Greenville, SC

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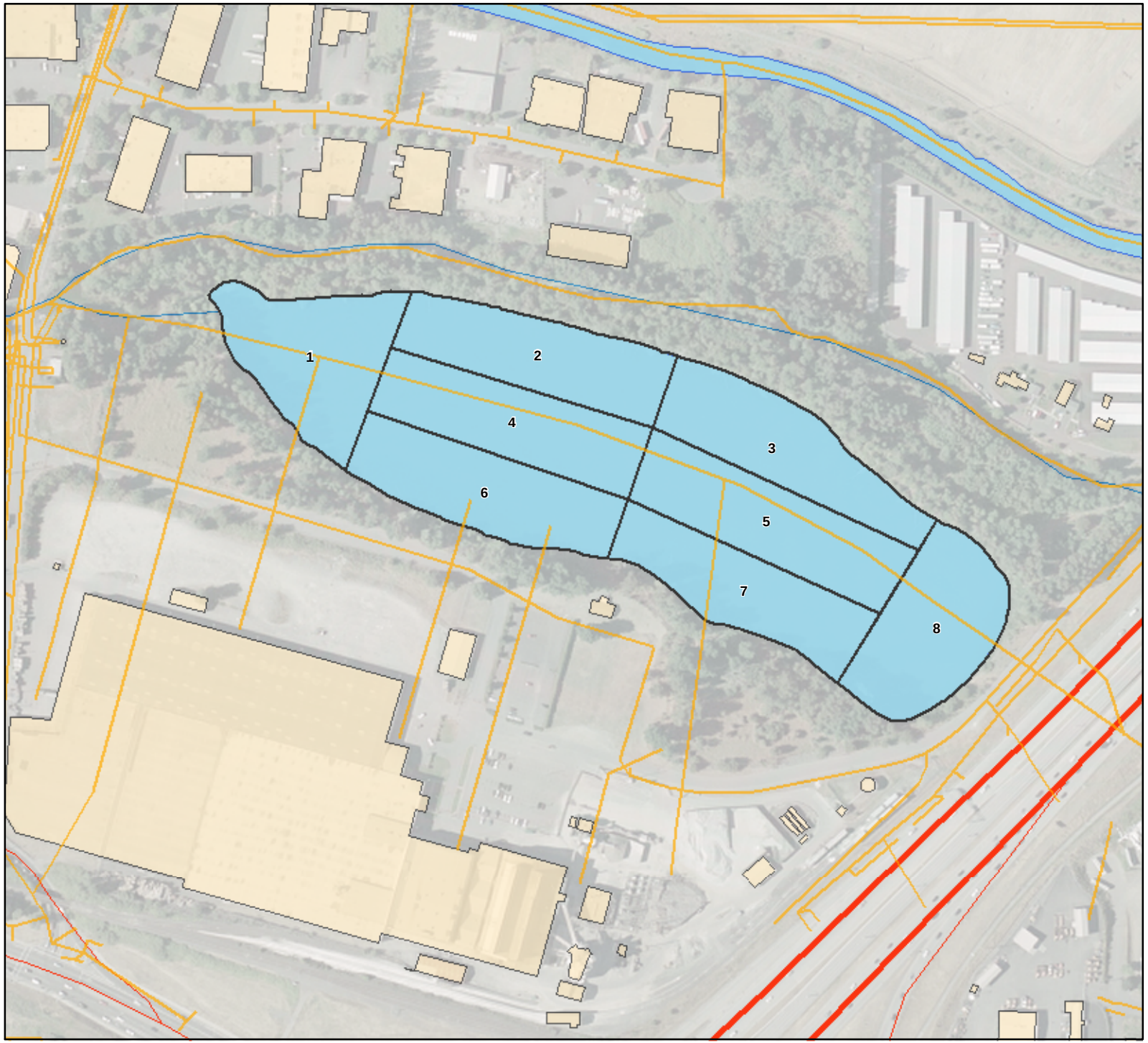


Figure 3-2.
Proposed Sampling Zones
Johnson Lake
Portland, Oregon

- Proposed Sampling Grid
- Surface Water
- Building Footprints
- Arterial Streets
- Freeway
- Sewer/Stormwater Lines

Basedata from METRO DRC 1"=100' orthophotos, City of Portland, and US Census Bureau 2000 Tiger Files. Positions may vary in accuracy due to scale differences between the data sets. Spatial information is meant for general reference only.

Lambert Conformal Conic projection, North American Datum 1983 HARN, Local coordinate grid State Plane Oregon (North) FIPS 3601.

Cartography by ARCADIS - Greenville, SC

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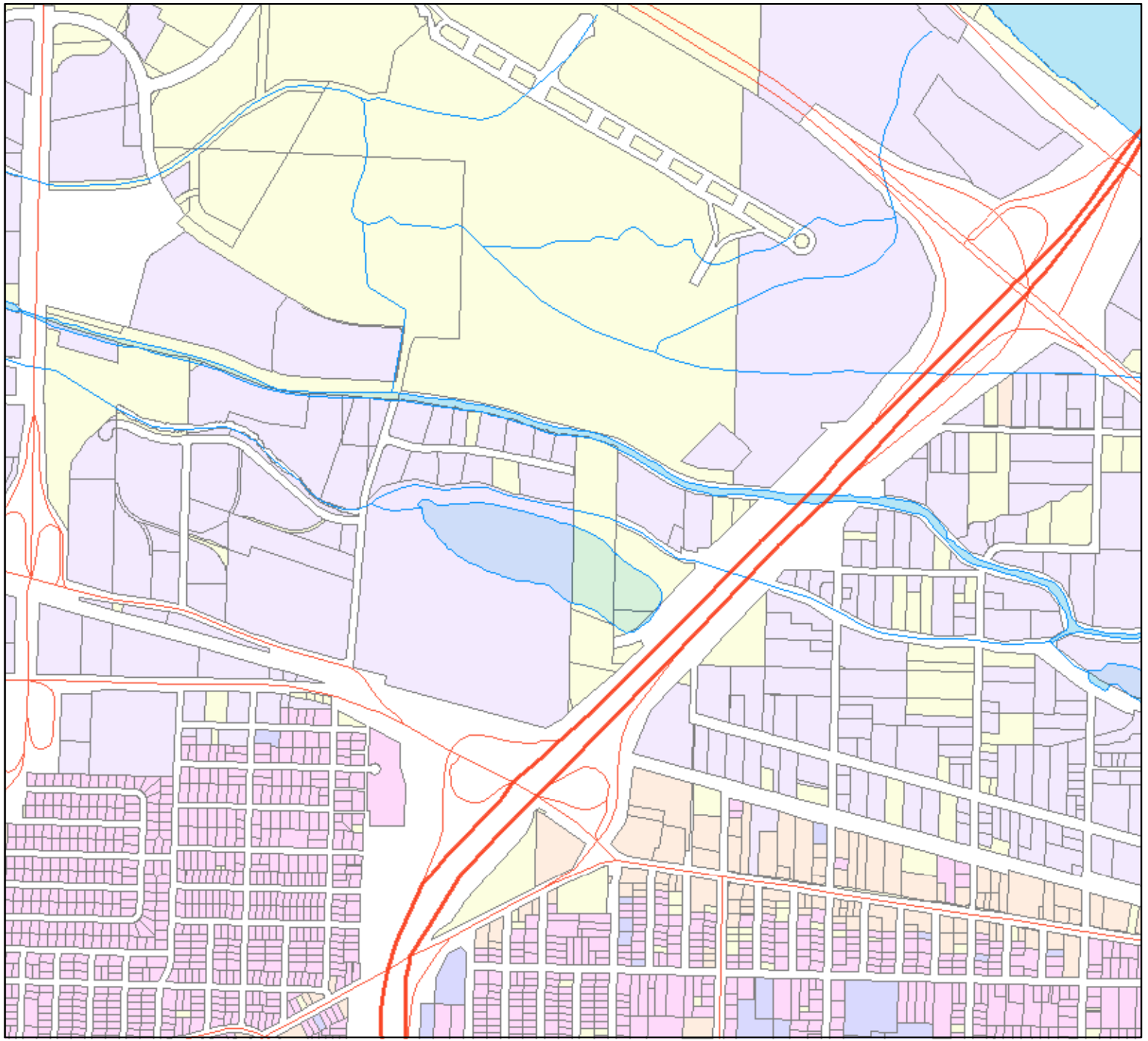


Figure 4-1. Current Land Use
Johnson Lake
Portland, Oregon

Legend

- Commercial
- Industrial
- Multi-family residential
- Single family residential
- Public/semi-public
- Undeveloped
- Johnson Lake
- River Line
- Freeway
- Arterial Streets

Basedata from METRO DRC 1"=100' orthophotos, City of Portland, and US Census Bureau 2000 Tiger Files. Positions may vary in accuracy due to scale differences between the data sets. Spatial information is meant for general reference only.

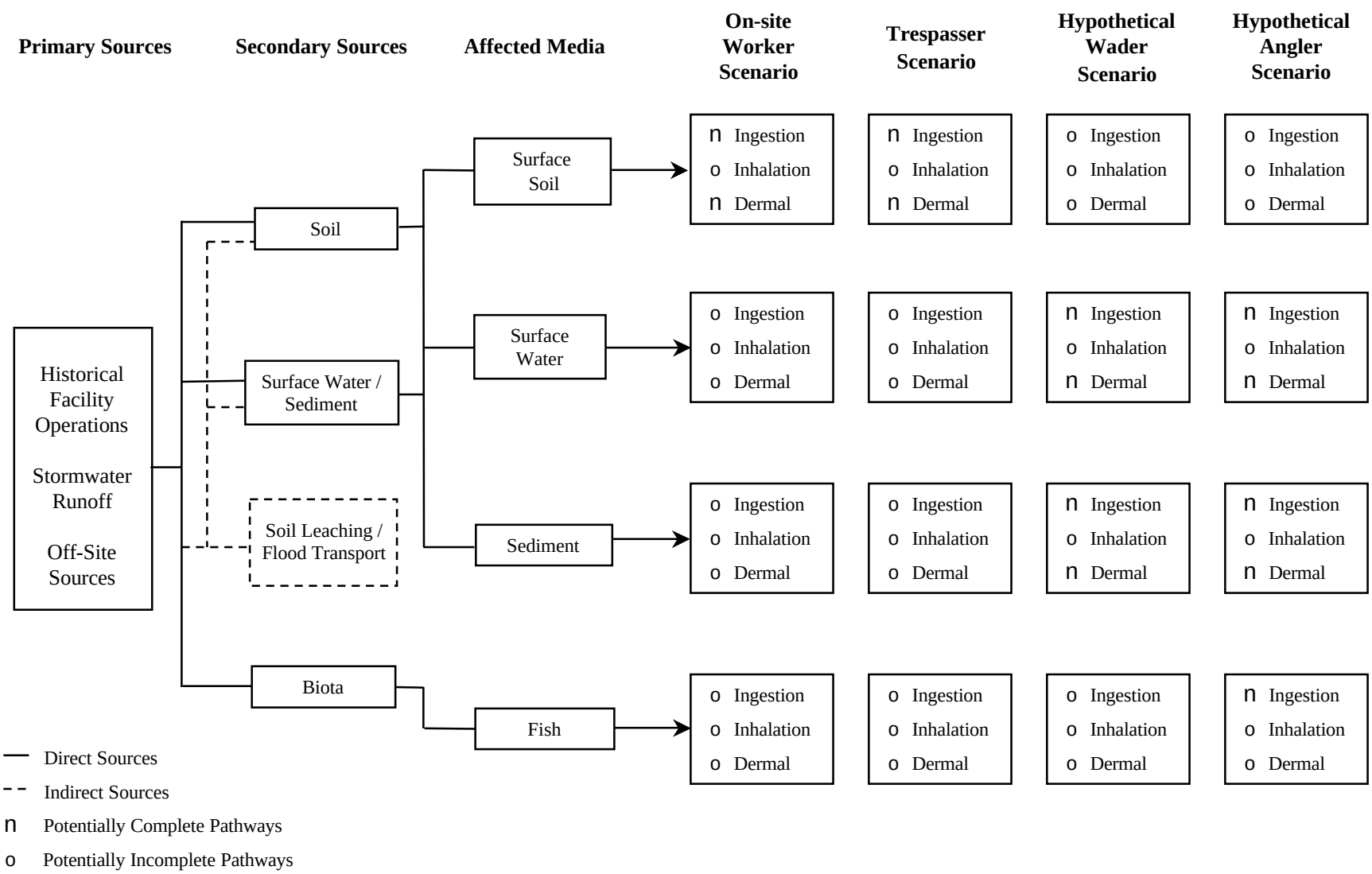
Lambert Conformal Conic projection, North American Datum 1983 HARN, Local coordinate grid State Plane Oregon (North) FIPS 3601.

Cartography by ARCADIS - Greenville, SC

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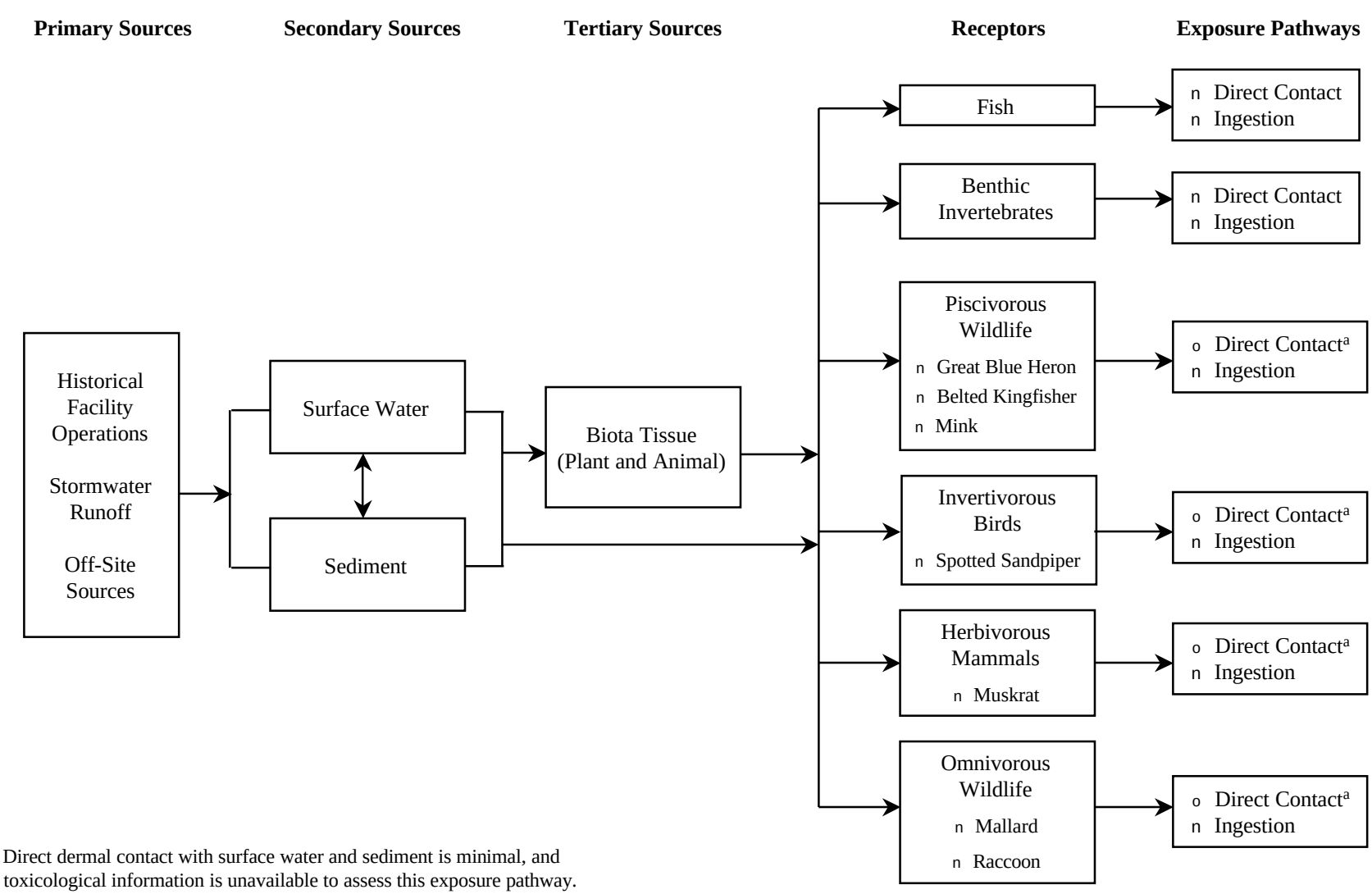
0 500 1,000 Feet

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Figure 4-2. Conceptual Site Model For Human Health Exposures



a. Direct dermal contact with surface water and sediment is minimal, and toxicological information is unavailable to assess this exposure pathway.

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Figure 4-3. Conceptual Site Model For Ecological Exposures

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Appendix A

Sampling and Analysis Plan (SAP)

Appendix A
Johnson Lake Investigation Sampling and Analysis Plan (SAP)

Owens-Brockway Glass Container, Inc.,
Portland, Oregon

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Infrastructure, buildings, environment, communications

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Table of Contents

1. Introduction **1**

2. Field Investigation **1**

2.1 Objectives and Study Design 1

2.2 Sampling Equipment 2

2.3 Sample Collection and Handling 3

2.3.1 Sediment 3

2.3.2 Biota 4

2.4 Sample Identification 5

3. Field Methodologies **6**

3.1 Health and Safety 6

3.2 Quality Control Samples 6

3.3 Decontamination 6

3.4 Waste Management Procedures 7

3.5 Field Documentation 7

4. Analytical and Toxicity Test Methods **7**

4.1 Quality Assurance 9

5. Data Validation, Reporting and Evaluation **9**

6. Schedule **10**

7. References **10**

Figures

A-1 Proposed Sampling Zones, Johnson Lake, Portland Oregon

1. Introduction

This Sampling and Analysis Plan (SAP) presents the approach for sampling sediment and biota tissue in Johnson Lake, Portland, Oregon. This SAP is an appendix to Revision 2 of the Johnson Lake Investigation Work Plan (IWP). In the Johnson Lake IWP (Revision 2), potential chemicals of interest for Johnson Lake were determined following a screening-level evaluation of existing sediment and surface soil data collected in and adjacent to Johnson Lake. The chemicals of interest in this SAP include polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), two groups of pesticides (DDT and its metabolites, DDE and DDD; and endosulfans, including endosulfan I and II and endosulfan sulfate), eight metals (arsenic, cadmium, chromium, copper, lead, nickel, selenium, and zinc), and total petroleum hydrocarbons (TPH). The objective of this SAP is to provide a consistent methodology for collection and analysis of environmental samples in Johnson Lake. All field personnel will have reviewed the SAP before sampling and will implement the plan during the sampling activities.

2. Field Investigation

2.1 Objectives and Study Design

The objective of the field sampling effort is to collect sediment and biota samples to support the characterization of conditions in Johnson Lake. Eight sampling zones will be determined in Johnson Lake (Figure A-1). Sediment and biota samples will be collected from each of the eight sampling zones.

Biota samples from Johnson Lake will include aquatic plants, benthic invertebrates, and fish. Because wildlife generally obtain plants and invertebrate prey from shallow water, plants and benthic invertebrates will be collected from the six sampling zones around the perimeter of Johnson Lake (Zones 1, 2, 3, 6, 7, and 8; see Figure A-1). Small and medium fish will be collected from each of the eight sampling zones in Johnson Lake (see Figure A-1), because they tend to inhabit smaller home ranges than large fish. Large fish (i.e., game, pan, and rough) will be collected on a lake-wide basis, because these fish are expected to be mobile throughout the lake. Small and medium fish will be submitted for whole-body analysis. Large fish (game, pan, and rough) will be filleted in the field, and the remaining carcass will be submitted separately for analysis. Therefore, whole-body chemical concentrations for large fish will not be measured directly, but will be calculated from the fillet and carcass data. Several individuals of each biota type from each sampling zone will be composited to

satisfy sample volume requirements and to represent average exposure concentrations. All composite samples will be weighed in the field, including separate fillet and carcass fractions of the large fish.

Sediment samples from each of the eight sampling zones will be analyzed for PCB homologues, alkylated PAHs, pesticides, acid volatile sulfide / simultaneously extracted metals (AVS/SEM), total metals, TPH, total organic carbon (TOC), moisture content, and grain size. Sediment porewater will be analyzed for ammonia and dissolved arsenic and selenium. All biota tissue will be analyzed for PCB homologues, pesticides, selenium, lead, and lipids. Additionally, a subset of the sediment and biota tissue samples will be analyzed for PCB congeners, including one sample each of large fish fillets (game, pan, and rough) and three samples each of sediment, medium whole fish, and benthic macroinvertebrates. The sampling zones in Johnson Lake for PCB congener analyses will be randomly selected. Sediment samples will also be collected for site-specific sediment toxicity tests.

All sediment and biota samples, except samples for PCB homologue analysis, will be submitted to Columbia Analytical Services of Kelso, Washington. PCB homologue sediment and biota samples will be submitted to STL Savannah Laboratories of Savannah, Georgia. Toxicity tests will be performed by Springborn Laboratories in Wareham, Massachusetts. Information on analytical methods and target analytes is provided in Section 4 of this SAP.

2.2 Sampling Equipment

This section describes the equipment that will be used to record sample locations and to collect and contain samples. Ancillary equipment (e.g., coolers, disposable gloves, etc.) is discussed in connection with specific field procedures in Section 2.3 of this SAP. Sample containers will be supplied by the appropriate laboratories and will be certified as pre-cleaned.

Surficial sediment samples (0 – 6 inches) will be collected using a grab sampler (e.g. Ponar sampler or stainless steel/plastic scoops). Several sediment grab samples from within each sampling zone will be collected to satisfy total sample volume requirements. Sample containers are described in Table B-1 of the Quality Assurance Project Plan (QAPP) (Appendix B of Revision 2 of the Johnson Lake IWP).

Aquatic plants will be collected by hand or using a knife or scissors, as appropriate. Invertebrates will be collected using a dip or sweep net. Fish will be collected using

boat electrofishing equipment. Biota samples will be packaged in aluminum foil and plastic bags (see Section 2.3.2).

2.3 Sample Collection and Handling

New, disposable, non-talc gloves will be used during all sample collection and handling activities. Precise sampling locations will be determined by the field team prior to sample collection activities. Records of all field observations and sampling procedures will be maintained in both a field notebook and the appropriate ARCADIS sampling log sheets.

2.3.1 Sediment

Prior to sediment sampling, surface water measurements of pH, temperature, specific conductance, and dissolved oxygen will be recorded in the field. A multiparameter water quality meter will be used to take the field readings. The instrument will be calibrated according to the manufacturer's instructions each day prior to use.

Sediment sampling will be conducted by lowering the grab sampler through the water to the underlying sediment until refusal. The sampler will then be brought to the surface where any free water will be decanted. Multiple sediment grab samples from within each sampling zone will be collected to supply the required total sample volume for all analyses. Sediment grab samples will be placed in a stainless steel bowl and mixed using a stainless steel spoon until it is visually observed to be homogeneous; excessive mixing will be avoided to maximize sample integrity.

Sample containers (Table B-1 of the QAPP) will be packed as full as possible to reduce headspace. The sampler will use disposable gloves and stainless steel or plastic trowels or spoons to handle the sediment material. The sample container will be affixed with a label that lists the sample name, date, time, the initials of the sample collector, and the analytical requirements. The analytical sample containers will be placed in the sample cooler with packing material and bagged ice and will be held at 4° (± 2°) Celsius prior to and during shipment to the laboratories. A complete chain-of-custody form will be enclosed in each cooler and a custody seal will be affixed to each shipping container. The coolers will be shipped by overnight delivery to the selected laboratories for chemical analyses.

2.3.2 Biota

Several individuals of each biota type (plants, macroinvertebrates, and fish) from each sampling zone in Johnson Lake will be composited to satisfy sample volume requirements. The dominant species present in Johnson Lake for each biota type will be determined in the field, and substitutions may be needed in the absence of a targeted species or for insufficient biomass for analytical testing. The weight of the biota samples will be determined in the field to provide adequate samples for analysis, including the separate fillet and carcass fractions of the large fish. Habitat disturbance during sampling will be minimized.

During collection in each sampling zone, biota types from each zone will be placed into temporary holding containers until sampling for that zone is completed. A cooler filled with lake water at an ambient temperature will serve as a live well. When sampling is completed at each zone, individuals will be identified to the lowest taxonomic group possible. Sediment will be removed from each specimen by rinsing with organic-free deionized water (provided by the laboratories). Each specimen or group of individuals will be lightly blotted dry with paper towels and weighed; the sample or composite sample will be complete when the target weight is reached. Any individuals not used for this sampling effort will be released into the environment at the sampling zone from which the individual was collected.

Each sample will be wrapped in aluminum foil (dull side against sample) and placed into a small, plastic bag. The bag will be labeled with the sample name, date, time, the initials of the sample collector, and the analytical requirements (see Section 2.4). Once labeled, the plastic bag will be placed into a cooler with wet ice held at 4°C (±2°C) prior to and during shipment to the laboratory. Euthanasia of live samples will be primarily long-term cold/freezing exposure. A complete chain-of-custody form will be enclosed in each cooler and a custody seal will be affixed to each shipping container. The coolers will be shipped by overnight delivery to the selected laboratories for chemical analyses.

The following describes the specific collection methods to be used for each biota type listed.

- Aquatic plants – Individual plants will be collected from each of the six sampling zones around the perimeter of Johnson Lake. Only the above-ground portion of the plant will be submitted for analysis. Care will be taken to rinse all sediment from the plant tissue.

- Benthic macroinvertebrates – Crayfish, insect larvae, worms, and mollusks are expected to be the dominant macroinvertebrates in Johnson Lake and will be sampled from each of the six sampling zones around the perimeter of Johnson Lake. When possible, larger invertebrates (e.g., crayfish, mollusks) will be sampled preferentially. A triangular dip/sweep net will be used for sampling all available habitats (e.g., rocks, sand, snags, macrophytes, etc.) within each sampling zone. For each habitat, the collector will disturb the substrate and then repeatedly sweep through the area with the net to collect invertebrates that have been dislodged. In addition, the collector will examine large substrates (e.g., logs and boulders). Care will be taken to rinse all sediment from the tissue.

- Fish – Small and medium fish will be collected from each of the eight sampling zones in Johnson Lake. Game, pan, and rough fish will be collected on a lake-wide basis. Fish will be collected by boat electrofishing, which involves shocking the water and collecting the stunned fish by net. The electrofishing will be conducted at night, consistent with recommendations for fish sampling in large rivers (Simon and Sanders 1999). Individual fish will be composited as needed to satisfy sample volume requirements. Game, pan, and rough fish will be filleted in the field, and the remaining carcass will be submitted separately for analysis.

2.4 Sample Identification

The samples for this investigation will be assigned a unique, alphanumeric identifier, according to the method described in this section. The sample designation system will facilitate identification and tracking of each sample collected by ensuring that the identifier is unique and provides details for each sample (i.e., sampling location, medium sampled, and sample number). The use of the alphanumeric identifier will allow data associated with each sample to be entered into a facility-wide database system. The code will be as follows:

- The first two letters will identify the medium sampled (i.e., sediment (SD), aquatic plants (AP), benthic macroinvertebrates (BM), small fish (SF), medium fish (MF), game fish (GF), pan fish (PF), and rough fish (RF)); and

- The next digit will identify the sampling zone location in Johnson Lake (i.e., 1 through 8; see Figure A-1). For game, rough, and pan fish, which will be

collected on a lake-wide basis, samples will be numbered sequentially (i.e., 1 through 3).

For example, the small fish sample collected from sampling zone 5 will be labeled SF-5.

3. Field Methodologies

3.1 Health and Safety

The Health & Safety Plan (HASP) for the Johnson Lake site has been developed to include the work under this SAP. The HASP addresses the potential chemical and physical hazards present on-site and includes provisions for health and safety monitoring and emergency procedures. We anticipate that the work under this SAP will be conducted under modified Level D personal protective equipment (PPE). The HASP is located in Appendix C of the Johnson Lake IWP (Revision 2).

3.2 Quality Control Samples

Sampling quality assurance and quality control (QA/QC) will be ensured by carefully following the procedures as established in this SAP. Quality control samples will include matrix spike/matrix spike duplicate (MS/MSD) samples. Equipment rinseate blanks will be collected from re-useable sampling equipment only. However, the use of sampling equipment dedicated to each sampling zone and/or disposal sampling equipment will minimize the need to collect equipment rinseate blanks. MS/MSDs are typically collected at a rate of one per every twenty samples. If needed, equipment rinseate blanks are collected by pouring laboratory grade deionized water over a decontaminated piece of sampling equipment and catching the rinseate in a laboratory container.

The sample identification for the MS/MSD samples will consist of the appropriate code described in Section 2.4 followed by "MSMSD" (e.g., SD5-2MSMSD). The sample identification for equipment blanks will consist of "EB" followed by sequential numbers (i.e., EB01, EB02).

3.3 Decontamination

Decontamination procedures will include washing any reusable sampling equipment with a clean scrub brush and a mixture of deionized water and laboratory grade

detergent. The equipment will be rinsed twice with deionized water. Decontamination fluids will be collected for proper disposal. Decontaminated utensils will be kept in their appropriate equipment containers while not in use to reduce the potential for contamination. To minimize decontamination requirements and to prevent cross-contamination, disposable equipment will be used whenever possible. For specific decontamination procedures please refer to Appendix B (QAPP) of this Johnson Lake IWP (Revision 2).

3.4 Waste Management Procedures

Fluids generated during decontamination will be collected and placed in approved waste receptacles for disposal. Sediment and biota samples collected during sampling procedures not used in sample analyses will be placed back at the location from which they were taken.

3.5 Field Documentation

A field notebook will be maintained throughout the sampling effort and will contain the following information:

- Date;
- Personnel;
- Sampling zone;
- Time of sample collection;
- Description of sample characteristics, including color and odor (if evident) for sediment, and species and abnormalities (if evident) for biota;
- Weight of each biota sample;
- Documentation of photographs taken of site locations (i.e., roll and frame number or digital photograph identification); and
- Description of prior and current weather conditions.

4. Analytical and Toxicity Test Methods

Method detection limits and analytical standard operating procedures are provided in the QAPP (Appendix B of the Johnson Lake IWP (Revision 2)). The constituents of interest for this sampling event are PCBs, PAHs, two groups of pesticides (DDT and its

metabolites and endosulfans), eight metals (arsenic, cadmium, chromium, copper, lead, nickel, selenium, and zinc), and TPH. Porewater will be extracted from sediment using the method described by Hoffman (1998). The analytical method for each constituent is listed below:

- PCB homologues (sediment and biota) – U.S. EPA Method 680;
- PCB congeners (sediment and biota, selected samples) – U.S. EPA Method 8082;
- Alkylated PAHs (sediment) – U.S. EPA Method 8270C;
- Pesticides (sediment and biota) – U.S. EPA Method 8081;
- AVS/SEM metals (sediment) – Allen, Fu et al. 1992;
- Arsenic (sediment porewater (dissolved)) – U.S. EPA Method 6010/200.8;
- Selenium (biota and sediment porewater (dissolved)) – U.S. EPA Method 6010/200.8;
- Lead (biota) – U.S. EPA Method 6010/200.8;
- Total metals (sediment) – U.S. EPA Method 6010;
- TPH (sediment) – TPH D (C-10 to C-34);
- TOC (sediment) – ASTM D4129-82M;
- Moisture content (sediment) – ASTM D2216;
- Grain size (sediment) – ASTM D422/D1140;
- Ammonia (sediment porewater) – U.S. EPA Method 350.2;
- pH (porewater) – U.S. EPA Method 150.1; and
- Lipids (biota) – U.S. EPA Method OB1090.

Toxicity tests will be conducted using the following standard methods:

- Ten-day, whole-sediment toxicity tests with the amphipod *Hyalella azteca*, measuring survival (U.S. EPA 2000e); and
- Ten-day, whole-sediment toxicity tests with the midge *Chironomus tentans*, measuring survival and growth (U.S. EPA 2000e).

4.1 Quality Assurance

All sediment and biota samples, except samples for PCB homologue analysis, will be submitted to Columbia Analytical Services of Kelso, Washington. PCB homologue sediment and biota samples will be submitted to STL Savannah Laboratories in Savannah, Georgia. Toxicity tests will be performed by Springborn Laboratories in Wareham, Massachusetts. Analytical checks are performed in each laboratory. These procedures are based upon U.S. EPA analytical methods guidance and generally accepted standards of good laboratory practice. Key components of the laboratories' QA/QC Program are discussed in the Quality Assurance Manuals, located in Appendix B-1 of the QAPP.

5. Data Validation, Reporting and Evaluation

Data validation is a systematic procedure of reviewing a body of data against a set of known criteria to verify its validity prior to its intended use. All of the data developed from this investigation will be validated using Level II quality control guidelines that are part of the ARCADIS Analytical Quality Assurance and Laboratory Contract Program (AQA/LCP). Procedures used under this program were developed from the National Functional Guidelines for Organic and Inorganic Data Review (U.S. EPA 1999; 2002).

Validation procedures will include: verification of chain-of-custody documentation; comparison of blank values to sample values (equipment blanks, lab blanks, and method blanks); verification of sample analysis dates and preparation dates with respect to sample holding times; evaluation of data on MS/MSD samples; a review of field documentation (where necessary); summarizing and assigning validation codes to the sample data; and documentation of data that do not meet validation criteria.

6. Schedule

A schedule for the sampling activity, laboratory analyses, data reporting, data validation and receipt of analytical results is provided in the Project Management Plan (PMP), Appendix E of the Johnson Lake IWP (Revision 2).

7. References

Allen, H.E., G. Fu, and B. Deng. 1992. Analysis of acid-volatile sulfide (AVS) and simultaneously extracted metals (SEM) for the estimation of potential toxicity in aquatic sediments. Environmental Toxicology and Chemistry 12:1441-1453.

Hoffman, E. 1998. Tributylin analysis: clarification of interstitial water extraction and analysis methods – interim. DMMP Clarification Paper / SMS Technical Information Memorandum. U.S. Army Corps of Engineers.

Simon, T.P. and R.E. Sanders. 1999. Applying an index of biotic integrity based on great-river fish communities: Considerations in sampling and interpretation. In: Simon (ed.) Assessing the sustainability and biological integrity of water resources using fish communities. CRC Press, Boca Raton, Florida. pp. 475-505.

U.S. EPA. 1999. Contract Laboratory Program National Functional Guidelines for Organic Data Review. EPA 540/R-99/008.

U.S. EPA. 2002. Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. EPA 540/R-01-008.

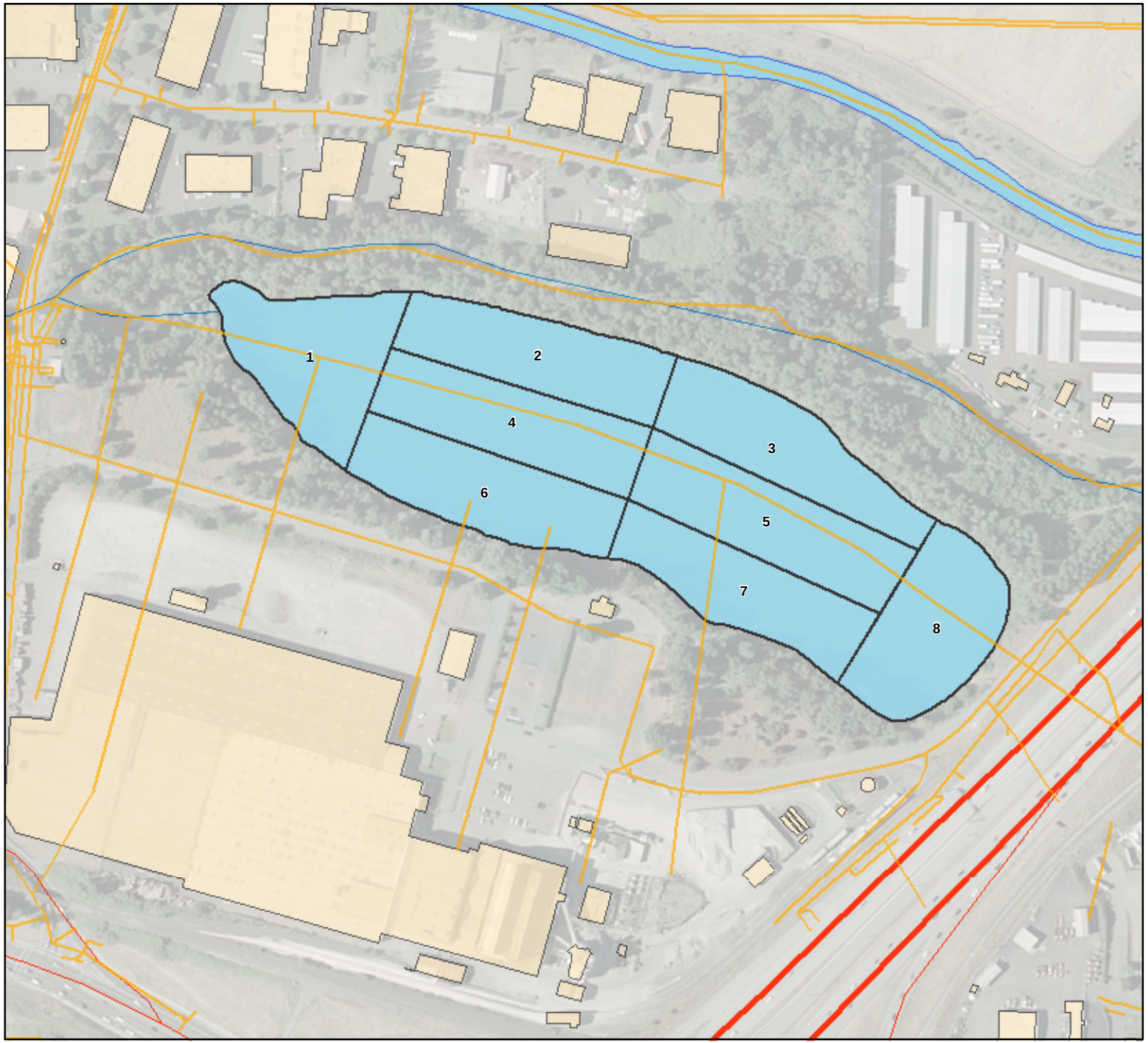


Figure A-1.
Proposed Sampling Zones
Johnson Lake
Portland, Oregon

- Proposed Sampling Grid
- Surface Water
- Building Footprints
- Arterial Streets
- Freeway
- Sewer/Stormwater Lines

Basedata from METRO DRC 1"=100' orthophotos, City of Portland, and US Census Bureau 2000 Tiger Files. Positions may vary in accuracy due to scale differences between the data sets. Spatial information is meant for general reference only.

Lambert Conformal Conic projection, North American Datum 1983 HARN, Local coordinate grid State Plane Oregon (North) FIPS 3601.

Cartography by ARCADIS - Greenville, SC

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Appendix B

Quality Assurance Project Plan (QAPP)

Appendix B
Johnson Lake Investigation Quality Assurance Project Plan (QAPP)

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Infrastructure, buildings, environment, communications

Owens-Brockway Glass Container, Inc.,
Portland, Oregon

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Table of Contents

1. Project Description 1

1.1 Introduction 1

1.2 Sampling and Analysis Plan 1

2. Project Management 2

2.1 Project Organization and Responsibility 2

2.1.1 Laboratory Responsibilities 2

2.2 Field Responsibilities 3

3. Quality Assurance Objectives 3

3.1 Precision 4

3.2 Accuracy 4

3.2.1 Completeness 5

3.3 Representativeness 5

3.4 Comparability 6

4. Special Training and Certification 6

5. Sampling Procedures 7

5.1 Sample Containers, Preservatives, and Volume Requirements 7

5.2 Field Equipment Decontamination 7

5.3 Sample Packaging and Shipping Procedures 8

5.4 Field Records 8

5.5 Sample Identification 8

6. Custody Procedures 9

6.1 Sample Container Custody 9

6.2 Cooler Temperature Indicator 10

7. Calibration Procedures and Frequency 11

7.1 Field Instrument Calibration 11

7.2 Laboratory Instrument Calibration 11

8. Laboratory Procedures 11

9. Internal Quality Control Checks 12

9.1 Field Quality Control Samples 12

9.1.1 Duplicate Samples 12

9.1.2 Equipment Rinseate Blank Samples 12

9.1.3 Matrix Spikes and Matrix Spike Duplicates 13

9.1.4 Field Quality Control Sample Frequency 13

9.2 Laboratory Quality Control 13

9.2.1 Laboratory Internal Quality Assurance/Quality Control Program 13

10. Corrective Action 14

11. Data Reduction, Validation and Reporting 14

11.1 Data Reduction 14

11.2 Reporting Requirements 15

11.3 Data Validation 16

12. Data Management 18

13. References 18

Tables

B-1 Target Compounds, Analytical Methods, and Sampling Information, Owens-Brockway, Portland, Oregon

B-2 Laboratory-Specific Precision and Accuracy Control Limits for Analytical Parameters, Owens-Brockway, Portland, Oregon

B-3 Project Personnel and Responsibilities, Owens-Brockway, Portland, Oregon

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Appendices

B-1 Selected Analytical Laboratory Standard Operating Procedures

B-2 ARCADIS Standard Operating Procedures

B-3 Analytical Laboratory Quality Assurance Manuals

B-4 Level II Data Validation Checklist

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Appendix B
Johnson Lake Investigation Quality Assurance Project Plan (QAPP)
Owens-Brockway, Portland, OR

1. Project Description

At the request of Owens Brockway Glass Container, Inc., ARCADIS prepared this Quality Assurance Project Plan (QAPP). The purpose of a QAPP is to provide a description of the policy, organization, functional activities, and quality control protocols for the study objectives. The study objectives are further defined in Revision 2 of the Johnson Lake Investigation Work Plan (IWP). The QAPP provides consistent methodology for collection and analysis of sediment and biota samples from Johnson Lake, Portland, Oregon. The site location is presented in Figure 1-1 of the Johnson Lake IWP (Revision 2).

1.1 Introduction

The purpose of the investigation to be conducted in accordance with this QAPP is to determine the impact of chemicals of interest to the surrounding environment. Comprehensive and well-defined procedures for project management, quality assurance and quality control (QA/QC), and documentation are instrumental in the execution of a successful field effort and generation of high-quality data.

1.2 Sampling and Analysis Plan

The objective of the field sampling effort is to collect sediment and biota samples from Johnson Lake to assist in the characterization of conditions at the lake. As described in the Sampling and Analysis Plan (SAP) (Appendix A of the Johnson Lake IWP (Revision 2)), ARCADIS will be sampling sediment and biota from Johnson Lake for the following analytes: sediment will be sampled for polychlorinated biphenyls (PCBs), alkylated polycyclic aromatic hydrocarbons (PAHs), pesticides, acid volatile sulfide / simultaneously extracted metals (AVS/SEM), total metals, total petroleum hydrocarbons (TPH), total organic carbon (TOC), moisture content, and grain size. Sediment porewater will be analyzed for ammonia, pH, and dissolved arsenic and selenium. Biota tissue will be sampled for PCBs, pesticides, selenium, lead, and lipid content. These analytes are selected based on previous assessments of the study area as described in Section 2 of the Johnson Lake IWP (Revision 2). In addition, 10-day sediment toxicity tests will be conducted measuring *Hyalella azteca* survival and *Chironomus tentans* growth.

Laboratory analytical methods, reporting limits, and constituent-specific sampling information for samples to be collected by ARCADIS are provided in Tables B-1 and B-2. Laboratory standard operating procedures (SOP) are provided in Appendix B-1.

All sediment and biota samples, except PCB-homologues, will be submitted to Columbia Analytical Services of Kelso, Washington. PCB-homologue sediment and biota samples will be submitted to STL Savannah Laboratories of Savannah, Georgia. Toxicity tests will be performed by Springborn Laboratories of Wareham, Massachusetts.

2. Project Management

This section of the QAPP includes descriptions of the project management structure and procedures that relate to project quality assurance.

2.1 Project Organization and Responsibility

ARCADIS has responsibility for all phases of the investigation as described in Revision 2 of the Johnson Lake IWP. ARCADIS will perform the field investigation, prepare the report, and perform any subsequent studies. ARCADIS will provide project management, and specific individuals will be designated upon implementation of the project. Table B-3 identifies the personnel responsible for planning and implementing field and laboratory operations and QA/QC procedures for this project and describes each individual's tasks for project management and quality assurance.

2.1.1 Laboratory Responsibilities

The laboratories listed below are responsible for the analyses of samples. The responsibilities of each laboratory Project Director, Technical Director, and Quality Assurance Manager are provided in Table B-3.

The laboratory tasked with the responsibility for analysis of chemicals of interest in sediment, sediment porewater, and biota samples is:

Columbia Analytical Services
1317 South 13th Avenue
Kelso, Washington 98626
Phone: 360.577.7222

The laboratory tasked with the responsibility for analysis of PCB-homologues in sediment and biota samples is:

STL Savannah Laboratories
5102 LaRoche Avenue
Savannah, Georgia 31404-6019
Phone: 912.354.7858

The laboratory tasked with responsibility for toxicity testing is:

Springborn Smithers Laboratories
790 Main Street
Wareham, Massachusetts 02571
Phone: 508.295.2550

2.2 Field Responsibilities

The technical staff for this project will be drawn from ARCADIS' pool of corporate resources. The technical staff will be utilized to gather and analyze data and to prepare various task reports and support materials. All of the designated technical team members are experienced professionals who possess the degree of specialization and technical competence to effectively and efficiently perform the required work**.**

3. Quality Assurance Objectives

The overall quality assurance objective for this project is to develop and implement procedures for field sampling, laboratory analysis, chain-of-custody, and reporting that will provide results that are of known and acceptable quality and are representative of the conditions present at the site. This section will provide in greater detail specific project objectives and intended data usages mentioned in Section 1 of this QAPP. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal QC, preventive maintenance of field equipment, field sampling SOPs, field equipment, and calibration are described in other sections of this QAPP. Table B-2 details laboratory analytical methods and QA/QC objectives, including method detection limits (MDL), relative percent difference (RPD), accuracy (percent recovery [%R]), and precision (%RPD).

3.1 Precision

Precision is the degree of mutual agreement of independent measurements as a result of repeated application of a process under specific conditions. Precision is concerned with the closeness of results during repeated analysis of the same sample. Precision is addressed by calculating the RPD for each pair of duplicate analyses using the following equation:

$$\%RPD = \frac{(S - D)}{(S + D) / 2} \times 100$$

where:

S = First sample value; and

D = Second sample value.

Precision objectives for each analytical method in terms of the RPD of duplicate measurements have been specified for laboratory measurements.

The following QC samples may be used to evaluate precision in the laboratory:

- Laboratory duplicates,
- Reagent water blank spike duplicates, or
- Matrix spike duplicates.

The following QC samples may be used to evaluate precision in the field:

- Field duplicates (which are also used to determine precision for laboratory analyses). Field duplicates will be collected if future investigations specify the collection of aqueous samples. Sediment and biota exhibit sufficient spatial heterogeneity that field duplicates do not provide a useful indicator of precision for these media.

3.2 Accuracy

Accuracy is the degree of agreement of a measured value with the true or expected value. Accuracy objectives for each analytical method in terms of the percent recovery

(%R) from analysis of samples of known analyte concentration are presented in the laboratory QAPP. Accuracy is assessed by using the following equation:

$$\%R = \frac{A - B}{C} \times 100$$

where:

A = The analyte concentration determined experimentally from the spiked sample;

B = The background level determined by a separate analysis of the unspiked sample; and

C = The amount of the spike added.

The following QC samples will be analyzed to evaluate accuracy in laboratory measurements:

- Laboratory control samples,
- Matrix spikes, or
- Reagent water blank spikes.

3.2.1 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that is expected under the conditions of the measurement. At least 95 percent of all samples analyzed by the laboratory must be valid to be considered complete. Completeness is assessed using the following equation:

$$C = \frac{Valid\ Data\ Obtained}{Total\ Data\ Planned} \times 100$$

3.3 Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a

process condition, or an environmental condition. As such, representativeness describes whether samples collected, or the aliquot selected by the laboratory for analysis, are sufficient in number, type, location, frequency, and size to be characteristic of the substance analyzed.

Representativeness is a qualitative parameter that is dependent upon the proper design of the sampling program and proper laboratory protocol. The sampling network for this project was designed to provide data representative of site conditions. Representativeness will be met by ensuring that work specifications are followed, proper sampling techniques are used, and proper analytical procedures are followed.

3.4 Comparability

Comparability expresses the confidence with which one data set can be compared with another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedure used to obtain the planned analytical data, as documented in the sampling SOP and the laboratory QAPP, are expected to provide comparable data. However, it must be recognized that for different data sets to be truly comparable, all aspects of the collection and measurement process should be identical. Some of the parameters that should be consistent to ensure comparability include the following items:

- Samples must be from the same source;
- Samples must be collected in the same or similar manner;
- Samples must be preserved in the same manner;
- Samples must be prepared and analyzed by the same method;
- Data must be reported in the same units; and
- Data from each set must be of comparable quality.

4. Special Training and Certification

Procedures to be completed for this study are, for the most part, routine. Standard procedures will be used to collect sediment and tissue samples and to complete laboratory analyses (see Appendix A (SAP) of the Johnson Lake IWP (Revision 2)).

All field personnel will have completed the 40-hour Hazardous Waste Operations and Emergency Response training with annual refresher courses as required by the Occupational Safety and Health Administration (OSHA).

Quality control must be a continuous process in fish bioassessment and fish collection. Field fish identifications will be conducted by a qualified/trained fish taxonomist, familiar with local and regional ichthyofauna.

5. Sampling Procedures

This section presents the field data collection methods and procedures to be implemented during the planned investigation. The procedures to be used during the investigation were chosen for their ability to provide data representative of site conditions, compatibility with analytical considerations, practicality, and simplicity. The SAP is found in Appendix A of the Johnson Lake IWP (Revision 2). ARCADIS SOPs pertaining to this sampling event are provided in Appendix B-2.

5.1 Sample Containers, Preservatives, and Volume Requirements

The required sample containers, preservatives, and volumes for analyses of target compounds will be collected in accordance with the respective laboratory QAPP. The volume of sample collected must be sufficient to perform the required analyses with an additional amount for QC needs (matrix spikes, duplicates, etc.). All sample containers used for the collection of samples will be pre-cleaned by the manufacturer or by the laboratory supplying the sample bottles, as appropriate. Refer to Table B-1 for a list of target compounds, laboratory analytical methods, sample containers, and holding times.

5.2 Field Equipment Decontamination

Equipment decontamination procedures are provided in the SAP (Appendix A the Johnson Lake IWP (Revision 2)) and SOPs (Appendix B-2). The laboratory detergent used for equipment decontamination will be a standard brand of phosphorus-free laboratory detergent. The laboratory detergent used should not contain the analytes of concern. Distilled or deionized water will be used during cleaning procedures for field equipment. Laboratory detergent and rinse waters used to clean equipment shall not be reused.

Non-dedicated sampling equipment (stainless steel bowls, spoons, scoops, spades, augers, etc.) will be decontaminated using the appropriate procedures included in each sampling plan. All sampling equipment decontamination will be performed in the field. When possible, equipment will be decontaminated in batches at a central staging area. All non-disposable equipment will be decontaminated prior to use, following use, between each sampling location, and prior to storage.

5.3 Sample Packaging and Shipping Procedures

Samples will be maintained on ice at 4°C (± 2°C) under proper chain-of-custody until shipped via overnight delivery by common carrier or delivered in person to the laboratory. Aqueous samples will be shipped to the laboratory on the day they are collected.

All samples will be packed in ice chests along with proper chain-of-custody forms. All coolers will be sealed with packing tape and custody seals will be attached. Glass sample bottles will be wrapped in plastic bubble pack for protection from breakage. Other standard packing materials may also be used. Sample containers will be arranged to minimize movement when shipping to prevent contact between containers.

5.4 Field Records

All field activities will be recorded in the field at the time of the activity. Several different types of records may be created during the sampling event. All records will be prepared in permanent ink and will be dated and signed by the author, and any errors will be corrected by drawing a single line through the error, initialing, and dating the error. All records will be maintained in the project files as a permanent record.

Project field activities will be documented on daily work description logs. Standard ARCADIS log sheets for sampling will be completed, as appropriate, for each sample collected. These logs will be completed as samples are collected. Field notes will be signed at the end of each day. All information will be recorded in indelible ink.

5.5 Sample Identification

Sample labels are necessary to identify the samples. The labels shall be affixed to the sample container (not the caps) prior to or at the time of sampling. Pertinent information on the labels shall be filled out in waterproof ink at the time of collection and will include the following information:

- Sample number/identification code;
- Name (initials) of collector;
- Date and time of collection;
- Project title and/or project number;
- ARCADIS;
- Required analyses;
- Analysis method;
- Sample matrix; and
- Preservative.

After collection, all samples will be handled as little as possible. Field personnel will use extreme care to maintain the integrity of the samples. If samples are placed in a cooler with ice, personnel will package the ice so that melted ice will not leak into the sample containers or cause cross contamination.

6. Custody Procedures

Sample custody is a vital aspect of sampling procedures. The samples must be traceable from the time of sample collection, through sample analysis, data generation, and report preparation.

6.1 Sample Container Custody

All containers will be shipped from the supplier to the ARCADIS office or a location designated by the project manager (PM) by common carrier in sealed containers. The supplier will include a shipping form or laboratory chain-of-custody listing all containers shipped and the purpose of each container. It is ARCADIS policy that all containers are considered to be in the custody of the supplier until the sample containers are received by ARCADIS. Upon receipt, sample containers will be checked to determine if any breakage occurred during shipment. The containers will be maintained in the custody of the receiver in a clean, secure area until they are used for

sample collection. Once samples have been collected, they will be maintained in the custody of the sampling personnel. The procedure for maintaining and documenting chain-of-custody in the field will be as follows:

1. The sample label that is affixed to the container will be inspected to confirm that all of the required information has been provided.

2. The sample container will be sealed in a plastic bag or wrapped in bubble pack and packed in a cooler chest in a manner that will minimize movement. All samples will be cooled with wet ice.

3. For each cooler chest that is sent to the laboratory, a Chain-of-Custody Record will be completed. All information on the Chain-of-Custody Record and the sample container labels will be checked against field records and the samples will be recounted.

4. If a commercial courier service (e.g., FedEx) or lab courier is used to transport the samples to the laboratory, the Chain-of-Custody Record will be signed by a member of the field team and a copy of the Record will be retained by the field team. The remaining copy(ies) of the Record will be sealed in a plastic bag and placed in the cooler chest with the samples. The cooler chest will be sealed with packaging tape and two custody seals that have been signed and dated by a member of the field team. Lab couriers will sign the chain-of-custody prior to sealing it in the cooler chest; however, because commercial couriers will not sign chain-of-custody forms, the package routing documentation maintained by the courier service during shipping serves as chain-of-custody documentation for the samples.

For more specific discussion of custody procedures please refer to the SOP located in Appendix B-2. All laboratory analysts and technicians must follow chain-of-custody protocols described in the respective laboratory's QA Manual, which is provided in Appendix B-3.

6.2 Cooler Temperature Indicator

A temperature blank will be included in each cooler containing samples being sent for analysis to verify that the samples have been maintained at 4°C. The temperature blank will consist of a sample container filled with unpreserved water (potable or distilled), labeled "EPA Cooler Temperature Indicator" and dated. The temperature of

the blank will be taken and recorded on the chain of custody immediately upon receipt at the laboratory, prior to inventory and refrigeration.

7. Calibration Procedures and Frequency

This section describes procedures for calibrating all field and laboratory instruments used for conducting field measurements and laboratory analyses.

7.1 Field Instrument Calibration

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications and the pertinent SOP in Appendix B-1.

Equipment to be used during the field sampling will be examined prior to conducting the field investigation to certify that it is in operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to ensure that all maintenance requirements are being observed.

7.2 Laboratory Instrument Calibration

The calibration procedures and calibration frequency employed by the laboratory will be in accordance with the analytical methods specified in the respective analytical method SOPs as described in the laboratory's QA Manuals supplied in Appendix B-3. Records of instrument calibration, repairs, and replacement activities will be maintained. These records will be filed at the location where the work is performed and will be subject to QA audit at any time.

8. Laboratory Procedures

The laboratories (including the laboratory address and phone number) designated to perform sample analyses are provided in Section 2.1 of this QAPP. Analytical methods (SOP) for each parameter to be analyzed by the laboratories are presented in Appendix B-1.

Detection and reporting limits have been specified for soil according to the analytical methodology and are provided in Table B-2. All QA/QC procedures specified in each

method will be followed and control limits will be met or corrective action will be taken as described in the methods.

The actual reporting limits attained during this site investigation may be elevated with respect to target reporting limits if interferences are encountered from the sample matrices. Sample clean-up procedures will be used as necessary to minimize interferences and optimize detection limits.

9. Internal Quality Control Checks

9.1 Field Quality Control Samples

Field QC samples are necessary to monitor both field and laboratory performance. Field QC samples provide a means of checking the validity of the sample results. These data will be periodically examined to determine if any problems are evident with specific types of media samples or with the procedures used by the subcontracted laboratory. Field QC samples to be collected during sampling activities may include duplicate samples, equipment rinseate blanks, and matrix spikes and matrix spike duplicates (MS/MSDs).

9.1.1 Duplicate Samples

The precision of aqueous sample collection techniques (if any) will be monitored through the collection and analyses of field duplicate samples. This type of sample also may serve as a rough estimate of the laboratory analytical precision based on the sample homogeneity. Duplicate samples are collected by equally dividing the sample removed from a sample source, then placing the aliquots into separate, but identical, sample containers. This process will be repeated until each sample container is properly filled, thus resulting in two samples. Duplicate samples will be designated on the chain of custody.

9.1.2 Equipment Rinseate Blank Samples

The effectiveness of personnel sample-handling techniques may be monitored by submitting preserved equipment rinseate blank samples for laboratory analysis. These blank samples will be prepared by field personnel and treated in the same manner as actual samples. Equipment blanks are collected from pouring distilled water so that contact with the field sampling apparatus (grab sampler, spoons or bowls etc.) is made after it has been decontaminated. That water is collected in appropriate sample

containers, preserved in the same manner as the field samples, and analyzed for the parameters/analytes of investigation. Appropriate sample containers for each analyte group must be used. These blanks will be appropriately labeled and documented in field records and will be stored, transported, and analyzed with the samples. Equipment blanks will be designated on the chain of custody. Equipment blanks will be collected only for non-disposable sampling equipment.

9.1.3 Matrix Spikes and Matrix Spike Duplicates

MS/MSDs provide information about the effect of the sample matrix on the digestion and measurement methodology. All matrix spikes are performed in duplicate and therefore are referred to as matrix spike/matrix spike duplicate samples. A MS is an aliquot of sample spiked with known concentrations of specific compounds. The spiking occurs prior to sample preparation and analysis. The MS and MSD samples will be designated on the chain of custody form. Sediment and biota MS/MSD samples require the same volume as normal samples.

9.1.4 Field Quality Control Sample Frequency

If field duplicates are required (i.e., for any aqueous samples), one field duplicate sample will be taken for every 10 samples submitted to the laboratory. If equipment blanks are required (i.e., for non-disposable equipment), one equipment rinseate blank will be collected per matrix per day or per 20 samples per matrix, whichever is more frequent. One MS/MSD will be collected for every 20 investigative samples per sample matrix.

9.2 Laboratory Quality Control

Laboratories are required to demonstrate their ability to produce acceptable results using the methods selected by the generation of acceptable QC data. Analytical data is evaluated by the laboratory prior to submittal based on internal reviews of the QC data. Internal laboratory QC procedures used to assess laboratory performance are specified in the respective laboratory SOPs, listed in their QAPP.

9.2.1 Laboratory Internal Quality Assurance/Quality Control Program

Analytical QC checks are performed in each laboratory. These procedures are based upon United States Environmental Protection Agency (U.S. EPA) analytical methods guidance and generally accepted standards of good laboratory practice. Key

components of the laboratory's QA/QC Program are discussed in the laboratory QA Manuals (Appendix B-1).

10. Corrective Action

Corrective actions may be required for two classes of problems: analytical and equipment problems and noncompliance problems. Analytical and equipment problems may occur during sampling, sample handling, sample preparation, laboratory instrumental analysis, and data review. Noncompliance problems occur when activities are conducted outside the requirements of the QAPP. For both types of problems, a formal corrective action program will be implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the PM who will then contact the project managers/coordinators of the involved parties, if necessary. PMs responsible for implementing corrective action at the field, laboratory, and data assessment stages of the project are listed in Table B-3. If the problem is analytical in nature, information on these problems will be promptly communicated to the appropriate laboratory PM(s).

11. Data Reduction, Validation and Reporting

Data measured in the field will be recorded in appropriate field logs. Field personnel will submit all field notes and field logs to the PM at the end of each workweek. These documents include daily logs, any sampling logs, and laboratory chain of custody documents. These documents will be reviewed by the PM to ensure that proper procedures are being implemented during data collection activities.

11.1 Data Reduction

Data reduction is the process of converting analytical data from electronic form or instrument form into digital form and correcting for all sample weight, dilution factors, and percent solids calculations that may be applicable. This process is performed in the field and in the laboratory.

Raw data from field measurements and sample collection activities are recorded in the field records and on applicable field log sheets. Instrument readings are recorded directly into the field records. Recorded field data will be checked by the PM or designee during data validation of field data.

Raw data from analytical laboratory measurements may or may not be generated by digital read out instruments. In addition, due to dilution and concentration factors often developed during the analysis of the sample, careful data reduction is required to convert raw data to useable digital analytical data. All data reductions will be performed according to procedures presented in the laboratory QAPP.

11.2 Reporting Requirements

For analytical data quality to be adequately assessed, it is imperative that expected reporting levels are communicated to the laboratory. The laboratory must submit, in addition to the analytical results, all supporting QC data required for effective validation of the analytical data. If increased defensibility of the laboratory report data is required, additional documentation of analytical QC data should be available upon request to the laboratories to support validation conclusions and data usability determinations.

Laboratory data reports will consist of sample results plus the QA/QC data specified below. The following are general requirements for each sample analyzed by the laboratory:

- The results of each analysis;
- The list of the parameters of investigation;
- The method of analysis and the detection limit for each analyte;
- Dates of sample collection, receipt, preparation/extraction, and analysis;
- Copy of the chain-of-custody forms signed by the laboratories' sample custodian;
- A narrative summarizing any QA/QC deviances and the corrective action taken; and
- A master list relating laboratory ID to sample ID.

The list below describes the information to be provided for all analyses (where applicable):

- Results of laboratories' method blank, trip blank, water blank, and/or extraction blank samples;
- Results of the batch specific laboratories' duplicate analysis; or
- Results of analysis of a batch specific matrix spike or reagent water spike with the compound or element of investigation, the expected value, percent recovery, control limits, and source of control limits.
- Results of MS/MSD, control limits, expected value, RPD, and %R; and
- Hard copy data deliverables should be generated at the time of analyses.

11.3 Data Validation

Data validation is a systematic process for reviewing a body of assembled analytical results against a set of criteria to determine if the data is adequate for its intended use. Data validation involves: 1) data editing; 2) data screening and checking; 3) data auditing and verification; 4) data certification; and 5) final data review and informed decision-making whereby data are accepted, qualified, or rejected.

The data validation procedures employed for this project will include an evaluation of the field data package and an evaluation of the laboratory's analytical data packages. The field data package validation procedure will include but not be limited to the following tasks:

- A review of field data contained in sampling logs for accuracy and completeness;
- A verification that samples were properly prepared, preserved, and identified;
- A check on field analyses for equipment calibration and instrument conditions; and
- A review of each laboratory's task orders (if needed) and chain-of-custody forms for proper completion, signatures of field personnel, and the laboratory sample custodian and dates.

All of the analytical data will be validated according to criteria specified in the functional guidelines for data validation (U.S. EPA 1999; 2002). Data may be qualified as estimated or rejected if quality control samples and procedures do not meet

control limits, as described in the functional guidelines. Any non-conformance issues identified during the laboratory's quality assurance checks will be corrected and noted by the laboratory. Close contact will be maintained between the project QA/QC coordinator and the laboratory PM so that any quality issues can be resolved in a timely manner. Any data quality deviations will be discussed in the laboratory case narrative, including the direction and magnitude of any bias to the data, if possible.

Data validation and verification will be completed by ARCADIS prior to finalization of the data and release of the data set for interpretation. Chemical data will be validated according to U.S. EPA Level II criteria, and a Level II data validation checklist will be completed to verify that the laboratories have performed in accordance with requirements specified in this QAPP and the respective laboratories SOPs. The checklist is provided in Appendix B-4.

Validation will include a systematic review of the data for compliance with the established QC criteria for the methods used, which may be modified as appropriate by the requirements specified in the current U.S. EPA *Functional Guidelines for Organics and Inorganics Analyses* (U.S. EPA 1999; 2002 respectively). These guidelines address analytical procedures required in the Contract Laboratories Program national guidelines for data review and will be modified as appropriate to accommodate method-specific quality control requirements. When laboratory-developed control limits are not available, data qualifiers will be applied when the quality control results do not meet quality objectives. Data will be qualified as estimated when results do not meet control limits developed by the laboratory. Project data will be released, in a hard copy and a digital format, for interpretation only after validation has been completed and all qualifiers have been entered into the database.

The goal of data validation is to determine the quality of each data point and to identify data that do not meet the project quality objectives. Nonconforming data may be qualified as estimated or rejected as unusable during data validation if criteria for data quality are not met. Rejected data will not be used for any purpose. An explanation of the rejected data will be included in the data validation report.

Data qualified as estimated (J) will be used for data less precise or less accurate than unqualified data. The data users and PMs are responsible for assessing the effect of the inaccuracy or imprecision of the qualified data on statistical procedures and other data uses for the study.

12. Data Management

Computerized systems will be used to record, store, and sort the technical data that will be generated to support the study. Automated procedures will be used by the laboratories to capture and summarize analytical results. Electronic data files will be imported directly from the laboratory to the project database, minimizing both data entry effort and opportunities for error. Sampling location coordinates will be entered into the database to enable computer-generation of maps and figures.

Field logbooks, station/sample forms, and chain-of-custody/sample analysis request forms will be prepared by the field team while sample collection activities are in progress. Sample information from the field will be entered manually into the database. Each data record will include a unique sample code, station ID, sample type (matrix), analyte, analyte concentration, and concentration units. Electronic data summaries will be produced to support data validation procedures. Data qualifiers will be entered into the database when validation is completed and verified, and the data set is approved as final. All manual and electronic entries will be verified by field personnel, the data manager, or validation personnel.

Project data tables and reports are prepared using customized retrievals that filter and sort the data according to criteria specified by the user. The data are automatically formatted for direct use with statistics software packages and various types of geographic information system software. The maintenance of a single, authoritative database prevents the proliferation of multiple versions of data and the introduction and propagation of errors.

13. References

U.S. EPA. 1999. Contract Laboratory Program National Functional Guidelines for Organic Data Review. EPA 540/R-99/008. U.S. Environmental Protection Agency.

U.S. EPA. 2002. Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. EPA 540/R-01-008.

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Table B-1. Target Compounds, Analytical Methods, and Sampling Information, Owens-Brockway, Portland, Oregon

Target Compounds	Analytical Method	Containers, Preservatives, Holding Times Sediment	Tissue	Laboratory
PCB-Homologues	USEPA 680	250 mL glass jar 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	12 g wrapped in foil 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	STL Savannah Laboratories
PCB-Congeners	USEPA 8082	250 mL glass jar 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	12 g wrapped in foil 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	Columbia Analytical Services
Alkylated PAHs	USEPA 8270c	500 mL glass jar 4 degrees Celcius HT 14 days/ 40 days	NA	Columbia Analytical Services
Organochlorine Pesticides	USEPA 8081 (low level reporting limits)	250 mL glass jar 4 degrees Celcius HT 7 days/ 40 days	NA	Columbia Analytical Services
Organochlorine Pesticides	USEPA 8081 (low level reporting limits)	NA	12 g wrapped in foil (same sample as PCB-homologues) 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	Columbia Analytical Services
Total Metals	USEPA 6010	250 mL glass jar 4 degrees Celcius HT 6 months	NA	Columbia Analytical Services
Lead	USEPA 6010/200.8	NA	12 g wrapped in foil (same sample as PCB-homologues) 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	Columbia Analytical Services
Selenium	USEPA 7742	NA	12 g wrapped in foil (same sample as PCB-homologues) 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	Columbia Analytical Services
SEM/AVS[a]	Allen, Fu et al. 1992	500 mL widemouth glass jar, no headspace 4 degrees Celcius HT 14 days	NA	Columbia Analytical Services
Total Organic Carbon (TOC)	ASTM D4129-82M (sediment)	500 mL glass jar (same jar as alkylated PAHs) 4 degrees Celcius HT 14 days	NA	Columbia Analytical Services

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Table B-1. Target Compounds, Analytical Methods, and Sampling Information, Owens-Brockway, Portland, Oregon

Target Compounds	Analytical Method	Containers, Preservatives, Holding Times Sediment	Tissue	Laboratory
Total Petroleum Hydrocarbons (TPH)	TPH D (C-10 to C-34)	500 mL glass jar (same jar as alkylated PAHs) 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	NA	Columbia Analytical Services
Grain Size	ASTM D422	500 mL glass jar NA NA	NA	Columbia Analytical Services
Lipids	USEPA OB1090	NA	12 g wrapped in foil (same sample as PCB-homologues) 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	Columbia Analytical Services
Moisture Content	ASTM D2216	500 mL plastic NA HT 7 days	NA	Columbia Analytical Services
Porewater Extraction[b]	Hoffman 1998	2 Liters plastic 4 degrees Celcius HT 14 days	NA	Columbia Analyical Services
10-Day Toxicity Test w/ *Hyalella azteca*	USEPA 100.1	3.5 Liters 4 degrees Celcius HT 14 days	NA	Springborn Smithers Laboratories
10-Day Toxicity Test w/ *Chironomus tentans*	USEPA 100.2	3.5 Liters 4 degrees Celcius HT 14 days	NA	Springborn Smithers Laboratories

a. SEM metals = cadmium, copper, lead, nickel, silver, and zinc
b. The following analyses will be performed on sediment porewater: dissolved arsenic (USEPA 200.8), dissolved selenium (USEPA 200.8), ammonia (USEPA 350.2), and porewater pH (USEPA 150.1).

ASTM - American Society for Testing and Materials
AVS - acid volatile sulfide
g - grams
HT - holding time to extraction/holding time for the extract preparation
M - modified
mL - milliliters
NA - not applicable/available
PAHs - polycyclic aromatic hydrocarbons
PCBs - polychlorinated biphenyls
SEM - simultaneously extracted metals
USEPA - United States Environmental Protection Agency

References:

Allen, H.E., G. Fu, and B. Deng. 1992. Analysis of acid-volatile sulfide (AVS) and simultaneously extracted metals (SEM) for the estimation of potential toxicity in aquatic sediments. Environmental Toxicology and Chemistry 12:1441-1453.

Hoffman, E. 1998. Tributyltin analysis: Clarification of interstitial water extraction and analysis methods – interim. DMMP clarification paper/SMS Technical information memorandum. U.S. Army Corps of Engineers.

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Table B-2. Laboratory- Specific Precision and Accuracy Control Limits for Analytical Parameters, Owens-Brockway, Portland, Oregon

Parameters	Analytical Methods	MDL	RL	Units	Accuracy % Recovery	Precision % RPD
Sediment						
PCB-Homologues						
Monochlorobiphenyl	USEPA 680	0.68	3.3	µg/kg	30-130	50
Dichlorobiphenyl	USEPA 680	0.76	3.3	µg/kg	30-130	50
Trichlorobiphenyl	USEPA 680	0.68	3.3	µg/kg	30-130	50
Tetrachlorobiphenyl	USEPA 680	1.3	6.7	µg/kg	40-140	50
Pentachlorobiphenyl	USEPA 680	0.83	6.7	µg/kg	40-140	50
Hexachlorobiphenyl	USEPA 680	0.89	6.7	µg/kg	40-140	50
Heptachlorobiphenyl	USEPA 680	1.6	10	µg/kg	40-140	50
Octachlorobiphenyl	USEPA 680	0.95	10	µg/kg	40-140	50
Nonachlorobiphenyl	USEPA 680	1.9	17	µg/kg	30-130	50
Decachlorobiphenyl	USEPA 680	1.9	17	µg/kg	30-130	50
PCB Congeners						
PCB 77	USEPA 8082	0.05	0.5	µg/kg, dry wt.	60-130	40
PCB 81	USEPA 8082	0.1	0.5	µg/kg, dry wt.	60-130	40
PCB 105	USEPA 8082	0.09	0.5	µg/kg, dry wt.	60-130	40
PCB 114	USEPA 8082	0.3	0.7	µg/kg, dry wt.	60-130	40
PCB 118	USEPA 8082	0.04	0.5	µg/kg, dry wt.	60-130	40
PCB 123	USEPA 8082	0.08	0.5	µg/kg, dry wt.	60-130	40
PCB 126	USEPA 8082	0.1	0.5	µg/kg, dry wt.	60-130	40
PCB 156	USEPA 8082	0.09	0.5	µg/kg, dry wt.	60-130	40
PCB 157	USEPA 8082	0.2	0.5	µg/kg, dry wt.	60-130	40
PCB 167	USEPA 8082	0.3	0.7	µg/kg, dry wt.	60-130	40
PCB 169	USEPA 8082	0.1	0.5	µg/kg, dry wt.	60-130	40
PCB 189	USEPA 8082	0.09	0.5	µg/kg, dry wt.	60-130	40
Alkylated PAHs	USEPA 8270c	0.08-0.6	5	µg/kg, dry wt.	45-128	40
Organochlorine Pesticides	USEPA 8081 (LL)	0.3-2	5	µg/kg, dry wt.	42-150	40
Total Metals	USEPA 6010	0.004-0.2	0.02-1	mg/kg, dry wt.	70-130	30
SEM/AVS	Allen, Fu et al. 1992	Method	Method	mg/kg, dry wt.	50-150	20
Total Petroleum Hydrocarbons	TPH D (C-10 to C-34)	4	25	mg/kg, dry wt.	63-133	40
Total Organic Carbon (TOC)	ASTM D4129-82M	0.02	0.05	Percent	85-115	20
Grain Size	ASTM D422	NA	NA	Percent	NA	20
Moisture Content	ASTM D2216	NA	NA	Percent	NA	20
Sediment Porewater						
Porewater Extraction	Hoffman 1998	NA	NA	NA	NA	NA
Selenium-dissolved	USEPA 200.8	0.80	1	µg/L	85-115	20
Arsenic-dissolved	USEPA 200.8	0.20	0.5	µg/L	85-115	20
Ammonia	USEPA 350.3	0.03	0.05	mg/L	85-115	20
pH	USEPA 150.1	NA	NA	standard units	NA	NA

ARCADIS

Table B-2. Laboratory- Specific Precision and Accuracy Control Limits for Analytical Parameters, Owens-Brockway, Portland, Oregon

Parameters	Analytical Methods	MDL	RL	Units	Accuracy % Recovery	Precision % RPD
Biota						
PCB-Homologues						
Monochlorobiphenyl	USEPA 680	0.68	3.3	µg/kg	30-130	50
Dichlorobiphenyl	USEPA 680	0.76	3.3	µg/kg	30-130	50
Trichlorobiphenyl	USEPA 680	0.68	3.3	µg/kg	30-130	50
Tetrachlorobiphenyl	USEPA 680	1.3	6.7	µg/kg	40-140	50
Pentachlorobiphenyl	USEPA 680	0.83	6.7	µg/kg	40-140	50
Hexachlorobiphenyl	USEPA 680	0.89	6.7	µg/kg	40-140	50
Heptachlorobiphenyl	USEPA 680	1.6	10	µg/kg	40-140	50
Octachlorobiphenyl	USEPA 680	0.95	10	µg/kg	40-140	50
Nonachlorobiphenyl	USEPA 680	1.9	17	µg/kg	30-130	50
Decachlorobiphenyl	USEPA 680	1.9	17	µg/kg	30-130	50
PCB Congeners						
PCB 77	USEPA 8082	0.3	0.5	µg/kg, wet wt.	60-130	40
PCB 81	USEPA 8082			µg/kg, wet wt.	60-130	40
PCB 105	USEPA 8082	0.2	0.5	µg/kg, wet wt.	60-130	40
PCB 114	USEPA 8082	0.2	0.7	µg/kg, wet wt.	60-130	40
PCB 118	USEPA 8082	0.2	0.5	µg/kg, wet wt.	60-130	40
PCB 123	USEPA 8082	0.2	0.5	µg/kg, wet wt.	60-130	40
PCB 126	USEPA 8082	0.2	0.5	µg/kg, wet wt.	60-130	40
PCB 156	USEPA 8082	0.2	0.5	µg/kg, wet wt.	60-130	40
PCB 157	USEPA 8082	0.2	0.5	µg/kg, wet wt.	60-130	40
PCB 167	USEPA 8082	0.2	0.7	µg/kg, wet wt.	60-130	40
PCB 169	USEPA 8082	0.2	0.5	µg/kg, wet wt.	60-130	40
PCB 189	USEPA 8082	0.1	0.5	µg/kg, wet wt.	60-130	40
Organochlorine Pesticides	USEPA 8081 (LL)	0.1-0.4	1	µg/kg, wet wt.	70-130	40
Lead	USEPA 200.8	0.004	0.02	mg/kg, dry wt.	70-130	30
Selenium	USEPA 7742	0.05	0.1	mg/kg, dry wt.	70-130	30
Lipid Content	USEPA OB1090	NA	NA	percent	NA	NA

ASTM - American Society for Testing and Materials
AVS - acid volatile sulfide
LL - low level reporting limits
M - modified
MDL - method detection limit
mg/kg - milligrams per kilogram
mg/L - milligrams per liter
NA - not applicable/available
PAHs - polycyclic aromatic hydrocarbons
PCBs - polychlorinated biphenyls
RL - reporting limit
RPD - relative percent difference
SEM - simultaneously extracted metals
µg/kg - micrograms per kilogram
µg/L - micrograms per liter
USEPA - United States Environmental Protection Agency

References:

Allen, H.E., G. Fu, and B. Deng. 1992. Analysis of acid-volatile sulfide (AVS) and simultaneously extracted metals (SEM) for the estimation of potential toxicity in aquatic sediments. Environmental Toxicology and Chemistry 12:1441-1453.

Hoffman, E. 1998. Tributyltin analysis: Clarification of interstitial water extraction and analysis methods – interim. DMMP clarification paper/SMS Technical information memorandum. U.S. Army Corps of Engineers.

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Table B-3. Project Personnel and Responsibilities, Owens-Brockway, Portland, Oregon

Personnel	Responsibilities
Project Manager Tim Barber, PhD.	Final responsibility for project quality. Oversee all investigation activites under Thompson Hine/Owens Brockway Glass Container, Inc. direction, provide technical oversight, implement necessary actions and adjustments to activites to accomplish overall study objectives and quality objectives for this investigation.
Laboratory Project Director Jeff Christian, Columbia Analytical Angie Weimerskirk, STL Savannah Arthur Putt, Springborn Laboratories	Report directly to the project manager and will be responsible for the following: ensuring all resources of the laboratory are available on an as-required basis and overseeing production and final review of analytical reports.
Laboratory Technical Director-Organics Jeff Grindstaff, Columbia Analytical	Reports to the [laboratory] project manager. Responsibilities include: coordinating laboratory analyses, supervising in-house chain-of-custody, scheduling sample analyses, overseeing data review, overseeing preparation of analytical reports, and approving final analytical reports prior to submission to ARCADIS/Thompson Hine/Owens-Brockway.
Laboratory Quality Assurance Manager Lee Wolf, Columbia Analytical Kirstin McCracken, STL Savannah	Has the overall responsibility for data after it leaves the laboratory. The laboratory QA officer will be independent of the laboratory but will communicate data issues through the laboratory project manager. In addition, the laboratory QA officer will: oversee laboratory QA, oversee QA/QC documentation, conduct detailed data review, determine whether to implement laboratory corrective actions, if required, define appropriate laboratory QA procedures, and prepare laboratory SOPs.
ARCADIS Field Team Leader Keri Whetter	Responsible for leading and coordinating the day-to-day activities of the various resource specialists under his/her supervision. Responsibilities include: provision of day-to-day coordination with the project manager on technical issues in specific areas of expertise, developing and implementing field-related work plans, assurance of schedule compliance and adherence to management-developed study requirements, coordinating and managing field staff including sampling and drilling and supervising field laboratory staff, implementing QC for technical data provided by the field staff including field measurement data, adhering to work schedules provided by the project coordinator, authoring, writing and approving of text and graphics required for field team efforts, coordinating and overseeing technical efforts of subcontractors assisting the field team, identifying problems at the field team level, resolving difficulties in consultation with the project manager, implementing and documenting corrective action procedures and provisions of communication between team and upper management, and participating in preparation of the final report.

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Table B-3. Project Personnel and Responsibilities, Owens-Brockway, Portland, Oregon

Personnel	Responsibilities
Database Administrator Michael Bock	Organize and maintain project database, ensure that the data area processed and stored in accordance with the QAPP, and supervise data management personnel.
Quality Assurance Manager Melissa Kleven	Provide technical quality assurance assistance, oversee quality assurance acitivites to ensure compliance with the QAPP, coordinate and supervise data validation and data quality report preparation, and review and submit quality assurance report.
Health and Safety Coordinator Keri Whetter	Oversee activites to ensure health and safety compliance.

QA/QC- Quality Assurance/Quality Control
QAPP- Quality Assurance Project Plan
SOP- Standard Operating Procedure

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Appendix B-1

Selected Analytical Laboratory
Standard Operating Procedures

Polychlorinated Biphenyls (PCBs) by GC/MS (Method 680)

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Approved by:

Kristi McCracken 8/22/01
Date

Title: Quality Assurance Manager

STL X Savannah __ Tallahassee __ Mobile __ Tampa West

1.0 SCOPE AND APPLICATION

1.1 This method can be used to determine the concentration of polychlorinated biphenyls (PCBs) in groundwater, soils, sediments, wastes, and biological tissues by GC/MS. PCBs are reported by the level of chlorination: monochlorobiphenyls, dichlorobiphenyls, trichlorobiphenyls, etc., up to decachlorobiphenyl.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision limits for each target compound is given in Section 5 of the current revisions of the STL's *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan*.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is spiked with a surrogate and extracted using an appropriate extraction procedure. The extract is dried, concentrated to a volume of 1.0mL, and analyzed by GC/MS operated in the Selected Ion Monitoring Mode (SIM). Windows are established to monitor for the characteristic masses of the various PCB congeners. Qualitative identification of the target compounds in the extract is based on the presence of the peak within the SIM window and the mass ratio between the primary and confirmation ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion. Results are reported as total monochlorobiphenyls, total dichlorobiphenyls, etc.

The default identification and quantitation procedure will be to use only the quantitation and confirmation ions for the PCB congeners. Interference check ions, as described in Section 11.2.3, will not be used routinely to evaluate peaks as PCB congeners unless specified in a client QAP or agency requirement or the sample concentration is near a critical quantitation limit. Samples evaluated according to the default quantitation procedures may be slightly high biased.

2.2 This procedure is based on the guidance provided in EPA Method 680. The method has been modified to include the use of a carbon-13 labeled analogue of decachlorobiphenyl (13C12-DCB) as the surrogate in place of the labeled BHC and DDT compounds. 13C12-DCB can be subjected to the optional acid cleanup (the unlabeled analogue, decachlorobiphenyl, is routinely used in SW-846 Method 8082). A window-defining mix containing the first and last eluting isomers of each level of chlorination is used as an aid to establish and verify that the SIM windows are properly set. Preparation procedures are also referenced for soils and biological tissues, which are not included in EPA Method 680.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, and lab glasses or face shield should be worn while handling extracts and standards. Standard preparation, addition of the internal standard solution, and sample extract dilution should be performed in a hood or well ventilated area.

3.2 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to handle these materials safely.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Glassware and/or extraction vessels that have not been properly cleaned may contribute artifacts that make identification and quantification of the target compounds difficult. Elevated baselines may be due to oils, greases, or other hydrocarbons that may be extracted from improperly cleaned glassware or extraction vessels.

4.2 Matrix interferences may be caused by contaminants that are extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences. Sample extracts that contain high concentrations of non-volatile material such as lipids and high molecular weight resins and polymers may require the optional GPC cleanup prior to analysis. The GPC cleanup is generally not effective in removing non-target material that is associated with common petroleum products such as diesel or waste oil. GPC cleanup may be necessary for biological tissues.
Acid cleanup may be employed as an additional cleanup tool.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

MATRIX	Preservative/ Storage	Container	Sample Hold Time[1]	Extract Hold Time[1]
Aqueous	none; 4C +/- 2C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C +/- 2C	500-mL	14 days	40 days
Biological	Frozen	Glass or wrapped in aluminum foil	6 months	40 days

[1] Holding times are advisory - no holding times are defined in method 680.

6.0 APPARATUS AND MATERIALS

6.1 GC/MS System with compatible data system autosampler, splitless injector, and direct capillary interface.

6.2 Recommended Capillary column-HP-5MS, 30m x 0.25mm ID, 0.50um film thickness. Equivalent columns can be used.

6.3 Microsyringes-

6.4 Volumetric flasks, Class A-appropriate volumes

6.5 Analytical balance

7.0 REAGENTS

7.1 Hexane

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with STL SOP AN41:*Standard Material Traceability*. General guidance on the preparation of standards is given in STL SOP AN43:*Standard Preparation.*

The lab should purchase certified solutions from STL-approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See STL SOP AN43 for guidance for standard preparation from neat materials.

8.1 The recommended calibration standards are listed in Appendix A, Table 1. Prepare these standards at the stated concentrations in hexane.

8.2 The surrogate compound, 13C12-Decachlorobiphenyl, is prepared at a concentration of 1.0ug/mL and
1.0mL of this solution is spiked into all samples and QC items prior to extraction.

8.3 The matrix spiking solution contains one PCB congener of each chlorination level except for the nonachlorobiphenyls. The solution is prepared at the indicated concentrations in acetone and 1.0mL of this solution is spiked into all lab spikes and matrix spikes.

COMPOUND	CONCENTRATION (ug/mL)
2-Chlorobiphenyl	2.0
2,3-Dichlorobiphenyl	2.0
2,4,5-Trichlorobiphenyl	2.0
2,2',4,6-Tetrachlorobiphenyl	4.0
2,2',3,4,5'-Pentachlorobiphenyl	4.0
2,2',4,4',5,6'-Hexachlorobiphenyl	4.0
2,2',3,4',5,6,6'-Heptachlorobiphenyl	6.0
2,2',3,3',4,5',6,6'-Octachlorobiphenyl	6.0
Decachlorobiphenyl	10

9.0 SAMPLE PREPARATION

9.1 The sample extraction procedures are given in the following SOPs:

Matrix	SOP Number	Extraction Technique
Aqueous	EX30	Continuous Liquid-liquid Extraction
Aqueous	EX35	Separatory Funnel
Soils/Sediments and Biological	EX40, PS15	Sonication

9.2 The sample concentration procedures are given in STL SOP EX 50: Zymark Nitrogen Concentration.

9.3 Gel permeation chromatography (STL SOP EX61) may help to eliminate or minimize matrix interferences in a limited number of samples. The GPC cleanup is generally not effective on samples containing petroleum products. Acid cleanup (STL SOP EX60) is recommended as a routine cleanup prior to analysis. Sulfur cleanup may be necessary if the sample extract contains high levels of sulfur.

10.0 PROCEDURE

10.1 Instrument Conditions

Instrument conditions may vary according to the sensitivity of each instrument. The following conditions are provided for guidance. The lab must document the conditions used for the analysis of SVOC by GC/MS.

Recommended Column:

HP-5MS 30m x 0.25mm ID, 0.50um film thickness or equivalent

Column flow: Approximately 1mL/min helium

GC Oven temperatures:

Initial column temperature: 45 C for 1 minutes
Column temperature program 1 : 20C per minute to 150C, hold 1 minute
Column temperature program 2 : 10C per minute to 310C, hold until DCB and 13C12-DCB elute

GC injector parameters

Injector temperature : 250-260EC
Injector : splitless
Inlet purge time : 0.8 minutes
Injector liner : 4mm ID quartz or 4mm glass, deactivated
Sample injection volume : 2uL

Mass Spectrometer and interface parameters

Mass spectrometer interface: 300C
Mass spectrometer source temperature: Factory Set
Mass range: SIM (see Table 3 in Appendix B for ions to monitor)
Mass range for DFTPP analysis: 35-500amu at 1 scan per second or less.

10.2 Tune Criteria

Twenty nanograms of DFTPP are analyzed at the beginning of each 12 hour clock as a check on the "tune" of the mass spectrometer. The DFTPP analysis is performed using scan analysis and the same tune parameters are used for the SIM analysis of the calibration standards and samples.

10.2.1 Prepare a 10ng/uL solution of DFTPP column evaluation standard. The standard must also contain p,p'-DDT (4,4'-DDT).

10.2.2 Analyze a 1uL aliquot of the 10ng/uL DFTPP solution using the same temperature program that is used for SIM analysis of the calibration standards, samples, and QC samples.

10.2.3 Evaluate the DFTPP peak.

-The chromatogram should exhibit acceptable baseline behavior and the DFTPP peak should be symmetrical.

-The spectrum of the DFTPP must meet the criteria listed in the SOP Summary. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans +/- 2 scans from the apex can be evaluated for the DFTPP criteria. Consecutive scans within this range may be averaged to meet the criteria.

NOTE: The DFTPP analysis should be evaluated as to the relative size of the DFTPP peak under the m/z 198 profile. A benchmark area window should be established for each instrument and data system. Area outside of this window suggests instrumental problems such as a bad injection, clogged autosampler syringe, leaking injector, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open , etc.

If the DFTPP fails to meet the criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the DFTPP analysis, other corrective measures may include remaking the DFTPP standard, cleaning the mass spectrometer source, etc.

10.3 Window-Defining Solution and SIM Parameters

10.3.1 Analyze 1uL of the window-defining solutions in the scan mode from 45amu to 500amu at > 1 scan per second. Use the same temperature program that will be used for the SIM analysis of PCBs. The window defining solutions may be analyzed separately or may be combined into a single solution.

10.3.2 Determine the retention times of the first and last eluting congeners at each level of chlorination. The quantiation and confirmation masses are listed in Table 3 of Appendix B.

10.3.3 Set the SIM parameters as follows. Refer to Table 3 of Appendix B for the ion sets.

-Begin data acquisition with ion set #1 before the elution of PCB congener #1, Stop the acquisition of ion set #1 and begin acquisition of ion set #2 approximately 10 seconds before the elution of PCB congener #104.
-Stop the acquisition of ion set #2 and begin the acquisition of ion set #3 approximately 10 seconds after the elution of PCB congener #77..
-Stop the acquisition of ion set #3 and begin the acquisition of ion set #4 approximately 10 seconds after the elution of 4,4'-DDT. (the retention time of 4,4'-DDT is determined from the scan analysis of the DFTPP solution that is analyzed at the beginning of each 12-hour clock.).
-Stop the acquisition of ion set #4 and begin the acquisition of ion set #5 approximately 10 seconds before the elution of PCB congener #208.

10.4 Initial Calibration

After the SIM windows are established and verified and the DFTPP criteria has been met, the initial calibration standards are analyzed. Note that a single PCB congener of each chlorination level is used for calibration and quantiation. Decachlorobiphenyl is used to quantify nonachlorobiphenyls.

10.4.1 Prepare the initial calibration standards. The lowest calibration standard should be at the RL and the rest of the standards will define the working range.

10.4.2 Set up a sequence and analyze the calibration standards. The injection volume must be the same for the calibration standards and all sample extracts. The routine volume is 1uL.

10.4.3 Identify the internal standards, surrogates, and the target compounds. The data system must be updated with the proper retention times and ion data.

10.4.4The relative response factor for each compound is calculated as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where

Ax = area of the characteristic ion of the calibration congener
Ais = area of the characteristic ion for Chrysene-d10
Cx = concentration of the compound being measured (ug/mL)
Cis = concentration of the internal standard (40ug/mL)

NOTE: Use Chrysene-d10 as the internal standard unless matrix interferences are encountered. If phenthrene-d10 must be used, the calibration must be re-evaluated and verified using the second internal standard

10.4.5 Calculate the average relative response factor (RRF_{avg}) for each target compound and each surrogate compound:

$$RRF_{avg} = \frac{RRF1 + RRF2 + RRF3.... + RRFn}{n}$$

RRF1 = relative response factor of the first standard
RRFn = relative response factor of the last standard
n = number of calibration standards

10.4.6Calculate the standard deviation (SD) for the initial calibration standards:

$$SD = \sqrt{\frac{\sum_{i-1}^{n_1} (RRF_i - RRF_{avg})^2}{n-1}}$$

10.4.7Calculate the relative standard deviation (%RSD) of the target compounds in the calibration standards.

$$\%RSD = \frac{SD}{RRF_{avg}} x\,100$$

- If the %RSD of each target compound is less than or equal to 20%, the average response factor can be used for quantitation of samples.
- If the %RSD of the target compound is greater than 20%, the curve should be evaluated for errors and one or more standards re-analyzed. Take corrective action until the %RSD of each target is less than 20%.

10.4.8 Performance Criteria
In addition to meeting the calibration criteria, the following performance criteria must also be met for the mid-level standard.

-Mass abundance ratios of all calibration congeners within acceptance range (see Table 4,Appendix B)
-Baseline separation of PCB congener #87 from congeners #154 and #77
-Signal-to-noise ratio of >=5 for decachlorobiphenyl ion 499 and chrysene-d12 ion 241
-decachlorobiphenyl mass abundance: mass 500 >=70% but <=95% of mass 498

10.5 Continuing Calibration Verification
Samples are analyzed only after the DFTPP criteria and the calibration acceptance criteria have been met. The analytical system must be evaluated every 12 hours by the analysis and evaluation of the DFTPP and a mid-level calibration standard prior tot he analysis of samples and after the samples by the analysis and evaluation of a mid-level standard.

10.5.1The percent difference or percent drift between the continuing calibration RRF and the average relative response factor (RRFavg) is calculated for each target compound and each surrogate compound:

$$\%difference = \left| \frac{RRF - RRFavg}{RRFavg} \right| \otimes 100$$

where
RRF = relative response factor from CCV
RRFavg = average relative response factor from initial calibration curve

If the percent difference is less than or equal to 20% for each target compound, the initial calibration is verified.

If the continuing calibration criteria are not met, action must be taken to bring the analytical system into compliance with the criteria. This action may include injection port maintenance, source cleaning, changing the column, or replacement of injection port lines and assembly. In any case, if the criteria are not met, the analysis of the continuing calibration standard must be repeated. The analyst must be aware of the 12-hour clock-DFTPP criteria must be met prior to the analysis of the calibration standards. If the continuing calibration standard repeatedly fails the calibration verification criteria, the initial calibration curve must be reanalyzed and reevaluated.

The performance criteria given in Section 10.4.8 must also be met prior to the analysis of samples.

10.6 Sample Analysis
Remove the sample extracts to be analyzed from the refrigerator and allow the sample to come to ambient temperature.

10.6.1 Add 30uL of the internal standard mix (25ug/mL) to each 1-mL aliquot of the sample extract. The concentration of the internal standard in the extract is 0.75 ng/uL.

10.6.2 Mix the contents of the autosampler vial by inverting several times.

10.6.3 Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

10.6.4 The dilution factor is calculated by dividing the volume of sample extract in microliters into 1000. For example, if 100uL of a sample extract is diluted to final volume of 1.0mL, the dilution factor is 10.
(1000/100 = 10). The following table gives some dilution factors:

Dilution Preparation

uL extract-Vext	uL MeCl2	volume of dilution (Vdil-uL)	uL ISTD (25ug/mL)-Vistd	DF
1000	0	1000	30	1
500	500	1000	15*	2
200	800	1000	24	5
100	900	1000	27	10
50	950	1000	28.5	20
20	980	1000	30	50

*assumes dilution of a 1.0mL extract or 1mL aliquot of an extract that has been spiked with the internal standard at 0.75ug/mL using 30ul of a 25.0ug/mL internal standard solution

The concentration of internal standards must remain constant for all extracts and extract dilutions at 0.75ug/mL. The following equation can be used to determine the volume of the 25.0 ug/mL internal standard solution to add to an extract when a dilution is prepared from an extract that has already been spiked with the internal standard solution:

$$Vistd(uL) = 30uL - \left(\frac{Vext}{Vdil} \otimes 30ul\right)$$

Vistd = volume of 25.0ug/mL internal standard to add to the diluted extract (uL)
Vext = volume of extract used to prepare the dilution (uL)
Vdil = final volume of the dilution (uL)-1000uL (1.0mL)

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative Analysis

The default procedure will be to use only the quantitation and confirmation ions for identification and quantification of PCB congeners. Interference check ions, as described in Section 11.2.3, will not be used routinely to evaluate peaks as PCB congeners unless specified in a client QAP or agency requirement or the sample concentration is near a critical quantitation limit.

11.1.1 Examine the Selected Ion Current Profiles (SICP) for the internal standards. Confirm that the RT and response of the internal standards are within the acceptance criteria specified in the SOP Summary. If the internal standard retention times have changed significantly or the peaks cannot be located, stop and analysis and correct the problem. Reanalyze any associated samples.

11.1.2 Evaluate the peaks for candidates to be identified as PCBs. A peak is tentatively identified as a PCB if

-The peak falls within the retention time range bordered by the first and last eluting isomer of that chlorination level

-The ratio of the quantitation and confirmation ions are present and the area ratios fall within the acceptance criteria in Appendix B, Table 4. The scans must maximize within one scan of each other. Examine the data for the presence of a coeluting PCB of higher chlorination if both ions and the M-70 ions are present and the ratio does fall within the acceptance limits.

-The areas for the quantiation and confirmation ions must be greater than three times the background noise and must fall within the working range of the calibration curve (must not saturate the detector

-At least one ion in the M-70 cluster must be present

11.1.3 Evaluate each PCB candidate in the Cl-3 to Cl-7 range for the presence of coeluting PCBs containing one or two additional chlorines. An intense M+35 ion at the retention time may indicate a PCB with one additional chlorine and the presence of an intense M+70 would indicate a co-eluting PCB containing two additional chlorines. Use the information in Tables 5 and 6 of Appendix B to correct for the interfering ion(s).

For example, if a Cl-7-PCB and a Cl-5-PCB coelute, the Cl-7-PCB will contribute to the quantiation and confirmation ions for the Cl-5-PCB. Cl-7-PCB produces a cluster of three ions by the loss of two chlorines-ions 322,324, and 326. Two of these ions-324 and 326-are also ions contained in the molecular ion cluster of Cl-5-PCB. To determine the ion 326 and 324 areas produced only by the Cl-5-PCB, calculate the contribution to each and subtract it from the measured areas. See Tables 5 and 6 in Appendix B for the percentage of the interference peak to subtract from the quantiation and confirmation ions. In this example, 164% of the area measured for ion 322 should be subtracted from the area measured for ion 324 and 108% of the area measured for ion 322 should be subtracted from ion 326.

NOTE: A coeluting PCB with one more chlorine will affect only the quantiation ion (Table 6). The interference from a coeluting PCB containing one more chlorine, due to the natural abundance of 13C12, is small and can usually be neglected except when measuring the area of a small amount of a PCB coeluting with a large amount of a another PCB containing one more chlorine.

11.2 Calculations for Samples-Internal Standard Technique
These calculations assume that the same volume is injected for standards and samples.

11.2.1 Aqueous Samples

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{V} \otimes DF$$

where

Ax =	sum of areas of the characteristic ion of the PCB chlorination level being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

The reporting limit (RL) for each sample is given:

$$RL(ug/L) = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Vqap}{V} \otimes DF$$

where

F =	final volume of extract (mL)
Fqap =	1.0mL
Vqap =	1.0L
V =	volume of sample extracted
DF =	dilution factor. The STL CQAP Table 5 RL(RLqap) assumes a DF of 1.

NOTE: If V = 800mL to 1200mL, assume that Vqap/ V = 1 in the calculation of the reporting limit.

11.2.2 Soils

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ax =	sum of areas of the characteristic ion of the PCB chlorination level being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
W =	weight of sample extracted (kg)
solids =	(percent solids)/100
DF =	dilution factor

The reporting limit (RL) for each sample is given:

$$RL = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Wqap}{(W)(solids)} \otimes DF$$

where

F = final volume of extract (mL)
W = weight of sample extracted (kg)
solids = (percent solids)/100

The STL CQAP assumes Wqap = 30g, solids = 1, Fqap = 1.0mL, and DF = 1.

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in AN02: *Analytical Batching*. STL SOP AN02 also describes the procedure for evaluating batch-specific QC. The attached SOP summary and Table 13.1 in the STL Corporate QA Plan provide guidance for evaluating sample data.

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each laboratory must demonstrate competence in the analysis of samples by this procedure. The minimum criteria for this demonstration are the preparation and analysis of spiked reagent water. The criteria for IDOC accuracy and precision are the accuracy and precision criteria listed in Table 5 of the STL QAP.

12.3 Method Detection Limit

The method detection limit is determined by each lab annually in accordance with STL SOP CA90:*Procedure for Determination of Method Detection Limit (MDL)*.

13.0 PREVENTIVE MAINTENANCE

Preventive maintenance items will be added at a later date. See Section 10 of the current STL Quality Assurance Plan.

14.0 TROUBLE-SHOOTING-Trouble-shooting items will be added at a later time.

15.0 REFERENCES

15.1 *STL's Comprehensive Quality Assurance Plan* and STL's *Corporate Quality Assurance Plan*, current revisions.

15.2 *Method 680: Determination of Pesticides and PCBs in Water and Soils/Sediment by Gas Chromatography/Mass Spectrometry*. November 1985. Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, Office of Research and Development, USEPA, Cincinnati, OH

APPENDIX A

TABLE 1 CALIBRATION STANDARDS

CALIBRATION COMPONENTS	CAL 1	CAL 2	CAL3	CAL4	CAL5
Calibration Congener					
2-chlorobiphenyl (1)	0.10	0.50	1.0	2.0	5.0
2,3-dichlorobiphenyl(5)	0.10	0.50	1.0	2.0	5.0
2,4,5-trichlorobiphenyl(29)	0.10	0.50	1.0	2.0	5.0
2,2',4,6-tetrachlorobiphenyl(50)	0.20	1.0	2.0	4.0	10
2,2',3,4,5'-Pentachlorobiphenyl (87)	0.20	1.0	2.0	4.0	10
2,2',4,4',5,6'-hexachlorobiphenyl(154)	0.20	1.0	2.0	4.0	10
2,2',3,4',5,6,6'-heptachlorobiphenyl(188)	0.30	1.5	3.0	6.0	15
2,2',3,3',4,5,5',6,6'-octachlorobiphenyl(200)	0.30	1.5	3.0	6.0	15
Decachlorobiphenyl	0.50	2.5	5.0	10	25
Retention Time Congeners					
3,3',4,4-tetrachlorobiphenyl(77)	0.20	1.0	2.0	4.0	10
2,2',4,6,6'-Pentachlorobiphenyl (104)	0.20	1.0	2.0	4.0	10
2,2',3,3',4,5,5',6,7'-nonachlorobiphenyl(208)	0.40	2.0	4.0	8.0	20
Surrogate					
13C12-Decachlorobiphenyl	0.50	2.5	5.0	10	25
Internal Standards					
Phenathrene-d10	0.75	0.75	0.75	0.75	0.75
Chrysene-d12	0.75	0.75	0.75	0.75	0.75

TABLE 2 –First and Last Eluting Isomers

Congener	First Eluting Isomer	Last Eluting Isomer
Cl-1	2-chlorobiphenyl	4-Chlorobiphenyl
Cl-2	2,6-dichlorobiphenyl	4,4'-dichlorobiphenyl
Cl-3	2,2',6-trichlorobiphenyl	3,4,4'-trichlorobiphenyl
Cl-4	2,2',6,6'-tetrachlorobiphenyl	3,3',4,4'-tetrachlorobiphenyl
Cl-5	2,2',4,6,6'-pentachlorobiphenyl	3,3',4,4',5-pentachlorobiphenyl
Cl-6	2,2',4,4',6,6'-hexachlorobiphenyl	3,3',4,4',5,5'-hexachlorobiphenyl
Cl-7	2,2',3,4',5,6,6'-heptachlorobiphenyl	2,3,3',4,4',5,5'-heptachlorobiphenyl
Cl-8	2,2',3,3',5,5',6,6'-octachlorobiphenyl	2,3,3',4,4',5,5',6-octachlorobiphenyl
Cl-9	2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl

APPENDIX B
SIM IONS

Table 3-Ions for SIM Acquisition

ION Set 1 (a)	ION Set 2 (b)	ION Set 3 (c)	ION Set 4 (d)	ION Set5 (e)
152	186	248	240	356
153	188	249	241	358
186	220	254	288	360
187	222	256	290	390
188	254	288	322	392
189	255	290	324	394
190	256	322	326	424
220	258	323	356	425
221	288	324	357	426
222	289	326	358	428
224	290	328	360	430
255	292	357	362	432
256	294	358	391	462
258	323	360	392	464
290	324	362	394	466
292	326	392	396	496
294	328	394	398	498
	358	396	428	499
	360	398	430	500
	362		432	502

(a) Cl-1 to Cl-4 and Phenthrene-d10
(b) Cl-3 to-Cl-6
(c) Cl5 to Cl-7
(d) Cl-6 to Cl-8 and Chrysene-d12
(e) Cl-8 to Cl-10 and 13C12-DCB

TABLE 4-Approximate Retention Times for PCB Isomer Groups and Calibration Congeners

PCB Isomer Group	Approximate RRT Range	Calibration Congener	Approximate Calibration Congener RRT
Cl-1	0.30-0.35	2-chlorobiphenyl (1)	0.30
Cl-2	0.38-0.50	2,3-dichlorobiphenyl(5)	0.43
Cl-3	0.46-0.64	2,4,5-trichlorobiphenyl(29)	0.54
Cl-4	0.55-0.82	2,2',4,6-tetrachlorobiphenyl(50)	0.56
Cl-5	0.64-0.92	2,2',3,4,5'-entachlorobiphenyl (87)	0.80
Cl-6	0.75-1.1	2,2',4,4',5,6'-hexachlorobiphenyl(154)	0.82
Cl-7	0.88-1.2	2,2',3,4',5,6,6'-heptachlorobiphenyl(188)	0.88
Cl-8	0.99-1.21	2,2',3,3',4,5,5',6,6'-octachlorobiphenyl(200)	1.03
Cl-9	0.16-1.28	Decachlorobiphenyl	1.3
Cl-10	1.3	Decachlorobiphenyl	1.3

RRT = retention time relative to Chrysene-d12

Table 4 Quantitation and Interference Check Ions

PCB Isomer Group	Quant ION	Confirmation ION	Expected Ratio(a)	Acceptable Ratio(a)	M-70 Confirmation ION	Interference Check ION M+70	Interference Check ION M+35
Cl-1	188	190	3.0	2.5-3.5	152	256	222
Cl-2	222	224	1.5	1.3-1.7	152	292	256
Cl-3	256	258	1.0	0.8-1.2	186	326	290
Cl-4	292	290	1.3	1.1-1.5	220	360	326
Cl-5	326	324	1.6	1.4-1.8	354	394	360
Cl-6	360	362	1.2	1.0-1.4	288	430	394
Cl-7	394	396	1.0	0.8-1.2	322	464	430
Cl-8	430	428	1.1	0.9-1.3	356	498	464
Cl-9	464	466	1.3	1.1-1.5	390		498
Cl-10	498	500	1.1	0.9-1.3	424		
Chrysene-d12	240	241	5.1	4.3-5.9			
Phenathrene-d10	188	189	6.6	6.0-7.2			
13C12-DCB (surrogate)	510	512					

(a) ratio of quantitation ion to confirmation ion

TABLE 5-Corrections for Interference of PCB Containing Two Additional Chlorines

PCB Isomer Group	Quant ION	Confirmation ION	Ion Measured to Determine Interference	Percent Ion area to be subtracted from QUNAT ION Area	Percent Ion area to be subtracted from CONFIRMATION ION Area
Cl-3	256	258	254	99	33
Cl-4	292	290	288	65	131
Cl-5	326	324	322	108	164
Cl-6	360	362	356	161	71
Cl-7	394	396	390	225	123

TABLE 6-Corrections for Interference of PCB Containing One Additional Chlorine

PCB Isomer Group	Quant ION	Ion Measured to Determine Interference	Percent Ion area to be subtracted from QUNAT ION Area
Cl-2	222	221	13.5
Cl-3	256	255	13.5
Cl-4	292	289	17.4
Cl-5	326	323	22.0
Cl-6	360	357	26.5
Cl-7	394	391	30.9
Cl-8	430	425	40.0

APPENDIX C
680 SOP SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C	500-mL	14 days	40 days
Biological	Frozen	Glass or aluminum foil	6 months	40 days

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
DFTPP 10ng on column Clock starts at injection	DFTPP 10ng on column Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification
Samples and the capping standard must be analyzed within 12 hours of the start of the clock	Samples and the capping standard must be analyzed within 12 hours of the start of the clock
Capping standard	Capping standard

DFTPP CRITERIA

m/z	Ion Abundance Criteria
127	40-60% of mass 198
197	<1.0% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass443
442	>40% of mass 198
443	17-23% of mass 442

ANALYTICAL BATCH
SEE STL SOP AN02.

CALIBRATION ACCEPTANCE CRITERIA

Initial Calibration	Continuing Calibration
RRFavg <= 20% RSD	Percent difference <= 20% difference from initial calibration

QC Item	Frequency	Acceptance Criteria	Corrective Action
Tune/Column Evaluation Standard DFTPP 20ng	Prior to analysis of calibration standards every 12 hours	DFTPP - within criteria	-Evaluate alternative scans -Reanalyze and evaluate -Retune and reanalyze -Clean source, retune, reanalyze
Initial Calibration-minimum of five calibration standards	After Tune Check and when calibration verification standard fails acceptance criteria.	%RSD <= 20%	-Reanalyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Retune and reanalyze standards -Replace column and reanalyze standards -Clean source and reanalyze standards
Performance Criteria	Evaluate mid level calibration standard each clock	-Mass abundance ratios of all calibration congeners within acceptance range (see Appendix B) -Baseline separation of PCB congener #87 from congeners #154 and #77 -Signal-to-noise ratio of >=5 for decachlorobiphenyl ion 499 and chrysene-d12 ion 241 -decachlorobiphenyl mass abundance: mass 500 >=70% but <=95% of mass 498	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate
Continuing Calibration Verification	After tune check; every 12 hours prior to analysis of samples and at the end of the analytical sequence	%Difference <= 20%	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate

QC Item	Frequency	Acceptance Criteria	Corrective Action
Internal Standard Areas	Evaluate all standards and samples	Areas in continuing calibration verification must be within 30% of the previous CCV and within 50% of the initial calibration Areas in samples should be evaluated for gross error . Consult superior	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available
Surrogate recovery	Evaluate for all samples and QC items if extract is not diluted OR If diluted, where >RL	Within Section 5 QAP limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Method Blank	Per batch	All targets < RL in Section 5 Table of QAP	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL SOP AN02 and STL QAP Table 13.1
Lab Control Standard (LCS) - QAP subset	See AN02	All spiked targets within the accuracy criteria in Section 5 Table of QAP	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL SOP AN02 and STL QAP Table 13.1
Matrix spike (MS) Matrix spike duplicate (MSD)	Per batch if sufficient sample volume/weight supplied See AN02	All targets within the accuracy and precision criteria in Section 5 Table of QAP	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL SOP AN02 and STL QAP Table 13.1

QC Item	Frequency	Acceptance Criteria	Corrective Action
Initial Demonstration of Capability (IDOC)	Per analyst	Accuracy and precision within method specified criteria	-Evaluate data -Reanalyze extracts if warranted -Re-extract and reanalyze for targets that fail criteria
Method Detection Limit (MDL)	See CA90	Evaluate according to STL SOP CA90	Evaluate according to STL SOP CA90

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STANDARD OPERATING PROCEDURE

CONGENER-SPECIFIC DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs) BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTION (GC/ECD)

SOC-8082C
Revision 3
MAY 22, 2002

Approved By:

Supervisor	5/22/02 Date
QA Manager	5-22-02 Date
Laboratory Manager	5/22/02 Date

COLUMBIA ANALYTICAL SERVICES, INC.
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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER: _____
Initials: _____ Date: _____

CONGENER-SPECIFIC DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBS) BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTION (GC/ECD)

1. SCOPE AND APPLICATION

1.1. This procedure is used specifically to determine the concentrations of certain PCB congeners in water, soil and sediment using EPA method 8082. This procedure may also be applicable to various tissue samples. The procedure is intended to determine these compounds at trace levels and therefore is not applicable to miscellaneous waste matrices and oils. More appropriate methods are available for the determination of PCBs (as Aroclors) in waste matrices.

1.2. There are 209 identified congeners for which the method may possibly be applied. Table 1 lists the analytes that can routinely be determined by this procedure and lists the method reporting limits (MRLs) for each compound in water and soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The reported MRL may be adjusted if required for specific project requirements, however, the capability of achieving other reported MRLs must be demonstrated. The Method Detection Limits (MDLs) which have been achieved are given in Table 1.

2. METHOD SUMMARY

This procedure is based on EPA Method 8082. This procedure provides sample preparation techniques and GC/ECD conditions for the detection PCB congeners at trace levels. Samples are extracted using an appropriate technique for the sample matrix. Cleanup steps are used to remove unwanted co-extracted compounds and/or matrix interferences. Depending on the sample matrix, some cleanup steps may not be necessary. The final sample extract is analyzed by simultaneous dual column gas chromatography using an electron capture detector. The compounds are separated on fused silica capillary columns with differing chromatographic separation characteristics. Identification of the analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard. Quantitative analysis is performed by using the authentic standard to produce a response factor or calibration curve. The calibration data is used to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume.

3. DEFINITIONS

Analysis Sequence - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with calibration standards. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

Initial (or Independent) Calibration Verification (ICV) - Initial calibration verification standards which are analyzed after initial calibration with newly prepared standards but prior to sample analysis, in order to verify the validity of the standards used in calibration. The ICV standards are prepared from materials obtained from a source different from that used to prepare calibration standards.

Matrix Spike/Duplicate Matrix Spike Analysis - In the matrix spike analysis, predetermined quantities of stock solutions of selected Aroclors are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, spiked, and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

Standard Curve - A standard curve is a calibration curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

Surrogate - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples, and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Method Blank - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

Continuing Calibration Verification Standard (CCV) - A mid-level standard injected into the instrument at specified intervals and is used to verify the initial calibration.

Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into other analyses.

4. INTERFERENCES

4.1. Solvents, reagents, glassware, and other sample processing equipment may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by analyzing method blanks. Specific selection of reagents and the use of solvents purified by distillation in all-glass systems may be required. Analysts should pay particular attention to the introduction of contamination from phthalate esters and use sample handling practices which minimize the introduction of the compounds into extracts.

4.2. Interferences coextracted from samples will vary considerably from source to source, depending upon the sample matrix. Cleanup techniques are generally required to eliminate

significant levels of interferences. The presence of organochlorine pesticides in the sample can represent a potential interference which may exist after cleanup steps have been taken.

5. SAFETY

5.1. The toxicity or carcinogenicity of each compound or reagent used in this method may not be precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level.

5.2. Follow all applicable safety procedures as described in the CAS Safety Manual. A reference file of material safety data sheets is available to all personnel involved in these analyses. CAS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Containers used to collect samples for the determination of semivolatile organic compounds should be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or teflon and have screw-top covers with teflon liners. In situations where teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

6.2. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g., if an automatic sampler is used), run reagent water through the sampler and use the rinseate as a field blank.

6.3. Water and soil samples must be iced or refrigerated at 4 ± 2°C from time of collection until extraction. Tissue samples should be stored in accordance with project requirements, typically refrigerated or frozen.Water and soil samples must be iced or refrigerated at 4 °C from time of collection until extraction.

6.4. Water samples should be extracted within 7 days and the extracts analyzed within 40 days. Soil and sediment samples should be extracted within 14 days and the extract analyzed within 40 days.

7. APPARATUS AND EQUIPMENT

7.1. Gas Chromatograph System

7.1.1. An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless or on-column injection and all required accessories, including syringes, analytical columns, and gases.

7.1.2. Gas Chromatograph - Equipped with cool-on-column or split/splitless injection port that is temperature programmable with dual ECD, HP/AGILENT 5890 or 6890. See Table 2 for typical chromatographic conditions.

7.1.3. GC Autosampler: The GC system should be configured with a compatible autosampler for automated injection of standards, samples, and QC samples.

7.1.4. Columns :

Column 1: 105 meter Restek RTX-5, 0.25mm i.d., 0.25 μm, or equivalent.
Column 2: 105 meter Restek RTX-200, 0.25mm i.d., 0.25 μm, or equivalent.

7.1.5. Data System - A computer system must be interfaced to the GC/ECD. The system must allow the continuous acquisition and storage on machine-readable media of all chromatographic data obtained throughout the duration of the chromatographic program. The computer must have software that performs analysis of chromatographic data (response vs. time) and allows the user to program qualitative and quantitative parameters which result in analyte identification and quantitation. The most recent version of the manufacturer's software is preferred (HP Chemstation/Enviroquant).

7.2. Analytical Balance (0.0001 g), syringes, bottles, and vials as required. All bottles and vials should use Teflon-lined caps or septa.

7.3. Sample preparation apparatus - Glassware and associated apparatus as required to perform sample extractions as described in SOPs for methods 3510C, 3520C, 3540C, 3545 and 3550B. Gel-Permeation Chromatography cleanup apparatus as described in EPA Method 3640. Other cleanup apparatus as specified in EPA Methods 3630 and 3665.

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Solvents: Methylene chloride, hexane, acetone, methanol, or other solvents as appropriate. Pesticide quality or equivalent.

8.2. Stock Standard Solutions

8.2.1. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or verified against an independent source. Standard concentrations can be verified by comparison versus an independently prepared standard. Alternatively, prepare stock standard solutions of at least 10 μg/ml by dissolving an appropriate weight (nearest .0001 g) reference standard in hexane, isooctane, or methylene chloride and diluting to volume in a 10 ml volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound

purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard.

8.2.2. Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4°C and protect from light. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

8.2.3. Stock standard solutions must be replaced after one year, or sooner, if comparison with check standards indicates a problem.

8.3. Calibration Standards: Calibration standards at a minimum of five concentration levels for each parameter of interest are prepared through dilution of the intermediate standards with hexane or isooctane. One of the concentration levels should be at the method reporting limit (MRL). The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define a linear range. Calibration solutions must be monitored closely for signs of degradation. The recommended replacement interval is 6 months.

8.4. Surrogate Standard: The performance of the extraction, cleanup, and analytical system and the effectiveness of the method in dealing with each sample matrix is monitored by spiking each sample, method blank, and matrix spike with a surrogate compound. For this analysis, the surrogate selected is hexabromobiphenyl. Prepare a spiking solution at 4 ug/ml. Store at 4°C and protect from light. Surrogate spiking solutions are replaced periodically based on the stability of the compounds and frequency of use.

8.5. Matrix Spike Standards: Matrix spike solutions are prepared at a level representative of the calibration mid-range. Prepare a solution that contains all QC target analytes. Either a subset of the target list or the entire target list is reported depending on project requirements. Store at 4±2°C and protect from light. Matrix spike solutions are replaced periodically based on the stability of the compound(s) and/or frequency of use.

8.6. Internal Standard Solution: The internal standard used is 2,4-Dibromobiphenyl. Prepare a working standard at a concentration of 10 ug/ml. Each extract undergoing analysis is spiked with 10μL of the internal standard solution, resulting in a concentration of 100 ng/ml.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2. Carrier gas - Inline purifiers or scrubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Gas Chromatograph

9.3.1. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, o-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chromatographic performance. In some cases liners and seals may be cleaned and re-used.

9.3.2. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool.

9.3.3. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility or the department supervisor/manager to document analyst training. Documenting method proficiency, as described in 8082, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

Water samples are extracted at a neutral, or as is, pH with methylene chloride, using either EPA Method 3510C or 3520C. Sediment, soil, and solid samples are extracted using either EPA Method 3540C, 3545, or 3550B. Refer to the CAS SOPs for those procedures. Additional sample cleanup procedures may be employed as appropriate for the samples. See Figure 1.

11.2. Calibration

NOTE: Refer to SOP for Calibration of Instruments for Organics Chromatographic Analyses. The calibration procedure(s) and options chosen must follow the CAS protocols. In general, the calibration procedure is as follows:

11.2.1. The GC is configured to perform simultaneous analysis using the two columns and dual ECD detectors. The columns are configured using a splitter which diverts flow from the guard column to the two analytical columns. Operating conditions must be the same for all calibration analyses and related sample analyses. The recommended GC operating conditions are given in Table 2.

11.2.2. Initial Calibration

Calibration standards at a minimum of 5 concentration levels are analyzed. Analyze 1 μL of each calibration standard and tabulate the peak area and concentration for each compound. Calculate response factors (RFs) for each compound as follows:

$$RF_x = \frac{(A_x)(C_{ISTD})}{(A_{ISTD})(C_x)}$$

where:

Ax = Area of the compound being measured.
Ais = Area of the internal standard.
Cis = Concentration of the internal standard (ng/mL).
Cx = Concentration of the compound being measured (ng/mL).

The average RF is calculated for each compound. The percent relative standard deviation (%RSD) is calculated for each compound. If the RF value is constant over the calibration range (<20% RSD) the average RF is used for quantitation. Refer to the SOP for Calibration of Instruments for Organics Chromatographic Analyses for alternate calibration procedures. If the initial calibration criteria cannot be met, the standards and/or GC system should be inspected and corrective action taken. Injection port cleaning and/or removal of a portion of the column inlet may be required.

11.2.3. The initial calibration is verified by an independent source. Prepare an independent calibration verification standard (ICV) by dilution of a stock solution purchased from a different vendor and analyze immediately after each initial calibration. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. Evaluate the ICV as described in the SOP for Calibration of Instruments for Organics Chromatographic Analyses.

11.3. Retention Time Windows

11.3.1. Establish retention time windows with the GC system in acceptable operating conditions. Make three injections of all analytes throughout the course of a 72-hour period. Serial injections over less than a 72-hour period may result in retention time windows that are too tight. Using retention times from these analyses, calculate retention time windows. Refer to EPA Method 8000B for detailed instructions.

11.3.2. Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. In those cases where the standard deviation for a particular standard is zero, substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

11.3.3. Calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. Retain this data in the method file.

11.4. Calibration Verification

11.4.1. The working calibration curve or calibration factor must be verified on each analytical sequence by the analysis of one or more mid-range calibration standards (CCV). A mid-level standard (CCV) must be injected at the start of each sequence. For internal standard calibration technique, a CCV standard is to be analyzed every 24 hours. The last analysis in the sequence must be started within 24 hours from the time of initiation of the analytical batch. If internal standard calibration is used, a CCV after samples is not required.

11.4.2. Compare the response factor with the average response factor from the initial calibration. The acceptance criteria for all analytes in the CCV analysis is a response (RF or concentration) within ± 15% D of the expected value, as compared to the initial calibration. If a calibration model other than average RF is used, compare the calculated result to the true value rather than using Rfs. Refer to the SOP for Calibration of Instruments for Organics Chromatographic Analyses for CCV evaluation protocols. Calculate the percent difference using the following:

$$\%\ Difference = \frac{\overline{RF_I} - RF_c}{\overline{RF_I}} \times 100$$

where:

RFi = Average response factor from initial calibration.
RFc = Response factor from current verification check standard.

11.5. Sample Analysis

11.5.1. Sample extracts are generally screened prior to analysis (see Figure 1). Dilution(s) may be made prior to analysis. Add the internal standard spiking solution to each extract to obtain the nominal extract concentration of 100 ng/ml. Analyze the extract using the recommended operating conditions specified in Table 2, ensuring that the same conditions are used for samples as for calibration.

11.5.2. Perform all qualitative and quantitative measurements as described in the following sections. Store the extracts at 4±2° C, protected from light in vials equipped with unpierced Teflon lined septa.

12. QA/QC REQUIREMENTS

12.1. Method Validation

12.1.1. Initial Precision and Recovery Validation

The precision of the extraction procedure and the GC procedure must be validated before analysis of samples begin, or whenever significant changes to the procedures have been made. To do this, four de-ionized water samples are spiked at a mid-level concentration, then extracted, and analyzed. Currently no specific criteria are available from method references. However, precision of the 4 replicates should approximate what is typically found with semi-volatile analytes analyzed by GC/ECD. The average recovery should approximate what is typically seen with PCB analyses. Refer to EPA Method 8082 for single lab IPR data.

12.1.2. Method Detection Limits

A method detection limit (MDL) study must be undertaken for a given matrix before analysis of samples can begin. To establish low detection limits that are precise and accurate, the analyst must perform the MDL procedure for each matrix of interest. Spike a minimum of seven samples with MDL spiking solution at a level below, or near the expected MRL. Follow the procedures outlined in the CAS SOP for The Determination of Method Detection Limits.

12.2. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for SAMPLE BATCHES. The Quality Assurance Manual (Section 12) provides guidance for interpretation of QC results. Ongoing QC samples include:

12.2.1. Method Blank

A method blank is extracted and analyzed with every batch of 20 samples, or daily, whichever is more frequent, to demonstrate that there are no method interferences. If the method blank shows any hits above the reporting limit, corrective action must be

taken. Corrective action includes recalculation, reanalysis, system cleaning, or reextraction and reanalysis.

12.2.2. Laboratory Control Sample

A laboratory control sample (LCS) must be extracted and analyzed with every batch of 20 samples, or daily, whichever is more frequent. The LCS is prepared by adding a known amount of the matrix spike solution to a blank sample matrix, then proceeding preparation and analysis. Calculate the % recovery as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

The acceptance criteria are given in current CAS QC acceptance criteria listed in QA document QCLIMITS.xls. If the LCS fails acceptance criteria, corrective action must be taken. Corrective action includes recalculation, reanalysis, or reextraction and reanalysis.

12.2.3. Matrix Spike

A matrix spike (MS) and duplicate matrix spike (DMS) must be extracted and analyzed with every batch of 20 samples. The MS/DMS is prepared by adding a known amount of the matrix spike solution to the sample then proceeding with preparation and analysis. Calculate percent recovery (%R) as:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{R1 - R2}{(R1 + R2)/2} \times 100$$

Where R1 = %recovery of the MS
R2 = %recovery of the DMS

The acceptance limits for the MS/DMS are given in current CAS QC acceptance criteria listed in QA document QCLIMITS.xls. If the MS/DMS recovery is out of

acceptance limits for reasons other than matrix effects, corrective action must be taken. Corrective action includes recalculation, reanalysis, or reextraction and reanalysis.

12.2.4. Surrogate

Surrogate spike is added to every sample, blank and spike prior to extraction. Add a known amount of the surrogate spike to all samples and QC samples in the extraction set. The surrogate is used to monitor extraction efficiency. Calculate surrogate percent recovery (%R) as:

$$\%R = S/V \times 100$$

Where S = The amount of surrogate recovered
V = The amount spiked/final volume

The surrogate acceptance limits are given in current CAS QC acceptance criteria listed in QA document QCLIMITS.xls. If the surrogate recovery is outside of acceptance limits for reasons other than matrix effects, corrective action must be taken. Corrective action include recalculation, reanalysis, or reextraction and reanalysis.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. Qualitative Analysis

13.1.1. The sample component RRT must compare within ± 0.06 RRT units of the RRT for the compound in the opening CCV.

13.1.2. Confirmation of all tentative hits must be made. Confirmation is made by injecting the sample extract on two columns with dissimilar phases simultaneously. If the retention time matches on both columns, then the hit for the analyte is considered a confirmed hit. As further confirmation, subsequent quantitations on the two columns should agree. Refer to the CAS Confirmation Policy for guidance. If on one column, known coeluting compounds exist, a quantitation difference may not be an appropriate measure of confirmation. In the case of known coeluting compounds on one column, using quantitation on the two columns as a measure of confirmation should take into account the relative amounts of the two components. When pesticides are known to be present, the quantitations on the two columns may not agree within 40%D and the analyst's judgement should be used to determine if the results represent a positive *congener* result at that quantitation level. Confirmation by GCMS may also be used if the concentration is high enough for detection and accurate spectral identification. A GCMS confirmation need only be qualitative in nature.

13.2. Quantitative Analysis

13.2.1. When a compound has been identified, the quantitation of the compound is based on the area of the compound identified. The quantitation technique used is the same as the calibration technique for the specific component.

13.2.2. Internal Standard quantitation.

Calculate the concentration of each identified analyte in the sample as follows:

Water: $$Concentration(\mu g/L) = \frac{(A_x)(I_s)(V_t)}{(A)_{is}(RF)(V_o)(V_i)}$$

where:

Ax = Area of the compound being measured.
Is = Amount of internal standard injected (ng).
Vt = Volume of total extract, taking into account dilutions (i.e. a 1-to-10 dilution of a 1 ml extract will mean Vt = 10,000 μL).
Ais = Area of the internal standard.
RF = Average response factor for compound being measured.
Vo = Volume of water extracted (ml).
Vi = Volume of extract injected (μL).

Sediment/Soil Sludge (on a dry-weight basis) and Tissue (on a wet-weight basis):

$$Concentration(\mu g/kg) = \frac{(Ax)(I_s)(V_t)}{(A_{is})(RF)(V_i)(W_s)(D)}$$

where:

Ax, Is, Vt, Ais, RF, Vi = Same as for water.
Ws = Weight of sample extracted or diluted in grams.
D = % dry weight of sample/100, or 1 for a wet-weight basis.

13.2.3. Quadratic Curve quantitation:

Using the peak area for the identified component, use the calibration curve constructed during initial calibration to determine the solution (extract) concentration. Use the solution concentration to determine sample concentration as follows:

Water: $$Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$$

where:

Cex = Calculated concentration in the extract analyzed (ug/mL)
Vf = Final volume of extract (mL).
D = Dilution factor.
Vs = Volume of sample extracted (L).

Sediment/Soil Sludge (on a dry-weight basis) and tissue (on a wet-weight basis):

$$Concentration\ (ng / g) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$$

where:

Cex = Calculated concentration in the extract analyzed (ng/uL)
Vf = Final volume of extract (uL).
W = Weight of sample extracted or diluted in grams.
D = % dry weight of sample/100, or 1 for a wet-weight basis.

13.2.4. If the quantitation result is over the calibration range for any compound, a dilution re-analysis is required.

13.3. Data Review

Data for the analytical sequence is compiled in a data "pack" which includes all calibration, QC, and sample data for the sequence. Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for Laboratory Data Review Process for details.

13.4. Reporting

13.4.1. Reports are generated using the STEALTH data reporting system, which compiles the SMO Login information. This compilation is then transferred to a file which STEALTH uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.4.2. As an alternative, reports are generated using Excel© templates located in R:\SVG\forms. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The detected analytes, surrogate and matrix spikes are then transferred, by hand or electronically, to the templates.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for Nonconformity and Corrective Action for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for Report Generation or in project-specific requirements.

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP for The Determination of Method Detection Limits (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the laboratory Safety Manual, Sections 6 and 7.

18. REFERENCES

Polychlorinated Biphenyls (PCBs) by Capillary Column Gas Chromatography, EPASW846, Test Methods For Evaluating Solid Waste, Update III, Method 8082, Revision 0, December, 1996.

Determinative Chromatographic Separations, EPASW846, Test Methods For Evaluating Solid Waste, Update III, Method 8000B, Revision 2, December, 1996.

Extraction Methods 3510C (Separatory Funnel Liquid-Liquid Extraction), 3520C (Continuous Liquid-Liquid Extraction), 3540C (Soxhlet Extraction), 3545 (Pressurized Fluid Extraction), and 3550B (Ultrasonic Extraction); EPASW846, Test Methods For Evaluating Solid Waste, Update III, December, 1996.

Cleanup Methods 3610B (Alumina Cleanup), 3630C (Silica Gel Cleanup), 3640A (Gel-Permeation Cleanup), 3665A (Sulfuric Acid / Permanganate Cleanup); EPASW846, Test Methods For Evaluating Solid Waste, Updates II and III, September 1994 and December 1996.

FIGURE 1

CONGENER-SPECIFIC DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBS)

SAMPLE CLEANUP FLOWCHART

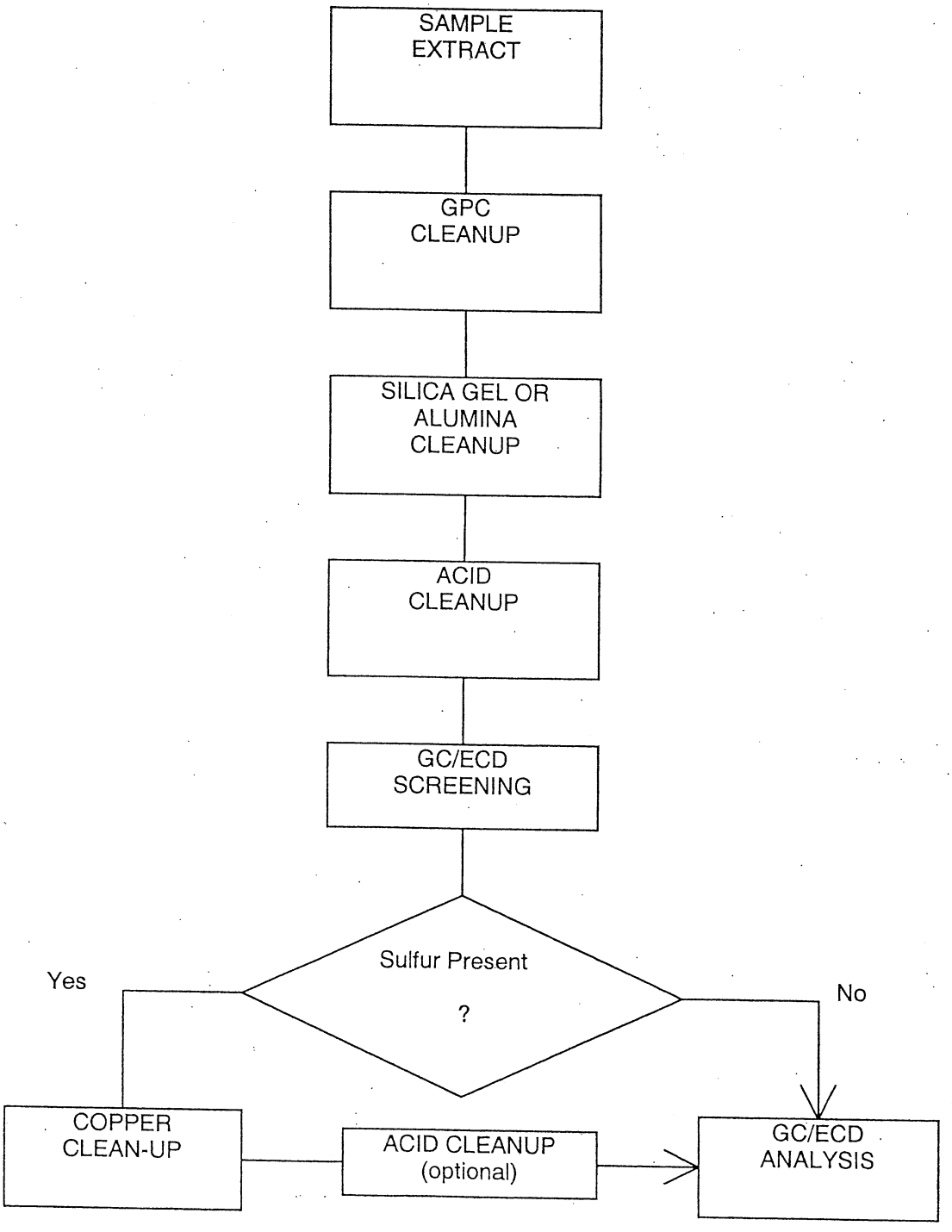


TABLE 1

Target Compound List

Compound Name	PCB I.D.	Water MRL (ng/L)	Water MDL (ng/L)	Soil/Sed MRL (ug/kg)	Soil/Sed MDL (ug/kg)
2,4'-Dichlorobiphenyl	PCB 8	5.0	2	0.50	0.1
2,2',5-Trichlorobiphenyl	PCB 18	5.0	2	0.50	0.05
2,4,4'-Trichlorobiphenyl	PCB 28	5.0	2	0.50	0.07
2,2',5,5'-Tetrachlorobiphenyl	PCB 52	5.0	1	0.50	0.1
2,2',3,5'-Tetrachlorobiphenyl	PCB 44	5.0	2	0.50	0.05
2,3',4,4'-Tetrachlorobiphenyl	PCB 66	5.0	0.8	0.50	0.1
2,3,4,4'-Tetrachlorobiphenyl	PCB 60	5.0	1	0.50	0.07
2,2',3,4',5-Pentachlorobiphenyl	PCB 90	5.0	0.8	0.50	0.05
2,2',4,5,5'-Pentachlorobiphenyl	PCB 101	5.0	0.7	0.50	0.03
3,4,4',5-Tetrachlorobiphenyl	PCB 81	5.0	0.7	0.50	0.1
2,2',3,4,5'-Pentachlorobiphenyl	PCB 87	5.0	2	0.50	0.07
3,3',4,4'-Tetrachlorobiphenyl	PCB 77	5.0	2	0.50	0.05
2',3,4,4',5-Pentachlorobiphenyl	PCB 123	5.0	0.7	0.50	0.08
2,3',4,4',5-Pentachlorobiphenyl	PCB 118	5.0	0.9	0.50	0.04
2,3,4,4',5-Pentachlorobiphenyl	PCB 114	5.0	0.6	0.70	0.3
2,2',3,4,4',6,6'-Heptachlorobiphenyl	PCB 184	5.0	0.6	0.50	0.05
2,2',4,4',5,5'-Hexachlorobiphenyl	PCB 153	5.0	0.5	0.50	0.1
2,3,3',4,4'-Pentachlorobiphenyl	PCB 105	5.0	2	0.50	0.09
2,2',3,4,4',5'-Hexachlorobiphenyl	PCB 138	5.0	0.7	0.50	0.04
2,3,3',4,4',6-Hexachlorobiphenyl	PCB 158	5.0	0.8	0.50	0.04
3,3',4,4',5-Pentachlorobiphenyl	PCB 126	5.0	1	0.50	0.1
2,3,4,4',5,6-Hexachlorobiphenyl	PCB 166	5.0	0.8	0.50	0.1
2,2',3,4',5,5',6-Heptachlorobiphenyl	PCB 187	5.0	0.6	0.50	0.1
2,2',3,4,4',5',6-Heptachlorobiphenyl	PCB 183	5.0	0.7	0.50	0.09
2,2',3,3',4,4'-Hexachlorobiphenyl	PCB 128	5.0	2	0.50	0.3
2,3',4,4',5,5'-Hexachlorobiphenyl	PCB 167	5.0	2	0.70	0.3
2,3,3',4,4',5-Hexachlorobiphenyl	PCB 156	5.0	0.9	0.50	0.09
2,3,3',4,4',5-Hexachlorobiphenyl	PCB 157	5.0	1	0.50	0.2
2,2',3,4,4',5,5'-Heptachlorobiphenyl	PCB 180	5.0	0.6	0.50	0.04
3,3',4,4',5,5'-Hexachlorobiphenyl	PCB 169	5.0	2	0.50	0.1
2,2',3,3',4,4',5-Heptachlorobiphenyl	PCB 170	5.0	0.9	0.50	0.06
2,3,3',4,4',5,5'-Heptachlorobiphenyl	PCB 189	5.0	0.7	0.50	0.09
2,2',3,3',4,4',5,6-Octachlorobiphenyl	PCB 195	5.0	0.7	0.50	0.1
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	PCB 206	5.0	0.6	0.50	0.1
Decachlorobiphenyl	PCB 209	5.0	0.5	0.50	0.06

TABLE 2

Gas Chromatograph Operating Conditions*

Gas Chromatograph:	Hewlett-Packard Model 5890 and 6890 or equivalent w/ECD
Injection Port Temperature:	Cool on-column, oven track
Oven Temperature Program:	60°C for 0.5 minute, 20°/min ramp to 200°C, hold 1 min., ramp 2°/minute to 280° C, hold for 0 minutes, ramp 5°/minute to 310° C, hold for 9.5 minutes.
Detector Temperature:	350°C
Injection Volume:	1μL
Column:	105 m, RTX-5 and 105 m RTX-200.
Carrier Gas:	Helium, programmed: 39.3psi/min. @80°C constant flow
Auxillary Gas:	Argon/Methane
Data System:	HP Enviroquant

* The instrument temperatures may be modified depending on the instrument used. Also, the GC column diameter and film thickness depend on the instrument used. All conditions must be the same for initial calibration, continuing calibration, sample, and QC analyses.

STANDARD OPERATING PROCEDURE

POLYCYCLIC AROMATIC HYDROCARBONS BY GC/MS SELECTIVE ION MONITORING

EPA Method 8270C SIM

SOC-8270CPAH
Revision 0
August 28, 2001

Approved By: _____ _____
Supervisor Date

_____ _____
QA Manager Date

_____ _____
Laboratory Manager Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

POLYCYCLIC AROMATIC HYDROCARBONS BY GC/MS SELECTIVE ION MONITORING
Method 8270C SIM

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentrations of Semi-Volatile Organic Compounds in water, soil, and tissue matrices using EPA Method 8270C SIM. This procedure may also be applicable to various miscellaneous waste samples. Tables 1 and 1A indicates compounds that may be determined by this method and lists their method reporting limits (MRLs) in water, soil, and tissue. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL.

1.2. The procedure is intended for samples containing trace-level amounts of target compounds. Samples containing high concentrations of target analyte will not be analyzed undiluted. All extracts are screened using GC/FID to estimate the hydrocarbon content and concentrations of individual polynuclear aromatic hydrocarbons (PAHs). Samples containing PAHs in excess of five times the high calibration standard will be diluted prior to analysis. All MRLs will be adjusted in accordance with this dilution. Therefore, samples containing high levels of PAHs will not be analyzed to achieve the optimum MRLs for the analysis.

1.3. This procedure can be used to quantitate most neutral organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone phase. This procedure is optimized for the analysis of polynuclear aromatic hydrocarbons.

1.4. Other compounds than those listed in Tables 1 and 1A may be analyzed. However, analytes not summarized in Table 1 have not been validated with a method detection limit study. Therefore, the lab will not use this procedure to analyze for non-routine analytes unless a similar analyte has been validated with a MDL study. As a general rule, the MRL for these compounds will equal the MRL of a similar compound in the routine analyte list. Results will not be reported below this estimated MRL.

2. METHOD SUMMARY

2.1. This method provides Gas Chromatography/Mass Spectrometry (GC/MS) conditions for the detection of Semi-volatile Organic Compounds. Prior to the use of this method, an appropriate sample preparation method must be used to recover the analytes of interest. All soil, tissue, and colored water extracts will be cleaned using EPA Method 3640 (gel permeation chromatography) prior to analysis.

2.2. A 1.0 µL aliquot of the extract is injected into the gas chromatograph (GC). The compounds are separated on a fused silica capillary column. Compounds of interest are detected by a mass selective detector in the selective ion mode. Identification of the analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard, and by comparing mass spectra of analytes with mass spectra of reference materials. Quantitative analysis is performed by using the authentic standard to produce a response factor and calibration curve, and using the calibration data to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume.

3. DEFINITIONS

3.1. **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence The sequence begins with injection of Decafluorotriphenylphosphine (DFTPP) acquired in full scan mode followed by initial calibration standard(s) acquired in SIM mode. Once calibrated, a CCV is evaluated and extracts can be run. The sequence ends after 12 hours based on the DFTPP acquisition time.

3.2. **Matrix Spike/Duplicate Matrix Spike Analysis** - In the matrix spike analysis, predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, then spiked and analyzed. Percent recoveries are calculated for each of the controlled analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.3. **Standard Curve** - A standard curve is a plot of concentrations of a known analyte standard versus the instrument response to the analyte.

3.4. **Surrogate** - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

3.5. **Method Blank** - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.6. **Continuing Calibration Verification Standard (CCV)** - A mid-level standard injected into the instrument at specified intervals and is used to verify the validity of the initial calibration.

3.7. **Second Source Verification Standard or Independent Verification Standard (SSV or ICV)** - A mid-level standard injected into the instrument after the calibration curve from a different source than the standards in the curve and is used to verify the validity of the initial calibration.

3.8. **Selective Ion Monitoring (SIM)** – Mass spectrometry technique where ions resulting from fragmentation are selectively monitrored, therefore excluding other ions. The technique enhances sensitivity as compared to full scan analysis. Because the analysis results in significantly less mass spectral information, this gain in sensitivity is made at the expense of analyte selectivity. Therefore, the use of SIM results in significantly lower instrument detection limits, but increases the uncertainty associated with the analysis.

4. INTERFERENCES

4.1. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation of the samples. Corrective action should be taken to eliminate the interferences.

4.2. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

5. SAFETY

5.1. The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level.

5.2. Follow all applicable safety procedures as described in the CAS Safety Manual. A reference file of material safety data sheets is available to all personnel involved in these analyses. CAS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Certified clean containers should be purchased for sample collection. Alternatively, containers used to collect samples for the determination of semivolatile organic compounds should be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or teflon and have screw-top covers with teflon liners. In situations where teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may <u>not</u> be used

for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

6.2. Water and soil samples should be iced or refrigerated at 4 ± 2°C from time of collection until extraction.

6.3. Water samples must be extracted within 7 days and the extracts analyzed within 40 days following extraction. Soil samples must be extracted within 14 days and the extract analyzed within 40 days following extraction. Extracts are stored at -10°C.

7. APPARATUS AND MATERIALS

7.1. Gas Chromatograph/Mass Spectrometer System

7.1.1. Gas Chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

7.1.2. Column: 5% Dipenyl, 95% Dimethyl Polysiloxane - 30 m x 0.25 mm ID x 0.25 µm film thickness silicone-coated fused-silica capillary column or equivalent. Recommended: Restek XTI-5 with Integra-guard, catalog #12223-124.

7.1.3. Mass Spectrometer - Capable of scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode, and capable of operating in the SIM mode.

7.1.4. GC/MS Interface - Any GC-to-MS interface that gives acceptable calibration points for each compound of interest and achieves acceptable tuning performance criteria may be used.

7.1.5. Data System - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

7.2. Appropriate analytical balance (0.0001 g), volumetric flasks, syringes, vials, and bottles for standards preparation.

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Solvents: Acetone, methylene chloride, methanol, and other appropriate solvents. Solvents must be of sufficient purity to permit usage without lessening the accuracy of the determination or introducing interferences.

8.2. Stock Standard Solutions

8.2.1. Commercially prepared stock standards are typically used when available at a concentration of 100 ug/ml or more. They must be A2LA or ISO9000 certified by the manufacturer. Standard concentrations can be verified by comparison versus an independently prepared standard. Alternatively, prepare stock standard solutions at a concentration of 1000 µg/ml by dissolving 0.0100 g of reference material in methylene chloride or other suitable solvent and diluting to volume in a 10mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard.

8.2.2. Transfer the stock standard solutions into amber Teflon-sealed crimp top autosampler vials and store at -10°C and protect from light, or store as recommended by the manufacturer. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

8.2.3. Unopened stock standards and neat materials have an expiration date equal to the manufacturer's recommendation. Neat material that does not have a manufacturer's recommended expiration date should be replaced after three years. Stock standard solutions received in sealed ampules with manufacture expiration dates in excess of 1 year have an expiration date of 1 year from the date of opening the sealed ampule.

8.3. Internal Standard Solutions - The internal standards are naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} (See Table 3 for corresponding compounds). The nominal concentration of the standard is 100 ng/µL. Each 1 ml of sample extract undergoing analysis should be spiked with 10 µL of the internal standard solution, resulting in a concentration of 1.0 ng/µL of each internal standard. Store at -10°C or less when not being used. When using premixed certified solutions, store according to the manufacturer's recommendations.

8.4. GC/MS Tuning Standard - A methylene chloride solution containing 50 ng/µL of decafluorotriphenylphosphine (DFTPP). The standard should also contain 50 ng/µL of pentachlorophenol, to verify injection port inertness and GC column performance. This injection is acquired in full scan mode and evaluated in accordance with the method

specified criteria. Store at -10°C or less when not being used, or store according to the manufacturer's recommendations.

8.5. Calibration Standards

8.5.1. A minimum of six initial calibration standards should be prepared from stock solutions (note that a seven point calibration is recommended). One of the calibration standards should be at a concentration at or below the method reporting limit. The others should correspond to the range of concentrations found in samples, but should not exceed the working range of the GC/MS system. Each 1 ml aliquot of calibration standards should be spiked with 10 µL of the internal standard solution prior to analysis. All calibration standards should be stored at -10°C or less and should be freshly prepared from stocks every 365 days, or sooner if check standards indicate a problem.

8.5.2. The following calibration standards are recommended: 0.010 ng/µl, 0.040 ng/µl, 0.10 ng/µl, 0.5 ng/µl, 1.0 ng/µl, 2.0 ng/µl, 5.0 ng/µl, and 10 ng/µl

8.5.3. The daily calibration standard (CCV) is prepared at a nominal 2.0 ng/µL concentration from stock solutions. The CCV is prepared weekly and can be stored at 4°C ± 2°C, or as recommended by the manufacturer.

8.6. QC Standards

8.6.1. Surrogates: Prepare a working solution in methanol containing Biphenyl-d8, Fluorene-d10, Fluoranthene-d10, and Terphenyl-d14 at 10 ng/µL. This solution may be combined with the surrogate solution used to monitor analyses for 8270 full list. Aliquots of the solution are spiked into all extracted samples, blanks, and QC samples according to the extraction SOP used.

8.6.2. Matrix Spike Standards: Prepare a working solution in methanol containing all analytes of interest ("full list spike"). All analytes are prepared at 25 ng/µl. Aliquots of the solution are spiked into the selected QC aliquots according to the extraction SOP used.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2. Carrier gas - Inline purifiers or scubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Gas Chromatograph

9.3.1. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, o-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chomatographic performance. In some cases liners and seals may be cleaned and re-used.

9.3.2. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool.

9.3.3. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

9.4. Mass Spectrometer

9.4.1. Tune the MS as needed to result in consistent and acceptable performance while meeting the required ion abundance criteria given in Table 4.

9.4.2. For units under service contract, certain maintenance is performed by instrument service staff, including pump oil changed, vacuuming boards, etc., as recommended by the manufacturer.

9.4.3. MS source cleaning should be performed as needed, depending on the performance of the unit. This may be done by the analyst or by instrument service staff.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in 8270C, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Water, soil, tissue and waste samples are prepared using the appropriate extraction and cleanup methods (refer to SOPs) and screened by GC/FID (see screening SOP). All soil, tissue, waste, and colored water extracts are cleaned using EPA Method 3640 (GPC).

11.1.2. Following sample preparation, sample extracts are then transferred to the extract cold storage unit. Extracts must be analyzed within 40 days of extraction.

11.2. The recommended GC/MS operating conditions:

Ion dwell time:	10 – 50 msec per ion
Initial temperature:	40°C, hold for 1 minutes
Temperature program:	40-140°C at 30°C/min hold for 0 minutes
Temperature program:	140-270°C at 15°C/min hold for 4 minutes
Final temperature:	270-325°C at 20°/min, hold for 3.25 minutes
Injector temperature:	320°C
Detector interface temp:	300°C
Injector:	splitless, electronic pressure control with pulse
Sample volume:	1.0 µL
Carrier gas:	helium at 1.2ml/min (constant flow)

11.3. Selected Ion Acquisition

11.3.1. Determine the ions to be monitored for the compounds of interest. Refer to Table 2 for characteristic ions. At a minimum, 2 ions should be monitored for each compound, and 3 monitored for compounds with more complex fragmentation patterns. Set the SIM windows in order to monitor the correct ions at the correct time, based on chromatographic elution of the compounds. This can be setup by analyzing a standard using a full scan analysis and using the GC conditions of the SIM analysis. This analysis will give retention time and spectral information for determining the location of start times for the SIM groups or windows. This is often referred to as a "locator" analysis.

11.3.2. Select the dwell times to be used for each group of ions to be monitored. Dwell times should be selected in order to provide a sufficient number of measurements across the chromatographic peak to accurately define the peak shape. Too few measurements across the peak will result in poor definition of the peak and

subsequently result in poor accuracy and precision of results. Too many measurements across the peak may result in inconsistent detector behavior over the calibration range. Significant differences in dwell times may also affect sensitivity. Typical dwell times are listed in section 11.2.

11.4. Initial Calibration

Refer to the SOP for *Calibration of Instruments for Organics Chromatographic Analysis*. The calibration procedure(s) and options chosen must follow the CAS SOP. In general, the calibration procedure is as follows:

11.4.1. Prior to calibration, analyze the GC/MS tuning standard using instrument conditions used for calibration. Obtain the spectrum for evaluation using one of the following options:

- Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.
- Use one scan at the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.
- Use one scan either directly preceding or following the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.
- Use the average across the entire peak up to a total of 5 scans. Peak integration must be consistent with standard operating procedure. If the peak is wider than 5 scans, the tune will consist of the peak apex scan and the two scans immediately preceding and following the apex. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.

11.4.2. Evaluate the spectrum obtained for DFTPP against the tuning criteria in Table 4 or 4A (dependent upon instrumentation). The GC/MS must meet the DFTPP ion abundance criteria prior to further analyses. To assess column performance and injection port inertness, pentachlorophenol should be present at an acceptable level and peak tailing should not be excessive. If excessive tailing or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to remove the first 15-30 cm of the GC column. If hardware tuning criteria can not be met, the source may need cleaning, filaments replaced or other maintenance.

11.4.3. The internal standards should permit most of the components of interest in the chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Refer to Table 3 for internal standards and corresponding analytes assigned for quantitation. Use the base peak ion from the specific internal standard as the primary ion for quantitation (See Table 2). If interferences are noted, use the next most intense ion as the quantitaion ion (i.e. for acenaphthene-d_{10}, use 162 m/z for quantitation).

11.4.4. Analyze 1.0 µL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound (as indicated in Table 2). Calculate response factors (RFs) for each compound relative to one of the internal standards as follows:

$$RF = (A_x C_{is})/(A_{is} C_x)$$

where:

A_x = Area of the characteristic ion for compound being measured.
A_{is} = Area of the characteristic ion for specific internal standard.
C_{is} = Concentration of the specific internal standard (ng/µL).
C_x = Concentration of the compound being measured (ng/µL).

11.4.5. A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. The minimum acceptable average RF for these compounds is 0.010. If they are not acceptable, perform GC maintenance (see section 9).

11.4.6. The percent relative standard deviation (%RSD) should be less than 15% for each compound. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units.

$$\%RSD = \frac{SD}{RF} \times 100$$

where:

RSD = relative standard deviation.

RF = mean of 5 initial RFs for a compound.
SD = standard deviation of average RFs for a compound.

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(RF_i - RF)^2}{N - 1}} 1$$

where:

RF_i = RF for each of the 5 calibration levels
N = Number of RF values (i.e., 5)

11.4.7. Linearity - If the % RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

11.4.8. In those instances where the %RSD for one or more analytes exceeds 15%, the initial calibration may still be acceptable if the following conditions are met:

11.4.8.1. The mean of the RSD values for all analytes in the calibration is $\leq$ 15% and the mean %RSD does not exceed 30% for any compound.

11.4.8.2. The mean RSD criteria applies to all target analytes in the calibration standards, regardless of whether or not they are of interest for a specific project.

11.4.8.3. The data user must be supplied with an initial calibration summary indicating the compounds which exceed 15% RSD and the result of the mean RSD calculation.

11.4.9. If all of the conditions in Section 11.4.8 are met, then the average response factor may be used to determine sample concentrations as described in Section 11.4.4.

11.4.10. Following initial calibration, analyze an ICV standard. The ICV solution must contain all analytes in the calibration standards. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. Evaluate the ICV as described in the CAS Organics Calibration SOP.

11.5. Continuing Calibration

11.5.1. Following an acceptable tune, a calibration standard, or standards, at mid-concentration containing all PAH analytes, and all required surrogates, must be analyzed every 12 hours during analysis.

11.5.2. For each daily calibration, a system performance check must be made. For each compound in the daily calibration standard, a minimum response factor of 0.010 must be obtained. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

11.5.3. If the percent drift for each compound of interest is less than or equal to 20%, the initial calibration is assumed to be valid.

11.5.4. The CCV may also be evaluated using the average %D for all analytes in the calibration (in accordance with SW-846 Method 8000B section 7.7). Specifically, the average %D for all analytes in the continuing calibration standard must be less than 15%. Additionally, no individual analyte in the calibration should exhibit a negative bias outside of –30%D.

11.5.5. If the criterion is not met (> 20% drift) for any one compound or >15% for the average responses for all analytes, corrective action must be taken. Problems similar to those listed in 11.5.2 could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new initial calibration must be generated. This criterion must be met before sample analysis begins.

Calculate the percent drift using:

$$\%\ Drift = \frac{C_I - C_c}{C_I} \times 100$$

where:

C_I = Compound standard concentration.
C_c = Measured concentration using selected quantitation method.

11.5.6. The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as required. If the EICP area for any of the internal standards changes by a factor of two (50% to 200%) from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as appropriate. When corrective action is taken, reanalysis of samples analyzed while the system was malfunctioning is required. Update the reference spectra and retention times in the quantitation database for the instrument method or ID file. The initial calibration average RF or calibration curve is then used in the quantitation of subsequent analyses.

11.5.7. A blank (method blank, GPC blank, or solvent blank) should be analyzed after the CCV to prove the system is free of contaminants. If contaminants are found in a method blank or GPC blank, then a solvent blank should be ran to help isolate the source of contamination.

11.6. GC/MS Analysis

11.6.1. Evaluate FID screen and make proper dilution (See FID screening SOP).

11.6.2. Spike the 1 ml extract obtained from sample preparation with 10 µL of the internal standard solution just prior to analysis. Use the same operating conditions as were used for initial calibration.

11.6.3. If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required 1.0ng/µL of each internal standard in the extracted volume. The diluted extract must be reanalyzed.

11.6.4. Store the extracts at -10°C or less, protected from light in vials equipped with unpierced Teflon lined septa. Archive extract in freezer for 3 months, or longer if required by client, after analysis in the instrument/date specific storage boxes.

12. QA/QC REQUIREMENTS

12.1. In addition to instrument criteria for calibration, the ability of each analyst/instrument to generate acceptable accuracy and precision must be documented prior to sample analysis (IPR study). This must be validated before analysis of samples begin, or whenever significant changes to the procedures have been made. To do this, four deionized water

samples are spiked with each target analyte, extracted, and analyzed. Refer to Method 8270C Section 8.3 for this requirement and acceptance criteria.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution (at a level below the MRL) for each target analyte, extract, and analyze. The MDL studies should be done for each matrix, prep method, and instrument. Refer to the CAS SOP *for The Determination of Method Detecion Limits.*

12.2.2. Calculate the average concentration found (x) in the *sample concentration*, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually.

12.3. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for *Analytical Batches and Analytical Sequences*. In general, these include:

12.3.1. Method blank - A method blank is extracted and analyzed with every batch of 20 or fewer samples to demonstrate that there are no method interferences. The method blank must demonstrate that interferences from the analytical and preparation steps minimized. No target analytes should be detected above the MRL in the method blank. For some project specific needs, exceptions may be noted and method blank results above the MRL may be reported for common lab contaminants (phthalate esters, etc.).

12.3.2. A lab control sample (LCS) must be extracted and analyzed with every batch of 20 or fewer samples. The LCS is prepared by spiking a blank with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

Acceptance criteria for lab control samples are listed in Table 5. If the lab control sample (LCS) fails acceptance limits for any of the compounds, the analyst must evaluate the system and calibration. If no problems are found, corrective action must be taken.

12.3.3. A matrix spike/duplicate matrix spike (MS/DMS) must be extracted and analyzed with every batch of 20 or fewer samples. The MS is prepared by spiking a sample aliquot with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{R1 - R2}{(R1 + R2)/2} \times 100$$

Where R1 = % recovery of the MS
R2 = % recovery of the DMS

The acceptance limits for the MS/DMS are given in Table 5. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken.

12.3.4. The acceptance limits for the surrogates are given in Table 5. If any surrogate recovery is outside acceptance criteria, the sample data must be closely evaluated for possible matrix interferences. If none are present, then corrective action must be taken. The sample should be re-analyzed if instrument factors (calibration, injection port) are suspected. If not, re-extraction and re-analysis is required, except in cases of high recovery and no positive hits in the sample for the analyte class represented by the particular surrogate.

12.3.5. Additional QA/QC measures include control charting of QC sample results.

12.3.6. Corrective action – When a data quality objective is not met, the initial corrective action will include a review of the raw data for potential calculation and/or integration errors. If this review does not correct the problem, the following corrective actions will be performed.

12.3.6.1. Method Blank – No target analyte should be detected in a method blank at or above the method reporting limit. If analytes are detected in the method blank, the sample data must be reviewed for possible laboratory contribution. Positive detections of common laboratory contaminants (e.g., phthalate esters) that are between the MRL and three times the MRL do not require reextraction. In this case, flag all detections that are not

more than 20X the amount detected in the blank with a "B" qualifier. Detections of other target analytes, and detection of common laboratory contaminants greater than three times the MRL, require a Nonconformity and Corrective Action Report (NCAR). A decision to reextract the associated samples will depend on the level of the contamination, data quality implications, and the intended use of the data. At a minimum, all positive detections in the associated samples that are not more than 20X the concentration in the blank will be qualified with a "B". Also, as part of the corrective action, the problem will be discussed with the appropriate sample prep personnel in an effort to identify the contamination source.

12.3.6.2. Laboratory Control Sample – The analysis should include a full list LCS spike. All target analytes will be evaluated. The following cases require corrective action:

- If any analytes are recovered less than 10%, the analytical batch should be considered out of control for that analyte. Corrective action includes reinjection to verify the result. If the result is confirmed, a NCAR will be filed. A decision to reextract the associated samples will depend on the data quality implications and the intended use of the data. If reextraction is not feasible, all reported results for that analyte will be qualified and the data quality implications will be discussed in the case narrative.
- In cases where a result is outside the upper control criterion, corrective action is only required if that analyte was also detected in field samples. The associated samples with positive results should be reextracted. In cases where a result is outside the lower control criterion, the associated samples should be reextracted. If reextraction is not feasible, all reported results for that analyte will be qualified and the data quality implications will be discussed in the case narrative.

12.3.6.3. Matrix Spike and Duplicate Matrix Spike Samples – A subset of analytes, as specified in Method 8270C, are monitored in the matrix spike and duplicate matrix spike analyses. If a recovery is outside of control criteria, review the consistency between the two analyses. If the result is supported between the two analyses, the outlier can be attributed to matrix inteference. Do not reanalyze the extract. If the results do not support each other, reanalyze the extracts to verify the results. If the results confirm, review the LCS recovery and take corrective action accordingly. If the LCS recovery is acceptable, flag the matrix spike data and discuss potential data quality implications in the case narrative.

12.3.6.4. Relative Percent Difference – For MS/DMS or LCS/DLCS, no corrective action is required based on RPD data alone. However, the data should be reviewed for information that will help determine if the RPD problem is

the result of a sample specific issue (e.g., the DMS was concentrated to dryness), or if the problem is representative of the entire analytical batch. When the problem is apparently universal to the batch, a NCAR will be filed and the batch will be reextracted. If results of the reextraction confirm the original analyses of the field samples, the original data is reported and the RPD problems are discussed in the case narrative. If results of the reextraction confirm a problem in the original data, only the reextracted data is reported.

12.3.6.5. Surrogates – Corrective action includes reinjection to verify the result. If the result is confirmed, a NCAR will be filed and the sample will be reextracted. If the reextraction confirms the original results are biased due to matrix interferences, report the original data. If reextraction is not feasible, the surrogate will be qualified and the data quality implications will be discussed in the case narrative.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. Qualitative Analysis - The qualitative identification of compounds determined by this procedure is based on retention time, and comparison of the sample mass spectrum with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the instrument and conditions used for the sample analysis. The characteristic ions from the reference mass spectrum are defined to be the ions monitored in the SIM mode and typically are the two or three ions of greatest relative intensity. Compounds are identified as present when the criteria below are met.

13.1.1. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

13.1.2. The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

13.1.3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

13.1.4. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the sum of the 2 peak heights. Otherwise, structural isomers are identified as isomeric pairs.

13.1.5. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When the gas chromatographic peaks appear to represent more than one component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification. When analytes coelute, the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

13.1.6. Evaluate internal standard areas in each sample. If the area in the sample is less than 50% or greater than 200% the area of in the CCV, corrective action must be taken. Depending on the analysis, this corrective action may include reinjection or dilution of the extract followed by reinjection.

13.2. Tentatively identified compound (TIC) can not be reported using this method.

13.3. Quantitation and Calculations

13.3.1. The GC/MS data stations, in current use, all use the H-P RTE Integrator to generate the raw data used to calculate the standards $\overline{RF_x}$ values, the sample amounts, and the spike values. The software does three passes through each data file. The first two identify and integrate each internal standard and surrogate. The third pass uses the time-drift information from the first two passes to search for all method analytes in the proper retention times and with the proper characteristic quantitation ions. When $\overline{RF_x}$ is used, calculate the extract concentration as follows:

$$C_{ex} = \frac{(Resp_x)(Amt_{ISTD})}{(Resp_{ISTD})(\overline{RF_x})}$$

Where: C_{ex} = the concentration in the sample extract (ppm);
$Resp_x$ = the peak area of the analytes of interest;
$Resp_{ISTD}$ = the peak area of the associated internal standard;
Amt_{ISTD} = the amount, in ppm, of internal standard added
$\overline{RF_x}$ = the average response from the initial calibration.

13.3.2. The concentration of analytes in the original sample is computed using the following equations:

Aqueous Samples: $$Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$$

Where Cex = Concentration in extract in µg/mL

Vf	=	Final volume of extract in mL
D	=	Dilution factor
Vs	=	Volume of sample extracted, liters

Nonaqueous Samples: $Concentration\ (mg / Kg) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$

Where	Cex	=	Concentration in extract in µg/mL
	Vf	=	Final volume of extract in mL
	D	=	Dilution factor
	W	=	Weight of sample extracted in grams.

13.4. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for *Laboratory Data Review Process* for details.

13.5. Reporting

13.5.1. Reports are generated in the CAS LIMS by compiling the SMO login, sample prep database, instrument, date, and client-specified report requirements (when specified). This compilation is then transfered to a file which the Stealth reporting system uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.5.2. As an alternative, reports are generated using Excel© templates located in R:\SVM\forms. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The detected analytes, surrogate and matrix spikes are then transferred, by hand or electronically, to the templates.

14. CONTINGENCIES FOR HANDLING OUT-OF- CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for *Nonconformity and Corrective Action* for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for *Report Generation* or in project-specific requirements.

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP for *the Determination of Method Detection Limits* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the laboratory Safety Manual, Sections 6 and 7.

18. REFERENCES

Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry, Method 8270C, EPA Test Methods for Evaluating Solid Waste, SW-846, Final Update III, December 1996.

TABLE 1

Target Analytes, Method Reporting Limits, and Method Detection Limits

	WATER µg/L (ppb)		SOIL µg/Kg Dry Weight (ppb)		Tissue µg/Kg Wet Weight (ppb)	
Analytes	**MRL**	**MDL**	**MRL**	**MDL**	**MRL**	**MDL**
Naphthalene	0.02	0.008	5	0.8	80	40
2-Methylnaphthalene	0.02	0.008	5	0.3	80	40
1-Methylnaphthalene	0.02		5			
Biphenyl	0.02		5			
2,6-Dimethylnaphthalene	0.02		5			
Acenaphthylene	0.02	0.006	5	0.3	80	30
Acenaphthene	0.02	0.007	5	3	80	30
Dibenzofuran	0.02	0.007	5	0.3	80	30
2,3,5-Trimethylnaphthalene	0.02		5			
Fluorene	0.02	0.006	5	0.4	80	30
Dibenzothiophene	0.02	0.02	5	0.6	80	30
Phenanthrene	0.02	0.007	5	2	80	20
Anthracene	0.02	0.006	5	0.5	80	20
1-Methylphenanthrene	0.02	0.03	5	2	80	30
Fluoranthene	0.02	0.007	5	0.6	80	30
Pyrene	0.02	0.007	5	0.5	80	30
Benz(a)anthracene	0.02	0.005	5	0.4	80	20
Chrysene	0.02	0.006	5	0.4	80	20
Benzo(b)fluoranthene	0.02	0.005	5	0.5	80	20
Benzo(k)fluoranthene	0.02	0.003	5	0.5	80	20
Benzo(e)pyrene	0.02		5			
Benzo(a)pyrene	0.02	0.006	5	0.5	80	20
Perylene	0.02		5			
Indeno(1,2,3-cd)pyrene	0.02	0.004	5	0.3	80	30
Dibenz(a,h)anthracene	0.02	0.004	5	0.5	80	30
Benzo(g,h,i)perylene	0.02	0.005	5	0.7	80	20

TABLE 1A

Additional Analytes

Target Analytes and Method Reporting Limits

	WATER µg/L (ppb)		SOIL µg/Kg Dry Weight (ppb)		Tissue µg/Kg Wet Weight (ppb)	
Analytes	**MRL**		**MRL**		**MRL**	
C2-Naphthalenes	0.02		5		80	
C3-Naphthalenes	0.02		5		80	
C4-Naphthalenes	0.02		5			
C1-Fluorenes	0.02		5		80	
C2-Fluorenes	0.02		5		80	
C3-Fluorenes	0.02		5		80	
C1-Dibenzothiophenes	0.02		5		80	
C2-Dibenzothiophenes	0.02		5		80	
C3-Dibenzothiophenes	0.02		5		80	
C1-Phenanthrenes/Anthracenes	0.02		5		80	
C2-Phenanthrenes/Anthracenes	0.02		5		80	
C3-Phenanthrenes/Anthracenes	0.02		5		80	
C4-Phenanthrenes/Anthracenes	0.02		5		80	
C1-Fluoranthenes/Pyrenes	0.02		5		80	
C1-Chrysenes	0.02		5			
C2-Chrysenes	0.02		5		80	
C3-Chrysenes	0.02		5			
C4-Chrysenes	0.02		5		80	

TABLE 2
TARGET COMPOUNDS AND CORRESPONDING PRIMARY AND SECONDARY IONS

Compound	Approximate Retention Time (min)	Primary Ion	Secondary Ion
Naphthalene-d 8 (I.S.)	5.86	136	68
Naphthalene	5.87	128	127
2-Methylnaphthalene	6.62	141	142
1-Methyl naphthalene	6.74	141	142
Biphenyl	7.18	154	153
2,6-Dimethylnaphthalene	7.37	156	155
Acenaphthlene-d 10 (I.S.)	7.95	164	162
Acenaphthylene	7.75	152	153
Acenaphthlene	8.00	154	153
Dibenzofuran	8.22	168	139
2,3,5-Trimethylnaphthalene	8.53	170	155
Fluorene	8.70	166	165
Phenanthrene-d 10 (I.S.)	10.10	188	94
Dibenzothiophene	9.95	184	152
Phenanthrene	10.13	178	179
Anthracene	10.20	178	176
1-Methylphenanthrene	11.10	192	150
Fluoranthene	11.99	202	203
Chrysene-d 12 (I.S.)	14.51	240	236
Pyrene	12.35	202	203
Benz (a) anthracene	14.48	228	226
Chrysene	14.57	228	226
Perylene-d 12 (I.S.)	18.48	264	260
Benzo(b)fluoranthene	17.50	252	126
Benzo(k)fluoranthene	17.56	252	126
Benzo(e)pyrene	18.22	252	126
Benzo(a)pyrene	18.33	252	126
Perylene	18.52	252	126
Indeno (1,2,3-cd) pyrene	20.42	276	277
Dibenz (a, h) anthracene	20.47	278	276
Benzo(g,h,i)perylene	20.86	276	277
Biphenyl-d8 (surr.)	7.15	162	163
Fluorene-d10 (surr.)	8.67	176	175
Fluoranthene-d10 (surr.)	11.97	212	213
Terphenyl-d 14 (surr.)	12.63	244	122

TABLE 3

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

Naphthalene-d8

Naphthalene
2-Methylnaphthalene
1-Methylnaphthalene
Biphenyl-d8 (surr.)
Biphenyl
2,6-Dimethylnaphthalene

Acenaphthene-d10

Acenaphthene
Acenaphthylene
Dibenzofuran
2,3,5-Trimethylnaphthalene
Fluorene
Fluorene-d_{10} (surr.)

Phenanthrene-d10

Dibenzothiophene
Phenanthrene
Anthracene
1-Methylphenanthrene
Fluoranthene
Fluoranthene-d_{10} (surr.)

Chrysene-d12

Pyrene
Benzo(a) anthracene
Chrysene
Terphenyl-d_{14} (surr.)

Perylene-d12

Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(e)pyrene
Benzo(a)pyrene
Perylene
Indeno (1,2,3-ccd)pyrene
Dibenz(a,h)anthracene
Benzo(g,h,i)perylene

TABLE 4
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA
for **5973** GC/MS systems

Mass	Ion Abundance Criteria
51	10-80% of mass 198
68	0-2% of mass 69
70	0-2% of mass 69
127	10-80% of 198
197	0-2% of 198
198	30-100% of 442 (alternate base)
199	5-9% of 198
275	10-60% of 198
365	1-50% of 442
441	0.01-100% of 443
442	30-100% of 198 (alternate base)
443	15-24% of 442

TABLE 4A
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA
for **5972** GC/MS systems

Mass	Ion Abundance Criteria
51	30-80% of mass 198
68	0-2% of mass 69
69	Present
70	0-2% of mass 69
127	25-75% of 198
197	0-1% of 198
198	100% Relative abundance, Base Peak
199	5-9% of 198
275	>0.75% of 198
365	1-50% of 442
441	0.01-99.99% of 443
442	40-110% of 198
443	15-24% of 442

Table 5
QC Acceptance Criteria

		Laboratory Control Sample		Matrix Spike	
Compound	**Control/ Advisory**	**Water (%rec)**	**Soil (%rec)**	**Water (%rec)**	**Soil (%rec)**
Naphthalene	c	37-100	34-125	45-135	10-158
2-Methylnaphthalene	c	46-105	46-147	45-135	10-140
1-Methylnaphthalene	a	45-135	45-135	45-135	45-135
Biphenyl	a	45-135	45-135	45-135	45-135
2,6-Dimethylnaphthalene	a	45-135	45-135	45-135	45-135
Acenaphthylene	c	45-111	45-123	45-135	34-138
Acenaphthene	c	44-109	45-123	28-151	16-147
Dibenzofuran	c	48-112	56-116	45-135	18-155
2,3,5-Trimethylnaphthalene	a	45-135	45-135	45-135	45-135
Fluorene	c	52-112	57-117	45-135	43-139
Dibenzothiophene	a	45-135	45-135	45-135	45-135
Phenanthrene	c	62-111	63-115	45-135	10-192
Anthracene	c	57-112	56-127	45-135	27-152
1-Methylphenanthrene	a	45-135	45-135	45-135	45-135
Fluoranthene	c	68-118	54-132	45-135	28-166
Pyrene	c	55-128	62-128	31-124	28-162
Benz(a)anthracene	c	62-124	63-128	45-135	64-145
Chrysene	c	66-121	66-128	45-135	54-128
Benzo(b)flouranthene	c	45-141	46-137	45-135	64-138
Benzo(k)fluoranthene	c	49-144	50-142	45-135	62-130
Benzo(e)pyrene	a	45-135	45-135	45-135	45-135
Benzo(a)pyrene	c	45-137	50-138	45-140	57-149
Perylene	a	45-135	45-135	45-135	45-135
Inden(1,2,3-cd)pyrene	c	33-153	46-145	45-135	52-156
Dibenz(a,h)anthracene	c	42-152	55-153	45-135	61-144
Benzo(g,h,i)perylene	c	37-136	57-129	45-135	40-127

		Surrogate	**Surrogate**
Compound	Control/ Advisory	Water (%rec)	Soil (%rec)
Biphenyl-d_8	a		
Fluorene-d_{10}	c	31-97	42-130
Flouranthene-d_{10}	c	31-113	55-130
Terphenyl-d_{14}	c	30-115	60-119

SOC-8270P SOP ADDENDUM : Procedure for quantifying alkylated homologs of polynuclear aromatic hydrocarbons.

1. Analyze a PAH ICAL using the A_PAHK method. These acquisition parameters will acquire data for the parent PAHs as well as the homologs.

Process the data and update the calibration table. Go into EDIT COMPOUNDS which is under the INITCAL menu. Enter the peak areas from the parent compounds into the corresponding levels in the associated homologs calibration table as listed below.

PARENT	HOMOLOG
Naphthalene	C2-Naphthalenes
	C3-Naphthalenes
	C4-Naphthalenes
Fluorene	C1-Fluorenes
	C2-Fluorenes
	C3-Fluorenes
Dibenzothiophene	C1-Dibenzothiophenes
	C2-Dibenzothiophenes
	C3-Dibenzothiophenes
Phenanthrene	C1-Phenanthrenes/Anthracenes
	C2-Phenanthrenes/Anthracenes
	C3-Phenanthrenes/Anthracenes
	C4-Phenanthrenes/Anthracenes
Pyrene	C1-Fluoranthenes/Pyrenes
Chrysene	C1-Chrysenes
	C2-Chrysenes
	C3-Chrysenes
	C4-Chrysenes

Use the same Curve Fit for the Homolog as is used for the Parent compound. Use the average RF if the %RSD is less than 20%. Use a quadratic curve if the %RSD exceeds 20%.

2. Analyze the PAH-ALKH SRM. This SRM is a petroleum crude oil which has been cleaned up using GPC and silica gel cleanup procedures. All of the homologs are present in the SRM.

Process the SRM data against the ICAL. Review the SRM data in QEDIT and manually integrate the homologs using the example in the SOP book as a guide. Print a graphics report for each homolog.

3. Update the calibration table using the PAH CCV. Enter the average retention time for each of the homologs into the processing method.

4. Analyze the sample extracts and process the data against the ICAL. Using the SRM data as a guide, manually integrate the homologs. Print graphics reports for each sample hit.

United States
Environmental Protection Agency
Office of Water

Office of Science and Technology
Health and Ecological Criteria Div.
Washington, DC 20460

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WATER

Draft Analytical Method

for Determination of Acid

Volatile Sulfide in Sediment

DETERMINATION OF ACID VOLATILE SULFIDES AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENT

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DETERMINATION OF ACID VOLATILE SULFIDES AND SIMULTANEOUSLY EXTRACTABLE METALS IN SEDIMENT

1. SCOPE AND APPLICATION

1.1 This method describes procedures for the determination of acid volatile sulfides (AVS) and for metals that are solubilized during the acidification step (simultaneously extracted metal, SEM). The conditions used have been reported to measure amorphous or moderately crystalline monosulfides (1). As a precipitant of toxic heavy metals, sulfide is important in controlling the bioavailability of metals in anoxic sediments (2). If the molar ratio of toxic metals measured by SEM to AVS exceeds one, the metals are potentially bioavailable. Because the relative amounts of AVS and SEM are important in the prediction of potential metal bioavailability, it is important to use the SEM procedure for sample preparation for metal analysis. This uses the same conditions for release of both sulfide and metal from the sediment and thus provides the most predictive means of assessing the amount of metal associated with sulfide.

2. SUMMARY OF METHOD

2.1 The AVS in the sample is first converted to hydrogen sulfide (H_2S) by acidification with hydrochloric acid at room temperature. The H_2S is then purged from the sample and trapped. The amount of sulfide that has been trapped is then determined. The SEM are metals liberated from the sediment during the acidification. These are determined after filtration of the sample.

2.2 Two types of apparatus for sample purging and trapping of H_2S are described. One uses a series of Erlenmeyer flasks while the other uses flasks and traps with ground glass stoppers. The former is less costly. The latter is less prone to leakage that causes low recovery of AVS. The latter is recommended when higher degrees of precision are desired and for samples containing low levels of AVS.

2.3 Three means of quantifying the H_2S released by acidifying the sample are provided. In the gravimetric procedure, the H_2S is trapped in silver nitrate. The silver sulfide that is formed is determined by weighing (2,3). This procedure is recommended for samples with moderate or high AVS concentrations. In the colorimetric method, the H_2S is trapped in sodium hydroxide. The sulfide is converted to methylene blue that is measured (4). This procedure is recommended for samples that have low to moderate AVS concentrations. In an alternative procedure the H_2S is trapped in an antioxidant buffer before using an ion-selective electrode (5,6).

2.4 After release of the H_2S, the acidified sediment sample is membrane filtered before determination of the SEM by atomic absorption or inductive coupled plasma spectrometic methods (7,8).

3. DEFINITIONS

3.1 ACID VOLATILE SULFIDES (AVS) - Amorphous, moderately crystalline monosulfides, and other sulfides that form hydrogen sulfide under the conditions of this test.

3.2 SIMULTANEOUSLY EXTRACTED METALS (SEM) - Metals, commonly cadmium, copper, lead, mercury, nickel and zinc, which form less soluble sulfides than do iron or manganese, and which are at least partially soluble under the conditions of this test.

3.3 METHOD DETECTION LIMIT (MDL) - The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined from the analysis of a sample that contains the analyte within a given matrix.

3.4 LABORATORY REAGENT BLANK (LRB) - An aliquot of reagent water or reagents that is treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, reagents or apparatus.

3.5 STOCK STANDARD SOLUTION - A concentrated solution of the analyte prepared in the laboratory using assayed reference compounds or purchased from a reputable commercial source.

3.6 CALIBRATION STANDARDS (CAL) - Solutions prepared from the stock standard solution that is used to calibrate the method response with respect to analyte concentration.

3.7 LABORATORY FORTIFIED BLANK (LFB) - An aliquot of reagent water or reagents to which a known quantity of the method analyte is added in the laboratory. The LFB is analyzed exactly like a sample. Its purpose is to determine whether the method is within accepted control limits.

3.8 LABORATORY FORTIFIED SAMPLE MATRIX (LFM) - An environmental sample to which a known quantity of the method analyte is added in the laboratory. The LFM is analyzed exactly line a sample. Its purpose is to determine whether the sample matrix contributes bias to the analytical results.

4. INTERFERENCES

4.1 Oxygen in the reagents and apparatus is the primary interference reported. Special precautions must be taken to insure that the analytical system is adequately purged with oxygen-free nitrogen. Argon may be substituted for nitrogen if it is important that an even lower level oxygen concentration be maintained (4).

4.2 The pH of the sample after the addition of the acid and during the purge process must be below 3. Typically, the pH is below 2.

5. SAFETY

5.1 The toxicity or carcinogenicity of reagents used in this method have not been fully established. Each chemical and environmental sample should be regarded as a potential health hazard and exposure should be minimized. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets should be available to all personnel involved in the chemical analysis.

5.2 Hydrogen sulfide is a highly poisonous, gaseous compound having a characteristic odor of rotten eggs. It is detectable in air by humans at a concentration of approximately 0.002 ppm. Handling of acid samples should be performed in a hood or well ventilated area. If hydrogen sulfide is detected in the air by the laboratory staff, sample handling procedures must be corrected.

6. APPARATUS AND EQUIPMENT

6.1 Glassware

6.1.1 AVS evolution and H_2S trapping - Glassware in Section 6.1.1.1 is recommended. Glassware in Section 6.1.1.2 may be used, but will not provide as high precision or accuracy for samples.

6.1.1.1 For highest precision and low AVS levels - For each analytical train 500 mL gas washing bottles, one 250 mL round bottom flask with a septum (Ace Glass 6934 or equivalent), 100 or 250 mL impingers. The round bottom flask contains the sediment and acid is introduced to it by a syringe inserted through the septum. The flasks are connected by plastic tubing. In all cases the inlets are below the liquid level and the outlets are above the liquid levels. The apparatus is assembled as shown in Figure 1 and more than one analytical train can be connected to a single cylinder of nitrogen if flow controllers are installed in the

line. Different amounts of glassware are required for each of the three means of sulfide determination.

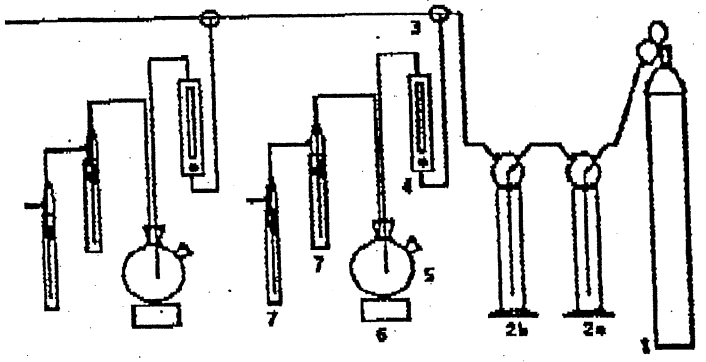


Figure 1. Apparatus for AVS determination: 1.N_2 cylinder; 2.Gas washing bottles: (a) oxygen scrubbing solution, (b) deionized water; 3.Three-way stopcock; 4.Purge flow controller; 5.Reaction flask; 6.Magnetic stirrer; 7.Sulfide traps.

6.1.1.2 For routine analysis - Erlenmeyer flasks, 250 mL, are substituted for the gas washing bottle, the round bottom flask and the impingers. The flask size should be consistent with sample size and reagent volumes. A thistle tube fitted with a stopcock or a separatory funnel is provided to introduce acid to the flask containing the sediment sample. This flask is fitted with a three hole stopper. One hole is for the thistle tube or separatory funnel and the other two are for the gas inlet and outlet. The other flasks are fitted with two hole stoppers; one hole is for the gas inlet and the other is for the gas outlet. The gas inlets are below the liquid level and the gas outlets are above the liquid level. The flasks are connected by plastic tubing.

6.1.2 Evaporating dishes, porcelain, 100 mL.

6.1.3 Assorted calibrated pipettes and volumetric flasks.

6.2 Drying oven - Capable of maintaining a constant temperature in the range of 100-104°C.

6.3 Analytical balance - of weighing to 0.0001 g.

6.4 Magnetic stirrer, thermally insulated, and Teflon-coated stirring bar.

6.5 Gravimetric method

6.5.1 Filtering flask.

6.5.2 Filter holder for 47 mm filter.

6.6 Colorimetric method

6.6.1 Spectrophotometer - Capable of measuring absorbance at 670 nm.

6.6.2 Spectrophotometer cells.

6.7 Ion-selective electrode method

6.7.1 Electrometer, pH meter or ion-selective meter - Compatible with the use of ion-selective electrodes.

6.7.2 Sulfide selective electrode.

6.8 Atomic absorption or inductive couple plasma spectrophometer for the determination of SEM.

7. REAGENTS AND CONSUMABLE MATERIALS

7.1 All water and reagents used in this method must be free of dissolved oxygen and sulfides. Freshly prepare and use deaerated, deionized water, DDIW, by purging dissolved oxygen from the deionized water by vigorously bubbling with oxygen free nitrogen for approximately one hour. Deaerate reagents by purging with nitrogen.

7.2 Sodium sulfide standard - Required for quality assurance and calibration.

7.2.1 Sulfide stock standard solution, approximately 0.05M or 50 µmoles/mL.

7.2.1.1 Place a few crystals of $Na_2S \cdot 9H_2O$ in a beaker. Wash them with distilled water and dry them by blotting with filter paper. Weigh about 12 gram of $Na_2S \cdot 9H_2O$ and dissolve it in 1,000 mL of DDIW. Store in a brown bottle.

7.2.1.2 Standardize against thiosulfate solution.

7.2.1.2.1 Pipette 10.00 mL of 0.025N standard iodine solution (Section 7.2.2) into each of two 125-mL Erlenmeyer flasks.

7.2.1.2.2 Pipette 2.00 mL of sulfide stock standard solution into one flask. Pipette 2.00 mL of DDIW, as a laboratory reagent blank, into the other flask.

7.2.1.2.3 Add 5.00 mL of 6M HCl into each flask, swirl slightly, then cover and place in the dark for 5 minutes.

7.2.1.2.4 Titrate each with 0.025N thiosulfate (Section 7.2.3), adding soluble starch indicator when the yellow iodine color fades. The end point is reached when the blue color disappears.

7.2.1.2.5 Calculate the sulfide concentration as follows:

$$\text{Sulfide } (\mu\text{mol / mL}) = \frac{(T_{blank} - T_{sample}) \times N_{S_2O_3^{2-}}}{V_{sample}} \times \frac{1 \text{ mole } S^{2-}}{2 \text{ equiv } S^{2-}} \times \frac{1000 \ \mu\text{moles}}{1 \text{ mmole}}$$

where T = volume of titrant used for the blank and sample (mL)

N = concentration of $S_2O_3^{2-}$ titrant

V = volume of sample used (2.00 mL)

7.2.2 Standard iodine solution, 0.025N - Dissolve 20 to 25 gram potassium iodide, KI, in a small volume of deionized water, add 3.2 gram iodine, and dilute to 1,000 mL. Standardize against 0.025N sodium thiosulfate (Section 7.2.3) using starch solution as indicator.

7.2.3 Standard sodium thiosulfate solution, 0.025N. May be purchased commercially or prepared in the laboratory. If prepared in the laboratory, it should be standardized against potassium dichromate.

7.2.3.1 Weigh approximately 6.2 g of sodium thiosulfate, $Na_2S_2O_3 \cdot 5\ H_2O$, into a 500 mL beaker. Add 0.1 g sodium carbonate, Na_2CO_3, and dissolve in 400 mL deionized water. Pour into a 1.0 L volumetric flask and dilute to volume with deionized water.

7.2.3.2 Standardize against potassium dichromate, K_2CrO_7.

7.2.3.2.1 Accurately weigh approximately 0.2 g dry K_2CrO_7 and place in a 500 mL Erlenmeyer flask. Dissolve in 50 mL deionized water.

7.2.3.2.2 Dissolve 3 g of potassium iodide, KI, in 50 mL distilled water, add 5 mL of 6M HCl, and add to KI solution. Swirl, cover and store in the dark for 5 minutes. Add 200 mL deionized water and titrate with the thiosulfate solution, adding starch indicator when the yellow iodine color fades, until the blue color turns to pale green.

7.2.3.2.3 Calculate the thiosulfate concentration as follows:

$$N\left(S_2O_3^{2-}\right) = \frac{g\ K_2CrO_7}{mL\ S_2O_3^{2-}} \times \frac{1\ mole\ K_2CrO_7}{294.19\ g\ K_2CrO_7} \times \frac{6\ equiv\ K_2CrO_7}{1\ mole\ K_2CrO_7} \times \frac{1000\ mL}{1\ L}$$

7.2.4 Starch indicator - Dissolve 1.0 gram soluble starch in 100 mL boiling deionized water.

7.2.5 Sulfide working standards - Prepare sulfide working standards using the sulfide stock standard solution in Section 7.2.1. The concentrations of the following standards will depend on the exact concentration of the sulfide stock standard determined in Section 7.2.1.2.5. Correct concentrations of the the standards in the following part of this section and the amount of sulfide in standards used in the colorimetric method in Section 12.2.5 by multiplying by a factor of the concentration determined in Section 7.2.1.2.5 divided by 50 µmoles/mL.

7.2.5.1 Prepare sulfide working standard A by diluting 1.00 mL of sulfide stock standard to 100.0 mL. This solution contains 0.5 µmole sulfide/mL, if the concentration of the sulfide stock standard is exactly 0.05M.

7.2.5.2 Prepare sulfide working standard B by diluting 10.00 mL of sulfide stock standard to 100.0 mL. This solution contains 5.0 µmole sulfide/mL, if the concentration of the sulfide stock standard is exactly 0.05M.

7.3 AVS evolution

7.3.1 Hydrochloric acid 6M - Dilute 500 mL of concentrated hydrochloric acid (sp. gr. 1.19) to 1L with deionized water.

7.3.2 Nitrogen gas, oxygen free, with regulator and flow controller. An oxygen gas scrubber may be required and is available commercially or deoxygenating solutions may be placed in the flask or gas washing bottle placed first in the analytical train.

7.3.3 Plastic hypodermic syringe, 30 mL, and needle.

7.4 Gravimetric method

7.4.1 Potassium acid phthalate, 0.05M - Dissolve 10.2 g of potassium acid phthalate, $KHC_8H_4O_4$, in DDIW and dilute to 1L.

7.4.2 Silver nitrate, 1M - Dissolve 17 g of silver nitrate, $AgNO_3$, in DDIW and dilute to 1L. Store in a dark bottle.

7.4.3 Glass fiber filters, 1.2 micron - Predry filters at 102°C.

7.5 Colorimetric method

7.5.1 Sodium hydroxide solution, 1M - Dissolve 40 g sodium hydroxide in 1000 mL DDIW.

7.5.2 Sodium hydroxide solution, 0.5M - Dissolve 20 g sodium hydroxide in 1000 mL DDIW.

7.5.3 Mixed diamine reagent, MDR

7.5.3.1 Component A - Add 660 mL concentrated sulfuric acid to 340 mL of DDIW. After the solution cools, dissolve 2.25 g N-N-dimethy-p-phenylenediamine oxalate in it.

7.5.3.2 Component B - Dissolve 5.4 g ferric chloride hexahydrate ($FeCl_3 \cdot 6 H_2O$) in 100 mL concentrated hydrochloric acid and dilute to 200 mL with DDIW.

7.5.3.3 Mixed diamine reagent, MDR - Mix components A and B.

7.5.4 Sulfuric acid solution, 1.0M - Dilute 56 mL concentrated sulfuric acid (H_2SO_4) to 1 L with DDIW.

7.6 Ion-selective electrode method

7.6.1 Sodium hydroxide solution - Dissolve 80 g of sodium hydroxide in 700 mL of DDIW with caution. Cool to room temperature.

7.6.2 Sulfide anti-oxidant buffer (SAOB) - To the sodium hydroxide solution in Section 7.6.1 add and dissolve 74.45 g of disodium ethylenediaminetetraacetic acid and 35.23 g of ascorbic acid. Dilute to 1L with DDIW.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Sulfide ion is unstable in the presence of oxygen. Protect sediment samples from exposure to oxygen during sample collection and storage.

8.2 During storage sulfides can be formed or lost due to biological activity and sulfide can be lost by volatilization or oxidation. Metal speciation can change as a result of changes in sulfide concentration and as a result of other changes in the sample.

8.3 Samples should be collected in wide mouth jars with a minimum of air space above the sediment. If possible, the headspace should be purged with oxygen free nitrogen. The jars must have Teflon or polyethylene liners.

8.4 Samples should be cooled to 4°C as soon as possible after collection. Samples maintained at 4°C have been found to have no significant loss of AVS for storage periods up to 2 weeks (4). Anoxic sediments stored at 4°C for 20 days show significant changes in metal partitioning, suggestive of oxidation of the sediment (9). Holding time for samples should not exceed 14 days.

9. CALIBRATION AND STANDARDIZATION

9.1 Calibrate the photometer with a minimum of four standards and a blank that cover the expected range of the samples. Prepare a calibration graph relating absorbance to the µmoles of sulfide taken.

9.2 Calibrate the sulfide electrode system with a minimum of three standards that cover the expected range of the samples. Standards must be made up in SAOB diluted 1+1 with DDIW. Follow the manufacturer's instructions for use of the electrode.

9.3 Overall sulfide recovery is determined by analysis of a known amount of sodium sulfide standard added to DDIW from which the sulfide is liberated in the analysis train (LFB). Recoveries of 95% ± 10% are expected.

10. QUALITY CONTROL

10.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirement of this program consists of an initial demonstration of laboratory capability, and the analysis of laboratory reagent blanks, fortified blanks and samples as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of data thus generated.

10.2 INITIAL DEMONSTRATION OF PERFORMANCE

10.2.1 The initial demonstration of performance is used to characterize instrument performance, method detection limits, and linear calibration ranges.

10.2.2 Method detection limit (MDL) - The method detection limit should be established for the analyte, using DDIW (blank) fortified at a concentration two to five times the estimated detection limit (10). To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$MDL = t \times s$$

where, t = students' t value for a 99% confidence level and a standard deviation

estimate with n-1 degrees of freedom (t = 3.14 for seven replicates), and

s = standard deviation of the replicate analyses.

Method detection limits should be determined every six months or whenever a significant change in background or instrument response is expected.

10.2.3 Linear calibration ranges - The upper limit of the linear calibration range should be established by determining the signal responses from a minimum of four different concentration standards covering the expected range, one of which is close to the upper limit. The linear calibration range that may be used for the analysis of samples should be judged by the analyst from resulting data. Linear calibration ranges should be determined every six months or whenever a significant change in instrument response may be expected.

10.3 ASSESSING LABORATORY PERFORMANCE - REAGENT AND FORTIFIED BLANKS

10.3.1 Laboratory reagent blank (LRB) - The laboratory must analyze at least one reagent blank (3.4) with each set of samples. Reagent blank data are used to assess contamination from the laboratory environment and reagents. If an analyte value in the reagent blank exceeds its determined MDL, then laboratory or reagent contamination should be suspected. Any determined source of contamination should be corrected and the samples reanalyzed.

10.3.2 Laboratory fortified blank (LFB) - The laboratory must analyze at least one fortified blank (3.7) with each batch of samples. Calculate accuracy as percent recovery. If the recovery of the analyte falls outside the control limits (see 10.3.3), the analyte is judged to be out of control, and the source of the problem should be identified and resolved before continuing analyses.

10.3.3 Until sufficient data become available from within their own laboratory (usually a minimum of twenty to thirty analyses), the laboratory should assess laboratory performance against recovery limits of 85-105%. When sufficient internal performance data becomes available, develop control limits from the mean recovery (x) and the standard deviation (s) of the mean recovery. These data are used to establish upper and lower control limits as follows:

$$\text{UPPER CONTROL LIMIT} = x + 3s$$

$$\text{LOWER CONTROL LIMIT} = x - 3s$$

After each five to ten new recovery measurements, new control limits should be calculated using only the most recent twenty to thirty data points.

10.4 ASSESSING ANALYTE RECOVERY - LABORATORY FORTIFIED SAMPLE MATRIX

10.4.1 The laboratory must fortify a minimum of 10% of the routine samples or one fortified sample per set, whichever is greater. Ideally, the concentration should at least double the background concentration. Over time, samples from all routine sample sources should be fortified.

10.4.2 Calculate the percent recovery for the analyte, corrected for background concentrations measured in the unfortified sample, and compare these values to the control limits established in Section 10.3.3 for the analyses of LFBs. Spike recovery calculations are not required if the spike concentration is less than 10% of the sample background concentration. Percent recovery may be calculated in units appropriate to the matrix, using the following equation:

$$R = \frac{(C_s - C_b)}{S} \times 100$$

where

R = percent recovery,

C_s = fortified sample concentration,

C_b = sample background concentration, and

S = concentration equivalent of the fortified sample.

10.4.3 If the recovery of the analyte in the fortified sample falls outside the designated range, and the laboratory performance on the LFB for the analyte is shown to be in control (Section 10.3) the recovery problem encountered with the fortified sample is judged to be matrix related, not system related.

11. GENERATION OF H_2S

11.1 Assemble glassware according to the detection method to be used. The setup in Figure 1 should be followed as a general guide. In all cases a flask or gas washing bottle containing a deoxygenating solution may be placed in the sample train between the nitrogen tank and the first flask. Glassware is specified in Section 6.1.1. It is recommended that nitrogen be controlled by a flow controller, but an equivalent flow rate may be regulated by a clamp and bubble rate determined. In all cases the glassware will minimally consist of a H_2S generating flask and a series of traps.

11.1.1 Gravimetric method - The first flask contains the sediment sample or standard. The second flask contains 175-200 mL of potassium hydrogen phthalate reagent

7.4.1 as an HCl trap. The third and fourth flasks contain 175-200 mL of silver nitrate reagent 7.4.2. If glassware specified in Section 6.1.1.1 is used, the second flask is a gas washing bottle and the third and fourth flasks are impingers.

11.1.2 Colorimetric method - The first flask contains the sediment sample or standard. The second and third flask contain an absorbant of 80 mL 0.5M NaOH reagent 7.5.2. If glassware specified in Section 6.1.1.1 is used, the second and third flasks are impingers.

11.1.3 Ion-selective electrode method - The first flask contains the sediment sample or standard. The second and third flask contain an absorbant of 50 mL SAOB reagent 7.6.2 and 30 mL DDIW. If glassware specified in Section 6.1.1.1 is used, the second and third flasks are impingers.

11.2 One hundred milliliters (100 mL) of DDIW, minus the volume of water contained in the wet sediment sample in Section 11.3, and a magnetic stirring bar are added to the flask that will contain the sediment. For the computation of the volume of water contained in the wet sediment, see Section 13.3. The traps are filled and deaerated by bubbling nitrogen for 10 minutes at a flowrate of 100 cm^3/min. Reduce flow to 40 cm^3/min.

11.3 Weigh approximately 10 g of wet sediment. If AVS concentration is high, a smaller amount of sediment may be required; use of sediment samples smaller than 1-2 grams is not recommended due to sulfide oxidation and sample heterogeneity. Use of large sediment samples is not recommended because significant amounts of acid may be neutralized. Place sediment in the standard taper round bottom flask or the Erlenmeyer flask fitted with the thistle tube or separatory funnel. Parafilm has been found to be free of sulfide (4). Samples may be weighed on 2 × 2 inch pieces of parafilm and the parafilm and sample introduced to the flask. Rinsing the sample into the flask is not recommended. Purge the sample for 10 minutes at a nitrogen flowrate of 40 cm^3/min. Stop the flow of nitrogen.

11.4 Using a 30 mL syringe, inject 20 mL of 6M HCl, which has been bubbled with N_2 gas for 30 minutes, into the reactor through the septum. If the apparatus described in Section 6.1.1.1 is used, add the HCl from the thistle tube or the separatory funnel. Bubble N_2 through the sample for 1 hour at a flowrate of 20 cm^3/min and magnetically stir the sample at the same time.

11.5 Analyze sulfide contained in sulfide trap by the appropriate analytical procedure in Section 12.

12. ANALYSIS OF SULFIDE

12.1 Gravimetric method

12.1.1 Insure that the final trap, the second silver nitrate trap, contains no precipitate.

12.1.2 Filter the silver sulfide contained in the first sulfide trap through a preweighed 1.2 micron filter. Dry at 102 °C and weigh.

12.1.3 Calculate the amount of silver sulfide as the difference between the weight of silver sulfide and the filter and the weight of the predried filter.

12.1.4 Calculate the amount of sulfide in the sample:

$$\text{Sulfide in wet sediment (}\mu\text{moles)} = \frac{\text{g } Ag_2S}{247.8} \times 10^6$$

12.2 Colorimetric method

12.2.1 If the AVS concentration is low, add 10 mL of the mixed diamine reagent (MDR) directly to the NaOH solution in each trap tube to develop the color. Transfer this solution to a 100 mL volumetric flask and dilute to the mark with DDIW. If the sulfide contained in the NaOH in the tube trap exceeds 18 µmoles, transfer the NaOH in each tube trap to a 100 mL volumetric flask. Rinse the trap with deaerated 0.5M NaOH and dilute to volume with NaOH. An appropriate volume aliquot of this solution is used for the analysis. In this case, the aliquot is transferred to a 100 mL. Sufficient 0.5M NaOH is added so that the total volume is 80 mL, 10 mL MDR is added and the solution is diluted to 100 mL with DDIW. Use of sediment samples smaller than 1-2 grams is not recommended due to sulfide oxidation and sample heterogeneity.

12.2.2 After 30 minutes, measure the absorbance of light at 670 nm using a half-inch diameter or 1 cm rectangular spectrophotometer cell.

12.2.3 If the absorbance of the sample is greater than 0.6, dilute 10-fold with 1.0M H_2SO_4 and compare to the high range calibration curve.

12.2.4 Normally, the sulfide concentration in second trap tube is close to the blank value in this procedure and is not significant in calculating the concentration of sulfide. If a significant color is developed, the flow rate and amount of sulfide in the standard or sediment should be checked

12.2.5 Preparation of calibration curve - The indicated amounts of sulfide are based on a 0.05 M concentration of the sulfide stock standard solution. The procedure indicated in Section 7.2.5 should be used to calculate the exact amount of sulfide in each of the standards.

12.2.5.1 Low range calibration curve - 0.0 - 2.5 µmoles S^{2-} (0.0 - 80 µg S^{2-})

Add 80 mL 0.5 N sodium hydroxide to each of a series of 100 mL of flasks and add 0.00, 1.00, 2.00, 3.00, 4.00, and 5.00 mL of sulfide working standard A to these flasks. These samples contain 0.00, 0.50, 1.00, 1.50, 2.00, and 2.50 µmoles S^{2-}, respectively. Add 10.0 mL of MDR to each and dilute to 100.00 mL with deionized water. After 30 minutes, measure the absorbance at 670 nm.

12.2.5.2 High range calibration curve - 0.0 - 20.0 µmoles S^{2-}

(0.0 - 640 µg S^{2-})

Add 80 mL 0.5M sodium hydroxide in 100 mL flasks and add 0.00, 1.00, 2.00, 3.00 and 4.00 mL of sulfide working standard B to these flasks. These samples contain 0.0, 5.00, 10.00, 15.00, and 20.00 µmoles S^{2-}, respectively. Add 10.0 mL of MDR and dilute to 100.00 mL with deionized water. After 30 minutes, dilute the solution 10-fold with 1.0M H_2SO_4, and measure the absorbance at 670 nm.

12.2.6 Calculate the amount of sulfide (µmoles) in the sample from the calibration curve. If the total volume of NaOH in the trap was not used in the analysis, account for the portion tested.

12.3 Ion-selective electrode method

12.3.1 Calibrate the sulfide electrode and meter according to manufacturer's recommendations, using sulfide standards prepared in SAOB reagent 7.6.2 diluted 1:1 with DDIW.

12.3.2 Transfer the contents of each sulfide trap into a 100-mL volumetric flask. Rinse the trap with DDIW, adding the rinses to the volumetric flask. Dilute to volume with DDIW.

12.3.3 Pour contents of volumetric flask into a 150-mL beaker, add a stirring bar and place on stirrer. Begin stirring with minimum agitation to avoid entrainment of air into the solution and minimize oxidation of the sample during the measurement.

12.3.4 Rinse sulfide and reference electrodes into waste container and blot dry with absorbent tissue. Immerse electrodes in sample solution.

12.3.5 Allow electrode response to stabilize (8-10 minutes), then take measurement of sulfide concentration. Depending on the meter used, the reading may be directly in concentration units if the meter is in the concentration mode and a 2-

point calibration has been performed. If the readings are in millivolts, convert millivolts to concentration using the calibration curve obtained from standard solutions.

12.3.6 Calculate the amount of sulfide (μmoles) in the sample.

13. CALCULATION OF AVS CONCENTRATION IN SEDIMENTS

13.1 The sediment dry weight/wet weight ratio (R) must be determined separately. Acid volatile sulfides can be oxidized or altered to non-volatile forms during drying.

13.2 Transfer an aliquot of the sediment to a tared 100-mL tared evaporating dish. Weigh the dish plus the wet sediment. Calculate the wet weight of the sample. Dry the sediment at 104 °C and weigh. Calculate the dry weight of sediment.

13.3 Determine the ratio of dry weight to wet weight for the sediment sample:

$$R = \frac{W_d}{W_w}$$

where R = ratio of dry weight. to wet weight,

W_d = dry weight of sediment sample (g), and

W_w = wet weight of sediment sample (g).

Also, the weight of water, W_{water}, taken in a sample for AVS analysis can be calculated. If the weight of the wet sediment sample taken for the AVS analysis is W_{s+w}, the weight of water contained in the sediment sample would be

$$W_{water} = W_{s+w} - (R \times W_{s+w})$$

The volume of water in the sample equals the weight of water, assuming the density is near unity.

13.4 Compute the sulfide concentration per gram dry weight of sediment:

$$\text{AVS } (\mu\text{moles / g}) = \frac{S}{R \times W_w}$$

where S = the amount of AVS in sediment (μmoles) from Section 12.1.4,

12.2.6. or 12.3.6. as appropriate,

R = ratio of dry weight to wet weight from Section 13.3, and

W_w = wet weight of sediment (g) taken for AVS analysis.

13.4 The QC data obtained during the analysis provides an indication of the quality of the sample data and should be provided with the sample results.

14. DETERMINATION OF SIMULTANEOUSLY EXTRACTED METALS (SEM).

14.1 After the generation of sulfide has been completed, filter the sediment suspension remaining in the H_2S generation flask (Section 11.4) through a 0.2 µ membrane filter resistant to attack by acid.

14.2 Transfer the solution to a 250-mL volumetric flask. Rinse the filtering flask with distilled water, adding the rinses to the volumetric flask. Dilute to volume with deionized water.

14.3 Determine the concentrations of cadmium, copper, mercury, nickel and zinc by atomic absorption, inductive coupled plasma spectrometic, or another approved method (7,8). Convert µg/L concentration values to µmoles/L. Multiply the µmoles/L by the solution volume to obtain the µmoles of metal.

14.4 Report the concentrations of each of the metals in the sediment on a µmole per gram dry sediment (µmol/g) basis.

14.5 Calculate the ratio of SEM to AVS:

$$\frac{\text{SEM}}{\text{AVS}} = \frac{\sum[\text{metal}]}{\text{AVS}}$$

where SEM is the sum of the concentrations of metals, Σ [metal], for the metals cadmium, copper, lead, mercury, nickel and zinc in Section 14.4, and

AVS is the acid volatile sulfide concentration determined in Section 13.4.

Both SEM and AVS are expressed on a µmole per gram dry sediment (µmol/g) basis.

14.6 A SEM:AVS ratio greater than one suggests that toxicity is possible while a ratio less than one suggests that the metals in the sediment are not toxic (2).

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STANDARD OPERATING PROCEDURE

SULFIDES, ACIDS VOLATILE

GEN-AVS
Revision 2
April 23, 1998

Approved By:

Supervisor	4/23/98 Date
QA Coordinator	4/23/98 Date
Laboratory Manager	5-6-98 Date

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: WFR Date: 8-20-99
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SULFIDES, ACIDS VOLATILE

1. SCOPE AND APPLICATION

1.1. This procedure describes the determination of acid volatile sulfide (AVS) in soil, sediment, and other solids. As a precipitant of toxic heavy metals, sulfide is important in controlling the bioavailability of metals in sediments. The procedure can be used to isolate those metals, referred to as Simultaneously Extracted Metals (SEM), solubilized during the acidification step. Analysis for these metals can then be done using the applicable metals determinative procedure.

1.2. Research has established that the relative amounts of SEM and AVS are important in the prediction of potential metal bioavailability; if the molar ratio of SEM for bivalent metals to AVS exceeds one, the toxic heavy metals in that sample are potentially bioavailable. This method uses the same conditions for release of both sulfide and metal from the sediment and thus provides a useful means of assessing the amount of metal associated with sulfide.

1.3. This method is capable of determining sulfides in the range of 0.5 mg/Kg to 30,000 mg/Kg dry weight. The Method Reporting Limit (MRL) 0.5 mg/Kg. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. Method detection limits are 0.01 mg/Kg on the spectrophotometer and 0.2 mg/Kg in the sample.

2. METHOD SUMMARY

2.1. The AVS in the sample is first converted to hydrogen sulfide (H_2S) by acidification with hydrochloric acid at room temperature. The H_2S is then purged from the sample and trapped in aqueous solution. The amount of sulfide that has been trapped is then determined. If SEM is to be determined, a portion of the aqueous solution is used to perform selected metals analyses.

2.2. The H_2S released by acidifying the sample is quantified using a colorimetric procedure. In the colorimetric method, the H_2S is trapped in sodium hydroxide. The sulfide reacts with N-N dimethyl-p-phenylenediamine to form methylene blue that is measured. This procedure is capable of determining AVS concentrations as low as 0.05 mg/Kg dry weight of sediment. By appropriate sample dilution, the maximum concentration of AVS which can be determined is at least 30,000 mg/Kg dry sediment.

3. DEFINITIONS

3.1. Acid Volatile Sulfide (AVS) - AVS is defined as sulfides that form hydrogen sulfide under the conditions of this test. This includes amorphous, moderately crystalline monosulfides, and other sulfides.

3.2. Method Detection Limit (MDL) - The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined as described in the SOP for The Determination of Method Detection Limits.

3.3. Stock Standard Solution - A concentrated solution of the analyte prepared in the laboratory using assayed reference compounds or purchased from a reputable commercial source.

3.4. Calibration Standards - Solutions prepared from the stock standard solution that is used to calibrate the method response with respect to analyte concentration.

4. INTERFERENCES

Contact with oxygen must be avoided in all stages from sampling to analysis. Consequently, the samples and standards should be protected from air from the time of sampling through the analytical procedure.

5. SAFETY

5.1. The toxicity or carcinogenicity of reagents used in this method have not been fully established. Each chemical and environmental sample should be regarded as a potential health hazard and exposure should be minimized. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets should be available to all personnel involved in the chemical analysis.

5.2. Hydrogen sulfide is a highly poisonous, gaseous compound having a characteristic odor of rotten eggs. It is detectable in air by humans at a concentration of approximately 0.002 ppm. Handling of acid samples should be performed in a hood or well ventilated area. If a high concentration of hydrogen sulfide is detected in the air by the laboratory staff, sample handling procedures must be corrected. Exposure to H_2S in air must not exceed guidelines or regulations. The OSHA Permissible Exposure Limit (PEL) is 50ppm.

5.3. If samples originate from a highly contaminated area, appropriate sample handling procedures to minimize worker exposure must be followed.

6. SAMPLE COLLECTION, PRESERVATION AND STORAGE

6.1. Sulfide ion is unstable in the presence of oxygen. Protect sediment samples from exposure to oxygen during sample collection and storage.

6.2. During storage, sulfide can be formed or lost due to biological activity, and sulfide can be lost by volatilization or oxidation. Metal speciation can change as a result of changes in sulfide concentration and as a result of other changes in the sample.

6.3. Samples should be collected in wide mouth jars with a minimum of air space above the sediment. If possible, the headspace should be filled with oxygen free nitrogen or argon. The jar lids must have Teflon or polyethylene liners.

6.4. Samples should be cooled to 4°C as soon as possible after collection. Samples maintained at 4°C have been found to have no significant loss of AVS for storage periods up to 2 weeks. Holding time for samples should not exceed 14 days.

7. APPARATUS AND EQUIPMENT

7.1. Glassware

7.1.1. AVS evolution and H_2S trapping - The Midi is typically used.

7.1.1.1. Midi setup: For each analytical train, use one 125 ml round bottom flask with a septum, 80 ml scrubber tower with impingers with non-fritted outlets. The round bottom flask contains the sediment and acid is introduced to it by a syringe inserted through the septum. The flasks are connected by tubing. Because sulfide may react with tubing and other surfaces, minimum lengths of tubing should be used as sleeves to connect the glass tubing.

7.1.1.2. Macro setup: For each analytical train, one 500 ml round bottom flask with a septum 250 ml scrubber towers with impingers with non-fritted outlets. The round bottom flask contains the sediment and acid is introduced to it by a syringe inserted through the septum. The flasks are connected by tubing. Because sulfide may react with tubing and other surfaces, minimum lengths of tubing should be used as sleeves to connect the glass tubing. The analyst should pay particular attention to the recovery of sulfide from standards in evaluating the apparatus.

7.1.1.3. The analyst should pay particular attention to the recovery of sulfide from standards in evaluating the apparatus used.

7.1.1.4. In all cases, the inlets are below the liquid level and the outlets are above the liquid levels. The apparatus is assembled as shown in Figure 1, and more than one analytical train can be connected to a single cylinder of nitrogen or argon if flow controllers are installed in the line.

7.2. Assorted calibrated pipettes and volumetric flasks.

7.3. Analytical balance - capable of weighing to 0.0001 g.

7.4. Magnetic stirrer, thermally insulated and Teflon-coated stirring bar.

7.5. Spectrophotometer - Capable of measuring absorbance at 670 nm.

7.6. Spectrophotometer cells.

8. REAGENTS AND CONSUMABLE MATERIALS

8.1. All water and reagents used in this method must be free of dissolved oxygen and sulfide. Prepare reagents fresh for each batch and use deaerated, deionized water by removing dissolved oxygen from the deionized water by vigorously bubbling with oxygen-free nitrogen or argon for approximately one hour. Deaerate reagents immediately before use by deaerating with oxygen-free nitrogen or argon.

8.2. Sulfide stock standard solution, approximately 0.05M or 50 µmoles/ml.

8.2.1. Weigh about 12 gram of $Na_2S \cdot 9H_2O$ and dissolve it in 1,000 ml of deionized water. Store in a brown bottle. To prevent air oxidation, the sulfide solution should be maintained under oxygen-free nitrogen or argon.

8.2.2. Standardize against thiosulfate solution.

8.2.2.1. Pipette 10.00 ml of 0.025N standard iodine solution into each of two 125-ml Erlenmeyer flasks.

8.2.2.2. Pipette 2.00 ml of sulfide stock standard solution into one flask. Pipette 2.00 ml of deionized water, as a laboratory reagent blank, into the other flask.

8.2.2.3. Add 5.00 ml of 6M HCl into each flask, swirl slightly, then cover and place in the dark for 5 minutes.

8.2.2.4. Titrate each with 0.025N thiosulfate until the yellow iodine color fades to a pale straw. Just before all the iodine has been titrated, add starch indicator dropwise to form a pale blue color. Continue the titration with the thiosulfate. The end point is reached when the blue color first disappears.

8.2.2.5. Calculate the sulfide concentration as follows:

$$Sulfide\ (mg/L) = \frac{(T_{blank}\ T_{sample}) \times N_{S_2O_3^{2-}}}{V_{sample}} X \frac{1\ mole\ S^{2-}}{2\ equiv\ S^{2-}} x \frac{1000\ \mu moles}{1\ mmole} x \frac{32 mmole}{mg}$$

where T = volume of titrant used for the blank and sample (ml)
N = concentration of $S_2O^{2-}_3$ titrant
V = volume of sample used (ml), 2.00 ml recommended

8.3. Iodine Solution (approximately 0.025N)

8.3.1. Dissolve 25 g potassium iodine, KI, in 700 ml of reagent water in a 1 liter volumetric flask. Add 3.2 g iodine, I_2. allow to dissolve. Add 2 ml of 6N HCl and standardize against sodium thiosulfate as follows.

8.3.2. Titrate until amber color fades to yellow. Add starch indicator solution. Continue titration drop by drop until the blue color disappears.

8.3.3. Run in duplicate

8.3.4. Calculate the normality as follows:

$$\frac{mls\ S_2O_3 \times Normality\ of\ S_2O_3\ (0.02SN)}{mls\ Iodine\ titrated\ (5\ mls)}$$

8.4. Standard sodium thiosulfate solution (0.025N): Dissolve 6.205 ± 0.005 g $Na_2S_2O_3 \cdot 5H_2O$ in 500 ml reagent water. Add 18 ml of 0.5N NaOH, and dilute to 1 liter.

8.5. Starch indicator - Dissolve 1.0 gram soluble starch in 100 ml boiling deionized water.

8.6. Sulfide working standards - Prepare sulfide working standards using the sulfide stock standard solution in Section 8.2.1. The concentrations of the following standards will depend on the exact concentration of the sulfide stock standard determined in Section 8.2.2.5.

Prepare sulfide working standard A by diluting 1.00 ml of sulfide stock standard to 1000 ml. This solution contains 1.6 mg sulfide/L, if the concentration of the sulfide stock standard is exactly 1600 mg/L.

8.7. AVS Evolution reagents

8.7.1. Hydrochloric acid 6M - Dilute 500 ml of concentrated hydrochloric acid to 1.0 L de-aerated reagent water.

8.7.2. Nitrogen gas, oxygen free, with regulator and flow controller.

8.7.3. Plastic hypodermic syringe, 30 ml, and needle.

8.8. Colorimetric reagents

8.8.1. Sodium Hydroxide solution, 0.5N - Dissolve 20 g sodium hydroxide per 1000 ml reagent water. Generally make 4.0 L at one time. (80 g NaOH to 4.0 L)

8.8.2. Mixed diamine reagent, MDR

8.8.2.1. Component A - Add 1320 ml concentrated sulfuric acid to 680 ml of reagent water. N-N-dimethyl-p-phenylenediamine oxalate in it.

8.8.2.2. Component B - Dissolve 10.8 g ferric chloride hexahydrate ($FeCl_3 \cdot 6\ H_2O$) in 200 ml concentrated hydrochloric acid and dilute to 400 ml with reagent water.

8.8.2.3. Mix components A and B.

8.9. Sulfuric acid solution, 1.0M - Dilute

9. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10. PREVENTATIVE MAINTENANCE

10.1. No specific maintenance steps are needed other than normal cleaning and inspection of apparatus.

10.2. Wipe down the Midi unit after use to remove any residual acid.

11. PROCEDURE

11.1. Macro distillation procedure

11.1.1. Place two neck flasks on stir plates and clamp into place.

11.1.2. Add one stir bar and 100 ml of reagent water to each flask. The amount of water added and the water present in the wet sediment should not exceed 120 mls.

11.1.3. Add 80 mls of 0.5N NaOH to each of the two towers for each flask. Label the first tower A, the second tower B.

11.1.4. Place septum stopper and impinger in two-neck flask. Place tower scrubbers in towers and connect the glassware with the tubing.

11.1.5. Purge the system with N_2 gas for approximately 10 minutes.

11.1.6. Weigh 10 g of wet sediment onto a 2x2 inch piece of parafilm. Remove the septum stopper and place the parafilm with sample into the flask. Do not rinse sample into flask. Purge the system for another 10 minutes.

Stop the flow of gas. Using the 30 ml hypodermic syringe, add 20 ml of 6M HCl to the flask via septum. Bubble nitrogen through the system for one hour while constantly stirring. For the LCS and spike, add the appropriate amount of sulfide standard before adding the acid.

11.1.7. Calibration and Standardization

11.1.7.1. Prepare a sulfide curve as follows:

11.1.7.1.1. Add 80 mls of 0.5N NaOH to each of 6 100 ml class volumetric.

11.1.7.1.2. Using working standard A, add 1 ml, 2 ml, 3 ml, 4 ml and 5 ml to each flask. Do not add sulfide standard to the first flask.

11.1.7.1.3. Calculate concentration of standards as follows:

$$\frac{(\text{mls STD added}) \times (\text{Conc. of STD})}{\text{Final volume (100ml)}} = __ \ mg/L$$

Example: $\frac{4\ ml \times 16\ mg/L}{100\ ml} = 0.64\ mg/L$

11.1.7.1.4. Use the 0 ml STD for the CCB and the 4 ml STD for the CCV.

11.1.8. After one hour has elapsed, remove the scrubber from tower B and transfer to a 250 ml plastic container. Repeat for tower A. Leave N_2 gas flowing through scrubbers so that the NaOH does not go back up into the scrubber.

11.1.9. Disconnect tubing from flow meter at the flask impinger and then turn off the N_2 at the flow meter. This prevents liquid from backing up into the flow meter and destroying it.

11.2. Midi distillation procedure

11.2.1. Rinse scrubbers with 6N HCl follwoed by a rinse with deionized water.

11.2.2. Place boiling tubes, with impinger and septa inserted, into the midi system. Connect gas lines to impingers. Clamp septum to impinger.

11.2.3. Add 40mL of 0.5N NaOH to each bubbler vessel and clamp scrubber to bubbler vessel. Connect scrubber, with bubbler vessel attached, to the impinger arm.

11.2.4. Purge the system for approximately 10 minutes.

11.2.5. Add 10.0g of sample (or less depending on expected sulfide concentration) to the boiling tube. Rinse sample to the bottom of the boiling tube with 40mL of reagent water. Make sure no sample remains above the reagent water level. Clamp the impinger to the boiling tube.

11.2.6. Perform calibration steps as described in section 11.1.7.

11.2.7. Purge system for 10 minutes.

11.2.8. Connect scrubber tube with a disposable pipet to scrubber extension. Place the pipet into 50mL centrifuge tube filled with 40mL of 0.5N NaOH. (These are the "B" towers.)

11.2.9. Purge for 10 minutes.

11.2.10. Add 10.0 mL of 6N HCl to each boiling tube through the septum using a 10 mL syringe.

11.2.11. Let system react for 1 hour.

11.2.12. Place scrubber solution from bubbler vessels into separate containers. Before screwing on the cap, cover each solution with nitrogen.

Note: The same procedure is used for B towers except that the scrubber solution is left in the original centrifuge tube.

11.2.13. After complete, take down the apparatus and rinse all parts with water. Use a damp paper towel to wip down midi still and surrounding area.

Note: If Simultaneously Extracted Metals are to be determined, swirl bubbler tube to homogenize sample and pour 50 mL into a centrifuge tube for metals analysis.

11.3. Color development and analysis

11.3.1. If the sample is known or suspected to fall within the range of the curve, transfer the entire volume of tower A to a 100 ml volumetric flask. Add 10 ml of MDR and dilute to 100 ml with reagent water. For the six standards for the curve, add 10 ml MDR and dilute to 100 ml with reagent water. These volumes listed are for the macro setup. For midi procedure, volumes are halved.

11.3.2. If the samples are known or are suspected to be high in concentration, dilutions may be performed as follows.

Calculate the dilution based on a 50 ml volume. Subtract the amount of scrubber solution to be analyzed from 40 mls. Add that amount of 0.5N NaOH to a 50 ml volumetric. Add the volume of scrubber solution to be analyzed. Add 5 mls of MDR and dilute to 50 mls with reagent water.

Example dilution:

2 ml $\rightarrow$ 50 ml dilution

Place 38 mls of 0.5N NaOH in a 50 ml volumetric. Next place 2 ml of solution form tower A for that sample into the same volumetric. The volume in the flask is now 40 mls. Add 5 mls MDR and dilute to 50 mls with reagent water.

11.3.3. If blue color change develops in the sample, the scrubber solution of tower B for that sample must also be analyzed. If no color change occurs for tower A, then tower B does not have to be analyzed.

11.3.4. For tower B, place the entire 80 mls of scrubber solution in a 100 ml volumetric flask, add 10 mls MDR and dilute to 100 mls with reagent water.

11.3.5. After adding the MDR, allow a full thirty minutes for color development, but read the samples before 2 hours have elapsed.

11.3.6. Allow a thirty minute warm-up of the spectrophotometer. Zero the instrument with D.I. water at 670.0 nm.

11.3.7. Calibrate the instrument with the prepared curve starting with the low standard. A calibration with a correlation coefficient (r) of ≥ 0.995 must be obtained. Corrective action for a correlation coefficient < 0.095 is to discard existing curve, obtain new volumetrics and make a new curve. Verify concentration of stock sulfide standard.

11.3.8. Analyze samples in the following order:

Analysis	Sample	Analysis	Sample
1	LCS-1	15	CCV-2
2	LCS-2	16	CCB-2
3	CCV-1	17	Sample-7
4	CCB-1	18	Sample-8
5	MB-1	19	Sample-9
6	Sample-1	20	Sample-10
7	Sample-1D	21	Sample-11
8	Sample-1S	22	Sample-12
9	Sample-1SD	23	Sample-13
10	Sample-2	24	Sample-14
11	Sample-3	25	Sample-15
12	Sample-4	26	Sample-16
13	Sample-5	27	CCV-3
14	Sample-6	28	CCB-3

11.3.9. Analyze a continuing calibration verification standard (CCV) and continuing calibration blank (CCB) every 10 sample or QC analyses. The CCV is the fifth point on the curve. The CCV must be $\pm$ 10% of the expected value. If the CCV fails, then the curve is not

correct. The correlation coefficient may be ≥ 0.995, but if the slope is to high or too low, then the CCV will not pass. Notify supervisor, re-make the curve and re-calibrate. The CCB must be < 0.01 ppm - The CCB is the first point of the curve.

11.3.10. Calculate AVS concentration as follows:

$$\frac{(Conc.\ from\ curve)\ (DF)\ x\ Final\ volume}{Initial\ wt.}\ x\ \%solids\ =\ ppm\ AVS$$

11.3.11. Final volume will always be 100 mls, unless a volume less than 80 mls is used in the tower scrubbers.

12. QA/QC REQUIREMENTS

12.1. Refer to the SOP for Analytical Batches and Analytical Sequences for guidelines for frequency of QC sample analyses. The QC samples required are as follows:

12.1.1. A method blank (MB) for every 20 samples analyzed.

12.1.2. One LCS for every 20 samples analyzed. Due to the time requirements for set-up and distillation, it is best to distill 2 LCS's for every 20 samples. Sulfides are very unstable and easily degraded and therefore it is not uncommon to have low sample recoveries.

12.1.3. Analyze one matrix spike and one matrix spike duplicate per 20 samples minimum. See 10.1.2 for explanation.

12.1.4. Analyze one sample duplicate for every 20 samples minimum.

12.2. Acceptance Criteria

12.2.1. The method blank (MB) must be < MRL. If MB > MRL, reanalyze MB. Try to determine source of contamination.

12.2.2. MRL calculated as follows:

$$\frac{0.05\ mg/L\ x\ Final\ volume(L)}{Initial\ wt.(kg)}\ x\ \%solids$$

12.2.3. The LCS must be ± 15% of the expected value. If the LCS > ± 15%, sulfides were lost at some point during distillation, or sample was spiked at the improper level. Re-analyze

LCS and all samples associated with that LCS. Notify supervisor. (Note - LCS's generally only fail high if the sample was spiked too high).

12.2.4. Matrix Spike recovery criteria is 75-125% recovery. If recovery is outside the criteria, re-analyze spike. If two spikes were analyzed initially, then matrix interferences caused low spike recoveries. No need to reanalyze at that point. Notify supervisor.

12.2.5. The relative percent difference (RPD) for duplicates should be $\leq$ 20% RPD. If the RPD is > 20%, verify homogeneity of sample. Reanalyze the duplicate. Notify supervisor. Calculate RPD as follows:

$$RPD = \frac{High\ value\ -\ Low\ value}{Average} \times 100$$

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Calculate samples results as described in section 11.6.12. Results for QC analyses are calculated and recorded as specified in section 12. Average, RPD, spike level and spike recovery are entered on benchsheet for corresponding samples. All data will be initialed, dated and attached to required data quality worksheet.

13.2. The appropriate benchsheets, located in Appendix A, should be in use at all times during AVS analysis.

13.3. The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in the CAS network directory R:\WET\WIP. These forms are made from templates located in R:\WET\FORMS.

13.4. Refer to the SOP for Laboratory Data Review Process for general guidelines for data review.

13.5. Reporting

13.5.1. Soils, sediments, and other solid matrices are reported as mg/Kg, either dry weight or as received, according to the project requirements.

13.5.2. The MRL is calculated as follows:

$$\frac{0.05\ mg/L \times Final\ volume(L)}{Initial\ wt.(kg).} \times \%solids$$

13.5.3. The MRL will vary depending on % solids content. For samples reported as received, the MRL = 0.05 mg/Kg.

13.5.4. For samples that did not require a dilution, 3 significant figures will be reported. For samples that required a dilution, report only 2 significant figures.

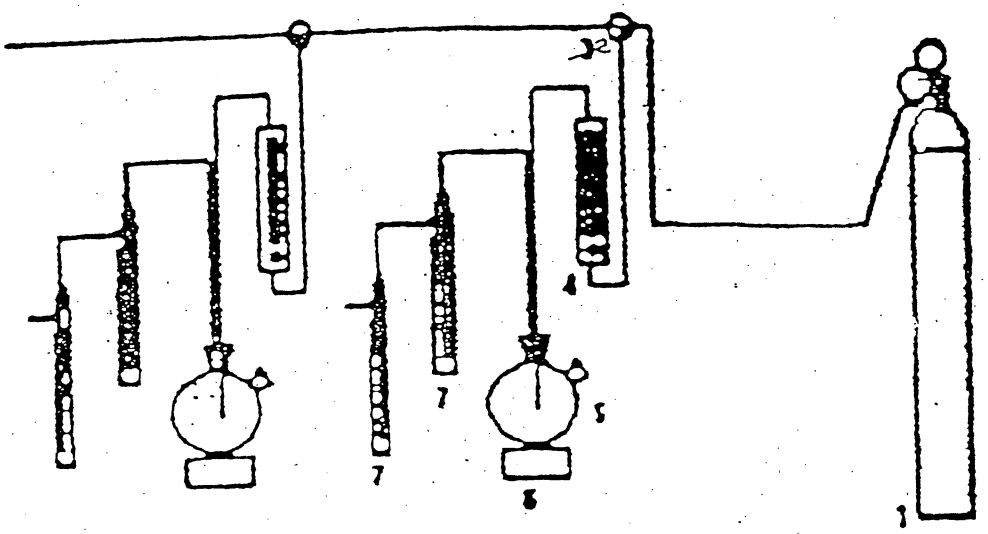
13.5.5. For samples below the MRL, report the number as < MRL.

14. REFERENCES

Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment, August, 1991

FIGURE 1

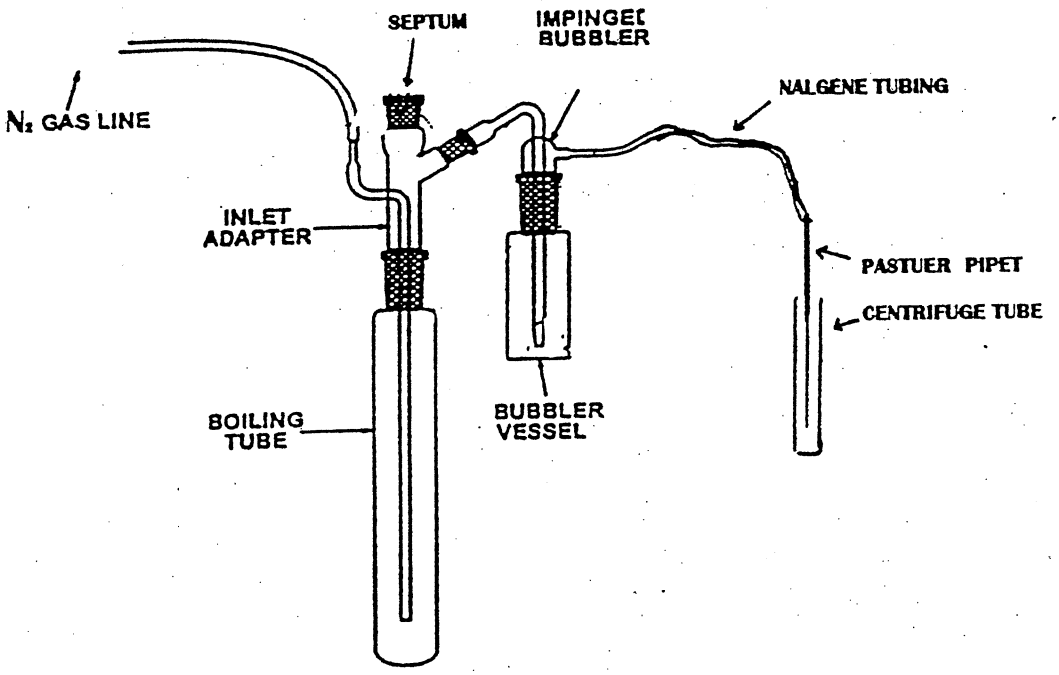
Macro Distillation Apparatus



Apparatus for AVS determination: 1 N_2 cylinder, 4. Flow controller, 5. Reaction flask, 6. Naguefic stirrer, 7. Impingers with non-fritted outlets.

FIGURE 2

Midi Distillation Apparatus



APPENDIX A

Benchsheet

COLUMBIA ANALYTICAL SERVICES, INC.

9030M/EPA Draft Aug, 1

Work Order #.: _____ Method: Methylene Blue Colorimet

Analysis: Total Sulfides/ AVS/ Soluble Sulfides

Date Prepared	Sample Name / Lab Code	Initial Wt./Vol (g) or (ml)	inal Volum (ml)	mg/L (in solution)	mg/L - mg/kg As Rec'd	% Solids	mg/kg Dry Wt.

LCS 1 = % REC =

LCS 2 = % REC =

Spike = % REC =

Spike Dup.= % REC =

x =

STD ID# = Conc. = RPD =

Prepared By:	Date Prepared:
Analyzed By:	Date Analyzed:
Reviewed By:	Date Reviewed:

COLUMBIA ANALYTICAL SERVICES, INC.

EPA Draft, Aug. 1991

Work Order #.: _____ Method: Methylene Blue Colorimetric

Analysis: AVS/SEM

Date Prepared	Sample Name / Lab Code	Initial Wt./Vol (g) or (ml)	Final Volume (ml)	mLs 50% HCl added	%HCl in Solution	% Solids	mg/kg Dry Wt.

LCS 1 = % REC =

LCS 2 = % REC =

Spike = % REC =

Spike Dup. = % REC =

x =

STD ID# = RPD =

Prepared By:	Date Prepared:
Analyzes By:	Date Analyzed:
Reviewed By:	Date Reviewed:

Final: 12/22/98

DMMP CLARIFICATION PAPER
SMS TECHNICAL INFORMATION MEMORANDUM

TRIBUTYLTIN ANALYSIS: CLARIFICATION OF INTERSTITIAL WATER EXTRACTION AND ANALYSIS METHODS - INTERIM

Prepared by Erika Hoffman (U.S. EPA, 206-553-0038) for the DMMP agencies.

INTRODUCTION

Tributyltin (TBT)[1] is a special chemical of concern under the Dredged Material Management Program (DMMP). Testing for this chemical in areas where it is likely to be found (e.g. marinas, ship repair facilities, navigation channels) may be required under this program. The available literature indicates that the toxicity and bioaccumulation of TBT are affected by a variety of factors, including organic carbon in sediment and water, pH, salinity, clay fraction, and the presence of constituents such as iron oxides. In addition, TBT exists in several forms, is released into the aquatic environment in different ways, and the mode of exposure to benthic organisms varies.

In 1996 the DMMP/SMS agencies reviewed the available literature and concluded that analysis of bulk sediment TBT was not the most environmentally relevant way to assess the bioavailability of TBT (EPA, 1996, Michelsen *et al.*, 1996). Due to the way in which TBT acts and reacts in the environment, the agencies determined that analysis of interstitial water for the presence of TBT was a more effective way to measure its potential for impact in the aquatic environment. As a part of the Sediment Management Annual Review process, the agencies adopted the requirement for interstitial water analysis in 1996.

PROBLEM IDENTIFICATION

A number of questions have been raised regarding sampling and analysis for TBT since the agencies initiated the requirement for interstitial water testing. Many different approaches are used for the extraction and preparation of interstitial water from sediment samples and it is generally believed that both technique and materials can cause large variations in the measured interstitial water TBT concentration. In particular, there are concerns that certain steps in the extraction and preparation process may contribute to the loss of TBT in the pore-water. Some of the most important unresolved issues involving the measurement of TBT in interstitial water include:

- The degree of TBT adsorption to various laboratory materials (such as stainless steel, and borosilicate glass) and equipment (such as centrifuge tubes, filters and filtration apparatus).

[1] Although this clarification paper focuses on tributyltin, the procedures and guidance presented herein apply to the measurement of all organotin species (e.g., mono-, di-, tri-, and tetrabutyltin).

- The effect of aerobic conditions on TBT solubility.
- The effect of sediment and interstitial water extract holding time on TBT solubility.
- Differing partitioning characteristics of TBT in fresh water versus marine environments.
- The relative toxicological importance if colloid-bound TBT versus soluble TBT in porewater.

Avocet Consulting and Striplin Environmental Associates (SEA) investigating these and other unresolved issues surrounding butyl tin extraction, analysis, and data use. The study is divided into two phases. The first phase includes distribution of a policy and comprehensive analytical questionnaire (November, 1998), a follow-up regulatory workshop (January, 1999), and finalization of the analytical work plan based on questionnaire and workshop responses. The second phase consists of analysis of actual environmental samples, data validation, data interpretation and write-up, and preparation of a SMARM clarification paper. The ultimate goal of the Avocet/SEA study and of this clarification paper is to further the development of a DMMP standard protocol for extracting and measuring TBT in sediment interstitial water.

The following clarifications are provided as ***interim*** **guidance to ensure that all projects reporting TBT data** utilize similar **methods for interstitial water extraction and TBT analysis. The agencies** recognize **that project-specific constraints may require deviations from the** methods and performance **criteria described below. Any such deviation must be** justified and **approved by the DMMP agencies prior to test initiation.**

Final: 12/22/98

PROPOSED CLARIFICATIONS FOR THE TBT-INTERSTITIAL WATER PROTOCOL

The following proposed ***interim*** guidance should be applied to all DMMP projects for which interstitial water TBT analysis is required. This guidance was developed based on input from researchers and technical staff who regularly extract and measure interstitial water TBT. It reflects current thought on the best methodology for limiting the loss of TBT due to sample handling. The DMMP agencies are aware that new methodologies to refine TBT analysis may be in development and will consider updating these interim guidelines with any alternative methods that may yield equal or better results. This interim guidance will be revisited at a later date by the DMMP agencies once the results of the Avocet/SEA study (described above) as well as results from other project-specific testing are available.

Copies of this Clarification paper can be obtained from the Seattle Corps Dredged Material Management Office (David Kendall (206) 764-3768) and are also available on the Seattle District Corps web site at:

http://www.nps.usace.army.mil/dmmo/smarm98/papers.htm.

1. Materials

Field collection and storage - Polycarbonate, borosilicate glass or stainless steel containers should be used for field collection, homogenization, and pre-centrifugation storage of sediments to be tested for interstitial water TBT.

Centrifugation and interstitial water collection– Polycarbonate containers should be used for centrifugation and for supernatant collection after the first round of spinning since use of polycarbonate minimizes loss of TBT due to sorption. Borosilicate glass storage containers may be used to store the acidified supernatant collected after the second round of centrifugation.

Interstitial water analysis – Borosilicate glass or polycarbonate equipment and containers may be used when handling and extracting acidified interstitial water samples.

2. Equipment Decontamination and Cleaning

All laboratory equipment and glassware used for sediment collection and storage, interstitial water collection and storage, and TBT analysis should be appropriately cleaned and rinsed with residue analysis grade solvents. The effectiveness of any given decontamination procedure will ultimately be demonstrated by meeting the appropriate QC performance criteria (see Table 1). The following are examples of acceptable procedures for the cleaning and decontamination of polycarbonate and other material:

Polycarbonate	All other materials
Detergent wash	Detergent wash
Rinse with dilute hydrochloric acid	Rinse with deionized water
Rinse with deionized water	Rinse with acetone
Rinse with 0.1% tropolone/hexane	Rinse with 0.1% tropolene/$MeCl_2$
Rinse with hexane	Rinse with methylene chloride

3. Sediment Sampling and Holding

When loading sediment samples into storage containers, headspace should be minimized and containers purged with nitrogen either in the field or upon arrival at the laboratory (before storage). Because the freezing of bulk sediment samples may result in structural changes in the sediment which will alter the availability of TBT, samples to be held for future TBT analysis should have interstitial water extracted prior to freezing. Sediment samples should be maintained in the dark at 4°C while in transport and once in the laboratory. It is important to minimize sample holding time prior to centrifugation in order to reduce the chance for adsorptive loss of TBT. No more than 7 days should elapse between sediment collection and centrifugation.

4. Interstitial Water Collection Method

Centrifugation is preferred for collecting sediment interstitial water following the procedure in paragraph 5 (below). Alternative interstitial water extraction methods may be used in cases where centrifugation is not an effective technique, (e.g., for very sandy sediments) and will be decided on a case-by-case basis by the DMMP agencies.

5. Centrifugation Procedure

Sample preparation - Pooled interstitial water that may collect on the surface of the sediment sample while in storage should be reintroduced into the sample prior to centrifugation. Samples should be maintained at or below 11 °C during processing.

Anaerobic extraction - Sample loading, supernatant/interstitial water decanting, transfer, and sample acidification should occur under anaerobic conditions (i.e., under a nitrogen atmosphere).

Centrifuge settings - Samples should be double centrifuged with the first run at 3000 G for 30 minutes and the second run within the range of 3000 – 9000 G for 30 minutes. Samples should be maintained at a temperature at or below 11°C during centrifugation. Extra care should be taken to minimize carryover when transferring supernatant after each centrifugation step. A new and potentially useful method for obtaining particle-free interstitial water using an aspiration system is described in Ozretich and Schults (19[illegible]).

6. Filtration

Filtration should not be performed on interstitial water samples. The DMMP agencies will reconsider the question of whether or not to filter interstitial water pending results of the Avocet/SEA study (described above) and any other studies addressing the influence of filters and the filtration process on TBT sorption.

7. Interstitial Water Handling

Interstitial water should be acidified with hydrochloric acid immediately after centrifugation and stored in pre-cleaned containers (borosilicate glass or polycarbonate) at 4°C. No more than 7 days should elapse between interstitial water collection and the initiation of TBT analysis.

8. Analytical Methods

Acceptable methods for measuring TBT involve tropolone/methylene chloride extraction, followed by Grignard derivitization and analysis by GC/MS (e.g., Krone *et al.*, 1989), GC/MS SIM (e.g., PSEP, 1997), or GC/FPD (e.g., Unger *et al.*, 1986).

9. Performance Criteria for Sample Collection and Interstitial Water TBT Analysis

The DMMP agencies have decided to recommend QC performance criteria rather than providing a step-by-step protocol for the extraction, derivitization, and analysis of TBT. The criteria presented in Table 1 must be met in order to verify that cleaning, extraction and derivitization methods are being performed correctly. Laboratories will be required to meet these performance criteria as well as take the specified corrective action if performance criteria are not met. Deviations from the specified performance criteria will be considered by the DMMP agencies on a project-specific basis. Justification for alternative performance criteria must be submitted in writing and receive agency approval prior to the initiation of testing. As discussed in earlier guidance (Michelsen, *et al.*, 1996), TBT analytical results and QC information should be reported as the TBT ion.

Table 1. Summary of Quality Control Procedures for TBT in Interstitial water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCS) *	1 per analytical batch (≤ 20 samples)	Recovery 50 – 150%	1. Check calculations. 2. Reanalyze (matrix or injection problems?) 3. If still out, re-extract and reanalyze LCS and assoc. samples (if available); If not available flag data.
Matrix spike (MS) and matrix spike duplicate (MSD) *	1 MS/MSD pair per analytical batch (≤ 20 samples)	Recovery 50 – 150% and relative percent difference (RPD) ≤ 30%	4. Evaluate for supportable matrix effect. 5. If no interference, re-extract and reanalyze MS/MSD once (if sample available). 6. If still out, report data, caveats of data.
Surrogate spike * (Tripentyltin recommended)	1 per sample	Recovery 50 – 150%	7. Check calculations. 8. Evaluate for supportable matrix effect. 9. If no interference is evident, re-extract and reanalyze affected sample(s) (if available) and flag any outliers.
Method blank **	1 per analytical batch (≤ 20 samples)	Target analyte ≤ 3x the reporting limit (RL)	10. Flag if target ≤ 3x RL but less than 0.075 ppb ***. 11. Rerun batch and ID contamination source if target ≥ 0.075 ppb.

*** All QC samples should be run using the same sample handling as is used on the environmental samples.**
**** Method blank can include centrifugation step or, alternatively a centrifugation blank can be run separately from the analytical method blank.**
***** 0.075 ppb TBT is used here as a benchmark for evaluating blank performance because it represents a concentration that is one-half the interstitial water screening level (0.15 ppb) that is being used by the DMMP agencies to determine the need for bioaccumulation testing. Note that a minimum interstitial water volume of 200-500 ml will be needed to attain reporting limits less than 0.075 ppb TBT.**

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Unger, M.A., W.G. McIntyre, K. Greaves, and R.J. Huggett, 1986. GC Determination of butyltins in natural waters by flame photometric detection of hexyl derivatives with mass spectrometric confirmation. Chemosphere 15:461.

10-Day Toxicity Test with Freshwater Amphipod (*Hyalella azteca*) to Meet U.S. EPA Guidelines.

1.0 INTRODUCTION

The objective of this study is to determine the toxicity of a contaminated sediment sample(s) to amphipod (*Hyalella azteca*) during a 10-day exposure. Amphipods are exposed to the sediment sample to assess survival and growth on test day 10 (test termination). The methods (Springborn Smithers Laboratories test method No.: SED-Ha-121) described in this study plan meet the standard procedures described in the "Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates, 2nd Edition", test method 100.1 (U.S. EPA 2000.

2.0 MATERIALS AND METHODS

2.1 Test System

2.1.1 Species

The freshwater invertebrate, *Hyalella azteca,* is the species used in this test. Test organisms will be 7 to 14 days old at initiation of the test. Amphipods used in the exposure will be the young amphipods produced by adult amphipods removed from culture tanks 7 to 14 days prior to test initiation. The adult amphipods are placed in 9.5 liter aquaria with approximately 8L of water. Young produced by these isolated adults will then be removed and pipetted into holding containers until test initiation. Amphipods will not be used if >10% mortality is observed during the 48 hours prior to test initiation.

2.1.2 Source

Hyalella azteca cultures will be maintained at Springborn Smithers Laboratories Amphipods will be cultured in 20 liter glass aquaria (containing approximately 10-L of culture water) under flow-through conditions. Water used to culture the amphipods is similar to the overlying water used during the 10-day test. Culture water will be maintained at 23 ± 1°C.

2.1.3 Feeding

While being maintained in the culture prior to the test, adult and juvenile amphipods will be fed every other day. They will be fed a combination of Yeast, Cereal leaves and flaked fish food suspension (YCT) and a unicellular green algae *Psueokirchneriella subcapitata.* During testing, 1.5 mL of YCT Suspension will be added daily to each test vessel. If food collects on the sediment surface during testing, feeding will be suspended for one or more days.

2.1.4 Handling

Wide-bore pipets will be used to transfer the amphipods, taking care to minimize possible stress due to handling. Amphipods that are damaged or dropped during transfer will not be used.

2.2 Physical System

2.2.1 Sediment

Sediment samples should be shipped overnight to Springborn Smithers Laboratories after collection. Upon receipt at Springborn Smithers, sample containers will be inspected for leakage or damage and the sample identity recorded. If storage is required, the samples will be refrigerated at approximately 4°C. In addition, a sediment sample will be collected from an uncontaminated location near the site of interest to be used as a reference sediment. A laboratory control sediment, prepared or collected by Springborn Smithers Laboratories, will be included in the test to evaluate performance of the test organisms and exposure system. The test will be initiated within 14 days of sediment collection.

2.2.2 Test Vessels

The test vessels used in the static-renewal toxicity test will be 300 mL glass beakers which are chemically clean. Each test vessels has a 2-cm hole cut on the top portion of the vessel and is covered with 40-mesh Nitex® screen for drainage. Each vessel will contain 100 mL (approximately 2 cm layer) of sediment and 175 mL of overlying water. Test vessels will be cleaned by an appropriate method to remove residue of test substance previously used (i.e., acid to remove metals and bases; detergents and organic solvents to remove organic compounds) and rinsed several times using diluent water.

2.2.3 Overlying Water

Water from a 100 meter bedrock well is pumped to a concrete reservoir where it is supplemented on demand with untreated, unchlorinated, Town of Wareham well water. The water is characterized as being "soft" with a normal pH range of 6.9 - 7.7, a total hardness of 30 - 60 mg/L and a specific conductance of 110 - 160 µmhos/cm. The pH, total hardness, alkalinity, and specific conductance of this water will be monitored weekly at a central location in the laboratory to assure that these parameters are within the normal, acceptable ranges. Total hardness and alkalinity will be determined according to *Standard Methods for the Water and Wastewater*, (APHA, 1992).

The quality of the water is judged by periodic analyses of representative samples conducted to ensure the absence of potential toxicants, including pesticides, PCBs and selected toxic metals, at concentrations which may be harmful to the amphipods, as well as the ability of amphipod cultures to survive and reproduce in the water free of stress.

2.3 Test Procedures

2.3.1 Test Concentration

Eight replicates will be maintained for each sediment sample consisting of 100% whole sediment sample (no dilutions). A reference control (if collected), conducted with eight replicates, will be used to evaluate the survival and growth potential of the test organism in a non-contaminated sediment. In addition, a laboratory control sediment, prepared or collected by Springborn Smithers Laboratories, will also be used to evaluate the survival and growth potential of the test organisms. The laboratory control sediment will also be conducted with eight replicates. Ten amphipods (7 to 14 days old) per replicate (80 organisms per sediment sample or control) will be used to initiate the test.

2.3.2 Test Initiation

The day before test initiation (day -1) test sediment, reference control and laboratory control sediments will be added to the replicate test vessels and the overlying water added. Prior to addition to the test vessels, each sediment sample will be wet pressed through a 2.0 mm stainless steel sieve to remove any potential predators. The water will be added gently to prevent resuspension of the sediment layer in the water column. This allows the sediment and water to equilibrate prior to addition of the test organisms

The juvenile amphipods (7 to 14 days old), produced by isolated adults, will be removed from the holding vessels (see section 2.1.1). Ten juvenile amphipods will be randomly selected and pipeted into a replicate test or control vessel. This procedure will be repeated until all vessels contain ten amphipods (eighty per test sample and control). Test vessels will be inspected within 1 hour after the juvenile amphipods are introduced to ensure organisms are not trapped in the surface tension or not burrowed into the sediment. During this one hour period, organisms observed to be trapped in the surface tension or not burrowed will be replaced with new juvenile amphipods.

2.3.3 Renewal of Overlying Water

During the 10-day study, the overlying water will be renewed by adding two volume additions (i.e., 350 mL) per day using an intermittent delivery system in combination with a calibrated water-distribution system (Zumwalt et al., 1994). The intermittent delivery system will be calibrated to provide 1 liter of water per cycle to the water-distribution system, which subsequently provides 50 mL of water per cycle to each replicate test chamber. The water delivery system cycles 7 times per day, providing 2 volume additions every 24 hours. Delivery of two volume replacements per day is sufficient to provide consistent and acceptable water quality characteristics throughout the duration of the 10 day exposure.

2.3.4 Photoperiod

The test vessels will be located in an area illuminated to a light intensity of 500 to 1000 lux using a combination of fluorescent bulbs. A 16-hour light, 8-hour dark photoperiod will be maintained with an automatic timer. Sudden transitions from light to dark and vice versa will be avoided.

2.3.5 Measurement of Water Quality Variables

Total hardness, alkalinity, specific conductance, pH and ammonia will be determined at test initiation and test termination in the overlying water from a composite sample from all eight replicate vessels. The composite sample will be taken from 1 to 2 cm from the sediment surface using a pipet. Dissolved oxygen and temperature will be measured in all replicate vessels at test initiation and test termination. Dissolved oxygen and temperature will be monitored daily in one alternating replicate during the course of the study (test days 1-9). Temperature will be monitored continuously in the waterbath using a minimum-maximum thermometer. Readings of temperature extremes will be recorded daily.

2.3.6 Dissolved Oxygen

Total dissolved oxygen will not be allowed to drop below 2.5 mg/L at 23°C. Aeration (with oil-free air) will be initiated to raise and maintain the dissolved oxygen concentration at or above 2.5 mg/L.

2.3.7 Temperature

Temperature of the overlying water will be maintained at 23 ± 1°C by conducting the study in a temperature controlled water bath maintained at the appropriate test temperature.

2.3.8 Biological Data

Survival of the amphipods will be determined in each test vessel at test termination (test day 10) by sieving the sediment to remove all surviving amphipods. The growth of surviving amphipods in each replicate will be recorded at test termination by pooling all surviving amphipods from each replicate vessel and drying at 60EC for 24 hours. Pooled amphipods will be weighed on a calibrated analytical balance to the nearest 0.01 mg. In addition, daily observations of organism behavior (e.g., sublethal effects) and characteristics of sediment and overlying water will also be observed and recorded daily. Dead organisms are removed from the exposure vessels daily.

2.3.9 Test Acceptability

At termination of the study, mean survival of the amphipods in the laboratory control must be ≥ 80%.

3.0 STATISTICAL ANALYSES

The mean survival and growth of organisms exposed in each test sediment and reference control sample will be tested for normality and homogeneity of variance using Shapiro-Wilks Test and Ba F-Test. If the data set passes these two tests, then a parametric method (e.g., ANOVA 2-Sample T-Test or Dunnett's Test) will be used to evaluate the results of the mean survival of each test sample for significant adverse effects. If the data set fails the test for normality and homogeneity of variance, then a non-parametric method (e.g., Steel's Many-One

Rank Test) will be used to determine significant adverse effects. If necessary, mean survival values will be transformed (e.g., arcsine square).

4.0 REPORTING

The raw data and the final summary report will be reviewed by the Study Director. The test results will be presented in an outline format on a per sample basis.

5.0 REFERENCES

APHA, AWWA, WPCF. 1992. Standard Methods for the Examination of Water and Wastewater. 18th Edition, Washington, D.C.

U.S. EPA. 2000. Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates. 2nd Edition. U.S. Environmental Protection Agency. Office of Research and Development. EPA/600/R-99/064.

Zumwalt, D.C., F.J. Dwyer, I.E. Greer and C.G. Ingersoll. 1994. A water-renewal system that accurately delivers small volumes of water to exposure chambers. Environmental Toxicology and Chemistry. pgs. 1311-1314.

10-Day Toxicity Test with Midge (*Chironomus tentans*) to Meet U.S. EPA Guidelines.

1.0 INTRODUCTION

The objective of this study is to determine the toxicity of a contaminated sediment sample(s) to midge (*Chironomus tentans*) during a 10-day exposure. Midges are exposed to the sediment sample to assess survival and growth on test day 10 (test termination). The methods (Springborn Smithers Laboratories test method #: SED-Ct-101) described in this study plan meet the standard procedures described in the "Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates, 2nd Edition", test method 100.2 (U.S. EPA 2000).

2.0 MATERIALS AND METHODS

2.1 Test System

2.1.1 Species

The dipteran midge larvae, (*Chironomus tentans*), will be used to conduct the toxicity tests. The midge larvae used in the test will be second (5 to 8 days post hatch) to third (9 to 11 days post hatch) larval instars at test initiation with at least 50% of the organisms at third instar.

2.1.2 Justification of Test System

Midges (*Chironomus tentans*) will be used for several reasons. The larvae are bottom dwellers, widely distributed throughout North America, and are considered a reasonable representative of aquatic benthic invertebrates (Adams et al., 1985). The organism is easily cultured and has a relatively short life cycle (approximately 30 days at 25°C), making it suitable for toxicity tests.

2.1.3 Source

The midge larvae will be obtained from in-house cultures and will cultured at the requested test temperature (23 ± 1°C). Second and third instar larvae will be collected from egg masses 5-11 days after hatching. During the final 48 hours of larval holding, the reserved population will be examined for mortality and general behavior. If significant mortality (e.g., 10%) is observed during the 48 hour period, then the reserved population will be discarded.

2.1.4 Feeding

During culturing and the static-renewal toxicity test, the midge larvae, generally, will be fed daily. The feeding regime may be adjusted to meet the nutritional requirements of the midges that may vary upon the stage of their development during both testing and culturing. The test midge will be fed 1.5 mL of a finely ground flaked fish food suspension (4.0 mg/mL).

2.1.5 Handling

Wide-bore pipets will be used to transfer the midge larvae, taking care to minimize possible stress due to handling. Midges that are damaged or dropped during transfer will not be used.

2.2 Physical System

2.2.1 Sediment

Sediment samples should be shipped overnight to Springborn Smithers Laboratories after collection. Upon receipt at Springborn Smithers, sample containers will be inspected for leakage or damage and the sample identity recorded. If storage is required, the samples will be refrigerated at approximately 4°C. In addition, if possible, a sediment sample will be collected from an uncontaminated location near the site of interest to be used as a reference sediment. A laboratory control sediment, prepared or collected by Springborn Smithers Laboratories, will be included in the test to evaluate performance of the test organisms and exposure system. Prior to use, each sediment sample will be sieved through a 2 mm stainless steel sieve to remove large particles. The test will be initiated within 14 days of sediment collection.

2.2.2 Test Vessels

The test vessels used in the static-renewal toxicity test will be 300 mL glass beakers that are chemically clean. Each test vessel has a 2-cm hole cut on the top portion of the vessel and is covered with 40-mesh Nitex® screen for drainage. Each vessel will contain 100 mL (approximately 2 cm layer) of sediment and 175 mL of overlying water. Test vessels will be cleaned by an appropriate method to remove residue of test substance previously used (i.e., acid to remove metals and bases; detergents and organic solvents to remove organic compounds) and rinsed several times using diluent water.

2.2.3 Overlying Water

Water from a 100 meter bedrock well is pumped to a concrete reservoir where it is supplemented on demand with untreated, unchlorinated, Town of Wareham well water. The water is characterized as being "soft" with a normal pH range of 6.9 - 7.7, a total hardness of 30 - 60 mg/L and a specific conductance of 110 - 160 µmhos/cm. The pH, total hardness, alkalinity, and specific conductance of this water will be monitored weekly at a central location in the laboratory to assure that these parameters are within the normal, acceptable ranges. Total hardness and alkalinity will be determined according to *Standard Methods for the Examination of Water and Wastewater*, (APHA, 1992).

The quality of the water is judged by periodic analyses of representative samples conducted to ensure the absence of potential toxicants, including pesticides, PCBs and selected toxic metals, at concentrations which may be harmful to midge larvae, as well as the ability of midge cultures to survive and reproduce in the water free of stress.

2.3 Test Procedures

2.3.1 Test Concentration

Eight replicates will be maintained for each sediment sample consisting of 100% whole sediment sample (no dilutions). A common reference control, conducted with eight replicates, will be used to evaluate the survival and growth potential of the test organism in a non-contaminated sediment. If a reference control sediment is not provided, a laboratory control sediment, prepared or collected by Springborn Smithers Laboratories, will be used to evaluate the survival and growth potential of the test organisms. The laboratory control sediment will also be conducted with eight replicates. Ten midges (second to third instar) per replicate (80 organisms per sediment sample or control) will be used to initiate the test.

2.3.2 Test Initiation

The day before test initiation (day -1) test sediment, reference control and laboratory control sediments will be added to the replicate test vessels and the overlying water added. The water will be added gently to prevent re-suspension of the sediment layer in the water column. This allows the sediment and water to equilibrate prior to addition of the test organisms.

Midge larvae (5-11 days old) will be removed from the cultures. Ten larvae will be randomly selected and pipetted into each replicate test or control vessel. This procedure will be repeated until all vessels contain ten larvae (eighty per test sample and control).

2.3.3 Renewal of Overlying Water

During the 10-day study, the overlying water will be renewed by adding two volume additions (i.e., 350 mL) per day using an intermittent delivery system in combination with a calibrated water-distribution system (Zumwalt et al., 1994). The intermittent delivery system will be calibrated to provide 1 liter of water per cycle to the water-distribution system, which subsequently provides 50 mL of water per cycle to each replicate test chamber. The water delivery system cycles 7 times per day, providing 2 volume additions every 24 hours. Delivery of two volume replacements per day is sufficient to provide consistent and acceptable water quality characteristics throughout the duration of the 10 day exposure.

2.3.4 Photoperiod

The test vessels will be illuminated to a light intensity of 30-100 footcandles using a combination of fluorescent bulbs. A 16-hour light, 8-hour dark photoperiod will be maintained with an automatic timer. Sudden transitions from light to dark and vice versa will be avoided.

2.3.5 Measurement of Water Quality Variables

Total hardness, alkalinity, specific conductance, pH and ammonia will be determined at the beginning and end of the test in the overlying water from a composite sample from all eight replicate vessels. The composite sample will be taken from 1 to 2 cm from the sediment surface using a pipet. Dissolved oxygen and temperature will be measured in

all replicate vessels at test initiation and test termination. Dissolved oxygen and temperature will be monitored daily in one alternating replicate during the course of the study (test days 1-9). Temperature will be monitored continuously in the waterbath using a minimum-maximum thermometer. Readings of temperature extremes will be recorded daily.

2.3.6 Dissolved Oxygen

Total dissolved oxygen will not be allowed to drop below 2.5 mg/L for the duration of the study. Aeration (with oil-free air) will be initiated to raise and maintain the dissolved oxygen concentration at or above 2.5 mg/L.

2.3.7 Temperature

Water temperature of the test solutions will be maintained at 23 ± 1°C by conducting the study in a temperature controlled water bath at the appropriate test temperature.

2.3.8 Biological Data

Survival of the midge larvae will be determined in each test vessel at test termination (test day 10) by sieving the sediment to remove all surviving midges. In addition, daily observations of organism behavior (e.g., sublethal effects) and characteristics of sediment and overlying water will also be observed and recorded daily. The growth of surviving midge larvae, as ash-free dry weight, in each replicate will also be recorded at test termination. All surviving midge larvae from each replicate vessel are pooled and then placed in an ashed weighing tin. The weighing tin and pooled larvae are then dried at 60°C for 24 hours. The weighing tins containing the pooled dried larvae are then ashed in an oven at 550°C for 2 hours. The ashed tins are then cooled to room temperature in a dessicator and then weighed on a calibrated analytical balance to the nearest 0.01 mg.

2.3.9 Test Acceptability

At termination of the study, mean survival in the laboratory control must be ≥ 70% and mean growth must be ≥ 0.48 mg ash free dry weight per midge larvae.

3.0 STATISTICAL ANALYSIS

The mean survival and growth of organisms exposed in each test sediment and reference control sample will be tested for normality and homogeneity of variance using Shapiro-Wilks Test and Ba F-Test. If the data set passes these two tests, then a parametric method (e.g., ANOVA 2-Sample T-Test or Dunnett's Test) will be used to evaluate the results of the mean survival and growth of each test sample for significant adverse effects. If the data set fails the test for normality and homogeneity of variance, then a non-parametric method (e.g., Steel's Many-One Rank Test) will be used to determine significant adverse effects. If necessary, mean survival values will be transformed (e.g., arcsine square).

4.0 REPORTING

The raw data and final summary report will be reviewed by the Study Director. The test results will be presented in an outline format on a per sample basis.

5.0 REFERENCES

Adams, W.J., R.A. Kimerle, and R.G. Mosher. 1985. Aquatic safety assessment of chemicals sorbed to sediments. In: *Aquatic Toxicology and Hazard Assessment: Seventh Symposium* ASTM STP 854. R.D. Cardwell, R. Purdy and R.C. Bahner, Eds. American Society for Testing Materials. 1985. pp. 429-453.

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STANDARD OPERATING PROCEDURE

CARBON, TOTAL ORGANIC IN SOIL

GEN-ASTM
Revision 3
July 3, 2001

Approved By:

Supervisor	7/27/01 Date
QA Manager	8-2-01 Date
Laboratory Manager	8/2/01 Date

COLUMBIA ANALYTICAL SERVICES, INC.
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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: 3/10/12
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DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

CARBON, TOTAL ORGANIC IN SOIL

1. SCOPE AND APPLICATION

1.1. This procedure is applicable to the determination of Total Organic Carbon (TOC) using ASTM method D4129-82, modified for soil and sediment matrices (Puget Sound Estuary Program and Lloyd Kahn). Total organic carbon is a measure of the total amount nonvolatile, partially volatile and particulate organic compounds in a sample. Sample should be treated to remove inorganic carbon (carbonates, bicarbonates, free CO_2 etc.), prior to analysis, as these compounds will interfere with true readings.

1.2. This method is applicable to all soils and sediments and most matrices that can be dried and shatter-boxed to a fine powder.

1.3. Results are reported as percent (%) carbon, and the applicable range is the MDL - 100%. The Method Reporting Limit (MRL) for TOC on soils is 0.05%, dry weight basis. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The Method Detection Limit (MDL) has been determined at 0.02%.

2. METHOD SUMMARY

2.1. Samples are combusted in an oxygen atmosphere to convert organic and inorganic forms of carbon to CO_2. The combustion temperature is selected to completely oxidize all carbon forms. The combustion product gases are swept through a barium chromate catalyst/scrubber to ensure that all of the carbon is oxidized to CO_2. Other potentially interfering product gases such as SO_2, SO_3, HX, and NO_x are removed from the gas stream in a series of chemical scrubbers. The CO_2 is then swept to the coulometer where it is detected by automatic, coulometric titration, with coulometric end point indication.

2.2. The coulometer cell is filled with a partially aqueous medium containing ethanolamine and a colorimetric indicator. When a gas stream passes through the solution, CO_2 is quantitatively absorbed. CO_2 reacts with the ethanolamine to form a strong titratable acid which caused the indicator to fade. The titration current automatically turns on and electrically generates base to return the solution to its original color.

3. DEFINITIONS

3.1. Analysis Batch - Samples are analyzed in a set referred to as an analysis batch. The batch begins with calibration/standardization followed by QC analyses and samples. The batch ends when the QC analyses and set of samples has been completed.

3.2. Method Blank - The method blank is an artificial sample (empty boat) designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.3. Laboratory Control Sample (LCS) - A standard of known TOC concentration which is used to ensure that the analysis produces an accurate measurement of TOC in samples analyzed in the batch.

4. INTERFERENCES

4.1. Acidic and other gases, including SO_2, SO_3, H_2S, HCl, HBr, HI, Cl_2, and NO_x can be effectively removed using scrubbers such as KI, Ag_2SO_4, $AgNo_3$, and MnO_2.

4.2. Volatile organics may be lost in the decarbonization process.

5. SAFETY

5.1. Refer to the CAS Safety Manual for general safety procedures. Protective equipment should be used at all times. Protective equipment includes safety glasses, gloves and a lab coat.

5.2. Disconnect teflon tubing from furnace at check valve whenever system is not in use or when O_2 flow is turned off or furnace temperature is reduced. If the carbon cathode solution should be siphoned through a failed check valve into the magnesium perchlorate scrubber potentially explosive DMSO-perchlorate could be formed.

5.3. Avoid skin contact with all chemicals.

5.4. Do not attempt to combust large samples of organic or other materials that will react with pure oxygen. Such samples can cause the pyrolysis tube to explode.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

Samples can be collected in glass or plastic containers. Samples are preserved by storage at 4±2°C. Samples are analyzed within 28 days of collection.

7. APPARATUS AND EQUIPMENT

7.1. Induction furnace, Coulometrics Incorporated.

7.2. Analytical balance, 0.1mg accuracy.

7.3. Desiccator.

7.4. Platinum or quartz combustion boats.

7.5. Hydrochloric acid (HCl), 10%.

7.6. Sample scoop.

7.7. Porcelain dishes.

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Standards

8.1.1. Urea - 20% carbon. Use 10 μg.

8.1.2. Benzoic Acid - 68.8% carbon. Use 5 μg. Alternatively, a purchased standard of a known TOC value can be used (ERA #542, TOC/TKN/T. Phos/Ammonia in Soil).

8.2. Reagents

8.2.1. Hydrochloric acid, 10%.

8.2.2. Carbon Cathode Solution. Dimethyl Sulfoxide; DMSO. Purchased from Coulometrics Inc. as a prepared solution. Used for coulometer solution.

8.2.3. Anode Solution. Dimethyl Sulfoxide and potassium iodide. Purchased from Coulometrics Inc. as prepared solution.

8.2.4. Manganese dioxide. Gas scrubber solution.

8.2.5. Potassium Hydroxide. Gas scrubber solution.

8.2.6. Potassium Iodide. Anode chemical.

8.2.7. Magnesium Perchlorate desiccant

8.3. Consumables - Platinum boats and miscellaneous glassware, specifically glass ladles.

9. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10. PREVENTIVE MAINTENANCE

Maintenance is performed as follows:

Maintenance Item	Frequency
Cell	Clean daily with methanol and water to clean frit
Mg Perchlorate Scrubber	change daily
KOH Scrubber	change monthly
NOX scrubber	change as needed
Repack Combustion Column	as needed
Post Combustion Column	as needed

11. PROCEDURE

11.1. Sample Preparation.

11.1.1. Turn furnace on to #6 (≈1000°C). Allow furnace to warm-up for about 2 1/2 hours. Turn on oxygen to ≈ 5 psi and 75 to 125 ml/min at flowmeter.

11.1.2. Clean quartz boats. Scrape out old sample and rinse boats with DI water. Place boats in crucible and muffle for at least 10-15 minutes. Remove boats and place in desiccator until ready for use.

11.1.3. Samples should be dried at 70°C and homogenized prior to analysis.

11.1.4. As a rule, the darker (or closer to black) a sample is, the more carbon it contains. Place a small portion of sample on a watch glass. Add 1 drop of 10%

HCl. Watch for effervescence or bubbling. If bubbles are present, the sample contains inorganic carbon (CO_3). If sample bubbles, reduce sample size to prevent sample from bubbling out of boat. If sample is dark, wood product or sludge reduce sample volume to 5 → 10mg. Normal sample volume = 50mg. After boats are loaded with sample add 1 to 2 drops 10% HCl. Place boats in 70°C oven to dry. If samples bubbled when acid was added, add 1 to 2 drops more acid and dry at 70°C. Continue acidifying and drying until samples no longer bubble. Place samples in desiccator until ready for analysis.

11.2. Apparatus Preparation.

11.2.1. Fill cell with carbon cathode solution to 100 → 125 ml, drop in stir bar. Place cell top on snug.

11.2.2. Cover bottom of anode cell with KI. About 2 small scoops.

11.2.3. Add carbon anode solution to cell such that when anode is inserted in the anode cell, the anode solution level is the same as the cathode solution level.

11.2.4. Place cell in coulometer cell holder.

11.2.5. Turn on detector lamp and stir plate. (Power on)

11.2.6. Rotate cell until maximum transmittance is obtained. 60% or better. 100% transmittance is difficult to obtain due to the color of the carbon cathode solution.

11.2.7. With oxygen bubbling to cell and maximum transmittance obtained, turn on the current to the anode and cathode. The carbon cathode solution will begin to titrate to a blue color.

11.2.8. Change Magnesium Perchlorate desiccant after each bracket.

11.2.9. The instrument is now ready to run.

11.3. Calibration and Standardization.

11.3.1. Burn both ladles for five minutes each to remove any residual TOC.

11.3.2. Establish baseline.

11.3.2.1. After placing ladles in sample inlet, allow system to purge for 1 minute.

11.3.2.2. Burn three boats empty five minutes each. The average of the three runs is the baseline.

11.4. Analysis.

11.4.1. Place one platinum boat in a ladle. Place the ladle in the sample inlet and purge for 1 minute. Simultaneously insert the sample into the furnace, press the reset button on the coulometer and start the timer for five minutes.

11.4.2. After five minutes, obtain a reading from the instrument. Remove the ladle from the furnace. (Occasionally, a high sample may require longer than 5 minutes to complete the titration).

11.4.3. Load the other ladle with the next platinum boat. Remove the ladle in use from the inlet port and insert the next ladle.

11.4.4. Repeat steps 10.4.1 through 10.4.3 until all samples are analyzed.

12. QA/QC REQUIREMENTS

12.1. Refer to the SOP *for Analytical Batches and Analytical Sequences* for batching instructions. Project-specific batching protocols may also be required.

12.2. QC Samples Required.

12.2.1. LCS (Benzoic Acid) - An LCS must be analyzed with each batch of 20 or fewer samples. Analyze ≈5mg. The acceptance criteria for the LCS is 58.5% - 79.1% carbon for benzoic acid. Alternatively, ERA standard (see 7.1) may be used. The acceptance criteria for this LCS is ± 15% of the true value.

12.2.2. Method Blank - Burn one empty boat per batch of 20 or fewer samples. Method Blank must be $<0.05\%$ carbon.

12.2.3. CCV (Continuing Calibration Verification) - A CCV must be analyzed every tenth analysis. Urea CCV must be 18.0% - 22.0% carbon.

12.2.4. CCB (Continuing Calibration Blank) - A CCB must be analyzed following every CCV.

12.2.5. Sample duplicate - One sample per batch of 20 or fewer samples must be analyzed in duplicate. Duplicates should be 20% RPD, if > five times the MRL.

12.2.6. Matrix Spike - One spike must be analyzed with each batch of 20 or fewer samples. The acidified sample will be spiked with a known amount of urea.

12.2.7. See Table 1 for a summary of acceptance criteria and corrective actions.

13. DATA REDUCTION AND REPORTING

13.1. Calculate % carbon as follows:

$$\%Carbon = \frac{(Gross\ reading\ -\ baseline\ \mu g)(0.1)}{mg\ sample\ analyzed}$$

13.2. For duplicate analyses, calculate relative percent difference as follows:

$$RPD = \frac{S_1 - S_2}{Avg} * 100$$

where S1 = Sample with higher value
S2 = Sample with lower value
Avg = Average of the two sample values

13.3. Calculate percent recovery as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

13.4. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified above. Average, RPD, spike level and spike recovery are entered on spreadsheet for corresponding samples. All data will be initialed, dated and attached to required data quality worksheet.

13.5. The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in the CAS network

directory R:\WET\WIP. These forms are made from templates located in R:\WET\FORMS.

13.6. Refer to the *SOP for Laboratory Data Review Process* for general guidelines for data review.

13.7. Reporting

13.7.1. Total organic carbon is reported as % carbon, normally on a dry weight basis. Results may be reported on an as received basis.

13.7.2. The Method Reporting Limit is 0.05% carbon, on a dry weight basis.

13.7.3. Report all results to three significant figures.

13.7.4. Bench sheets are labeled "Total Organic Carbon, TOC". These benchsheets, located in Appendix I, should be in use at all times during TOC analysis.

14. CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for Nonconformity and Corrective Action for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for Report Generation or in project-specific requirements.

15. METHOD PERFORMANCE

15.1. This method is validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP for The Determination of Method Detection Limits (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the

volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the laboratory Safety Manual, Sections 6 and 7.

18. REFERENCES

18.1. Coulometrics Inc. Instruction Manual, Model 5020.

18.2. EPA Method Modified 415.1.

18.3. Total Organic Carbon, Method 9060, EPASW846, Test Methods For Evaluating Solid Waste, Third Edition, September 1986, Revision 0.

18.4. Total Organic Carbon(TOC), Conventional Sediment Variables, Puget Sound Estuary Program, March 1986.

18.5. Determination of Total Organic Carbon in Sediment, Lloyd and Kahn, U.S.E.P.A Region II, July 1998.

18.6. ASTM Method D4129-88

TABLE 1

Summary of Corrective Actions

Method Reference	Analysis	Control Item	Acceptance Criteria	Corrective Action
ASTM Method D4129-82	TOC (Soil)	Urea	±10%	Re-analyze all samples affected.
		Benzoic Acid	± 15%	Re-Analyze.
		Method Blank	<0.05%	Re-analyze. If still high, clean boats and start over.
		Sample Duplicate	20% RPD	Analyze a triplicate. Homogenize again and reanalyze.
		Sample Spike	75-125%	Re-analyze.

APPENDIX I

BENCHSHEETS

Columbia Analytical Services, Inc.

Service Request #:

Analysis For: Total Organic Carbon (TOC)

Method: ASTM D4129-82 (Combustion/Coulometric)

Matrix: Soil / Dry Weight Basis

Sample Number	mg Sample Injected	Sample Reading, µg C	Baseline Reading, µg C	Net µg C	% Carbon

Acid Purge Time: 1 minute **Reading Time:** 5 minutes **TOC % =** (Net Reading)(µg 0.1) / mg Sample Injected

CCV : Urea Fisher (lot #0953513) ID#: TOCS/1-10-B TV = 20.0%C

CCV1 =97 CCV2 =

LCS: ERA Cat#: 542 Lot#: 1102 ID#: TOCS/1-10-D TV = 1.16%C REC%=

Comments :

X= RSD= SPK%REC=

Analyzed By:	Date:
Reveiwed By:	Date:

TOC.XLT

Columbia Analytical Services, Inc.

Service Request #:

Analysis For: Total Organic Carbon (TOC)

Method: ASTM D4129-82 (Combustion/Coulometric)

Matrix: Soil / Dry Weight Basis

Sample Number	mg Sample Injected	Sample Reading, µg C	Baseline Reading, µg C	Net µg C	% Carbon

Acid Purge Time: 1 minute **Reading Time:** 5 minutes

$$\text{TOC \%} = \frac{(\text{Net Reading})(\mu g\ 0.1)}{\text{mg Sample Injected}}$$

CCV : Urea Fisher (lot #0953513) ID#: TOCS/1-10-B TV = 20.0%C

LCS: ERA Cat#: 542 Lot#: 1102 ID#: TOCS/1-10-D TV = 1.16%C % Rec =

Comments :

Analyzed By:	Date:
Reveiwed By:	Date:

TOC Soil Benchsheet

Sample #	mg Sample	Reading	Date Baked

TOC Soil Benchsheet

Sample #	mg Sample	Reading	Date Baked	Baseline
CCV-1				
CCB-1				
LCS				
MB				Avg
CCV-2				
CCB-2				
CCV-3				
CCB-3				
CCV-4				
CCB-4				
CCV-5				
CCB-5				

ARCADIS

Appendix B-2

ARCADIS Standard Operating Procedures

ARCADIS

Table of Contents for the ARCADIS Standard Operating Procedures (SOPs)

Rinseate Blank Collection	No.	T16
Field Log Book Entries	No.	T102
pH, Specific Conductance, and Temperature Measurements with Horiba U-10	No.	T106
Field Chain-of-Custody	No.	T107
Equipment Decontamination	No.	T108
Surface Sediment Sampling for Sediment Chemistry and Toxicity Tests	No.	T113
Field Classification of Soils ASTM D2488-93	No.	T115

ARCADIS

Rinseate Blank Collection
Standard Operating Procedure **No. T16**

EQUIPMENT:

_____ Distilled water
_____ Water Sampling Log
_____ Sample containers

PROCEDURES:

1. Decontaminate equipment (split spoons, bailer, etc.) according to SOP T108.

2. Following the final distilled water rinse, rinse the sampling device with high purity distilled water and wash the rinseate into sample containers for laboratory analysis.

3. All rinseate blanks must be handled and analyzed in the same manner as investigative samples (see SOP T107 for Chain-of-Custody/Sample Shipment Procedures). Record details of rinseate blank collection on a Water Sampling Log.

QA/QC Requirements:

One rinseate blank per 20 investigative samples or one per day, whichever is greater, must be collected by each sampling crew (i.e., each drill rig team is one sample crew, each groundwater sampling team is one crew, etc.).

ARCADIS

FIELD LOG BOOK ENTRIES
STANDARD OPERATING PROCEDURE **No. T102**

By: M. Hamper
Approved by: G. Vanderlaan
Date: 4/19/96
Revision Number: 3
Date: 4/12/99

1. Applicability

1.1 ARCADIS Standard Operating Procedure (SOP) covers the entries needed in a field log book for environmental investigations.

1.2 This SOP does not address health and safety, emergency procedures, or all the entries that may be needed for a specific project. Refer to other ARCADIS SOPs, the project work plans including the quality assurance project plan, sampling plan, and health and safety plan, as appropriate.

2. Equipment

Field Log Book
Ball point (medium point) pen with black or blue ink (black preferred)

3. Procedure

3.1 The name of the project, project number and project location should be written in indelible ink on the outside of the field log book.

3.2 On the inside of the front cover, write "If Found, Please Return to ARCADIS" and include the appropriate address and phone number, and the name of the person to which the book is assigned.

3.3. Each day of field work, the following should be recorded in the field log book as applicable:

a) Project Name
b) Date and time arrived
c) Work Site Location
d) Names of people on-site related to the project including ARCADIS employees, visitors, subcontractor employees, agency personnel, etc.
e) Describe the work to be performed briefly

f) Indicate the health and safety (H&S) level to be used
g) Record instrument calibrations and checks
h) Record time and general content of H&S briefing
i) Describe the weather conditions

3.4 If H&S monitoring is performed, record the time and results of initial and follow-up monitoring results.

3.5 Note observations including collection of QA/QC samples, delays, well damage, accidents, work plan deviations, instrument problems, and problem resolutions.

3.6 Describe work performed and how documented such as sample core logs, water sampling logs, etc.

3.7 Note final instrument calibrations and checks.

3.8 Sign the log book at the end of each day at a minimum. Draw a line to the end of the page to indicate no further entries on that page. Sign the bottom of each page if possible.

3.9 Make all entries in ink using a ball point pen (medium). If weather conditions prevent the use of the pen, indicate so in the log and use an alternate writing instrument.

3.10 Print or write legibly.

3.11 Problems noted in the log book must be brought to the attention of the project manager in a timely fashion. Problems may be reported in person, on the telephone, or in a written daily log form. If daily logs are prepared and you will not be able to personally give the daily log to the project manager, send the daily log via FAX or overnight courier to the project manager.

ARCADIS

pH, SPECIFIC CONDUCTANCE, AND TEMPERATURE MEASUREMENTS WITH HORIBA U-10 STANDARD OPERATING PROCEDURE

No. T106
By: M. Hamper
Approved by: Greg Vanderlaan
Date: 1/29/97
Revision Number: 1
Date: 4/12/99

1. Applicability

1.1 ARCADIS Standard Operating Procedure (SOP) covers field measurement of pH, specific conductance, and temperature of water samples exsitu using a Horiba U-10 water quality checker. The Horiba can also determine dissolved oxygen (DO) and salinity, but this SOP does not address these parameters. The Horiba must be calibrated at least once per day of use.

1.2 The meter probe consists of a DO sensor, a glass hydrogen ion electrode to measure pH, an alternating four electrode sensor to measure specific conductance, and a thermistor to measure temperature. The DO is measured using the membrane-electrode method. The pH, DO, salinity, and specific conductance measurements are automatically compensated for temperature. The DO is automatically corrected for salinity. Salinity is calculated (as NaCl) from specific conductance and temperature.

1.3 This SOP does not address health and safety, water level measurements, decontamination, chain-of-custody, sample collection, or laboratory analysis. Refer to other ARCADIS SOPs, the project work plans including the quality assurance project plan, sampling plan, and health and safety plan, as appropriate. Refer to pump manufacturer's operation and maintenance manual for pump operating procedures.

2. Equipment

Horiba U-10 water quality checker (Manufacturer's Number 405077).

Parameter	Range	Resolution	Expanded	Temperature Compensation
pH	0-14 std. Units	0.1 pH	0.01 pH	0-50 ^{o}C
Conductivity	0-100 mS/cm	(micro Siemen per centimeter)		0-50 ^{o}C
	0-1 mS/cm	0.01 mS/cm	0.01 mS/cm	0-50 ^{o}C
	0-10 mS/cm	0.1 mS/cm	0.1 mS/cm	0-50 ^{o}C
	10-100 mS/cm	1.0 mS/cm	1.0 mS/cm	0-50 ^{o}C
Temperature	0-50 ^{o}	1 ^{o}C	0.1 ^{o}C	NA

Pre-cleaned glass beaker (150 ml to 250 ml)
Calibration beaker (Part Number 9037-0053-00)
Standard phthalate solution (calibration solution)
Spare meter battery (9-volt)
Tap and Distilled water in polyethylene wash bottle
Field Log Book SOP T102

3. Autocalibration Procedure

3.1 Check dates on the sensor solution. It cannot be used if it is over two months old.

3.2 The meter will autocalibrate pH, and specific conductance. Remove protective rubber cap from meter probe. Rinse meter probe with distilled water.

3.3 Fill the calibration beaker to about 2/3 full with the standard calibration solution.

3.4 Turn on the power and press the MODE key to put the unit into the MAINT mode. The lower cursor should be on the AUTO Sub-mode. If the lower cursor is not on the AUTO Sub-mode, use the MODE key to move the lower cursor to AUTO.

3.5 With the lower cursor on AUTO, press the ENT key. The readout will show CAL. Wait for the upper cursor to move across the four autocalibration parameters (i.e., pH, COND, TURB, and DO) one by one. The upper cursor will blink while the autocalibration is being made.

3.6 When the readout states END, the calibration is completed and the readout will switch to MEAS mode. Measurements may now be made.

3.7 Record that the calibration was performed in the log book.

4. Water Sample Measurement Procedure

4.1 Fill pre-cleaned glass beaker with water sample.

4.2. Turn power on. When the instrument is first turned on, it will be in the MEAS mode (measurement mode). Within 2 seconds, the readout will show that a measurement is being made. The readout will show the last parameter that the upper cursor was on when the previous measurement was made. Use the EXP key to toggle into the expanded readout mode for additional accuracy in the measurement.

4.3 Slowly immerse the meter probe into the sample contained in the glass beaker.

4.4 Observe the readout and when the reading stabilizes, record the measurement result and the type of measurement.

4.5 Use the SELECT key to move the upper cursor and select another type of measurement and repeat step 4.5 until a measurement for pH, specific conductance, and temperature are obtained.

4.6 Turn power off using the OFF key.

4.7 Rinse meter probe with tap water, carefully flushing all of the water sample from the meter probe.

4.8 Fill small rubber cap with tap water and place the rubber cap on the meter probe for storage.

4.9 Store meter in protective case when not in use. Report instrument problems to equipment supplier.

5. Maintenance

5.1 Remove the COND sensor guard and use a soft brush to clean off dust from the sensor unit. Replace the COND guard before taking measurements.

ARCADIS

FIELD CHAIN-OF-CUSTODY
STANDARD OPERATING PROCEDURE **No. T107**

By: M. Hamper
Approved by: G. Vanderlaan
Date: 1/24/97
Revision Number: 1
Date: 4/12/99

1. Applicability

1.1 ARCADIS Standard Operating Procedure (SOP) covers chain-of-custody procedures for the management and transportation of samples from the sampling location to the laboratory.

1.2 This SOP does not address health and safety, water level measurements, decontamination, sample collection, or laboratory analysis. Refer to other ARCADIS SOPs, the project work plans including the quality assurance project plan, sampling plan, and health and safety plan, as appropriate.

2. Equipment/Supplies

Chain-of-Custody Form (one per cooler)
Black or blue ball point pen or equivalent
Resealable bag
Packing Tape
Chain-of-Custody Seals (two per cooler)
Sample labels
Field Log Book SOP T102

3. Procedure

3.1 Complete the sample label with the following minimum information.

- Unique Sample Identification (See project plans or project manager for sample numbering protocol.)
- ARCADIS Project Number
- Sample collection date

- Sample collection time
- Sample type (water, soil, etc.)
- Collection mode (grab or composite)
- Analyses to be performed
- Preservative used
- Name of sampler

3.2 Record sample identification, sample collection time and date in the field log book.

3.3 Store the sample in a cooler (with ice if appropriate), which is maintained under the control of the sampler.

3.4 Complete one chain-of-custody form for each cooler. The receiving laboratory's form may be used, but the form must contain the following information.

- Project Number
- Project Name
- Laboratory Name
- Sampler(s) Name
- Sample Identification
- Sample Collection Date
- Sample Collection Time
- Sample Type (water, soil, etc.)
- Sample bottle/container description
- Analyses to be performed
- Number bottles per analysis
- Total number of bottles/containers
- Signature blocks for person(s) responsible for samples
- Sample delivery method
- Courier bill number (if courier used)

3.5 If samples are to be delivered in person, place the chain-of-custody form in a resealable bag and tape the resealable bag to the inside of the cooler lid. The samples must be maintained under the control of the sampler until delivery. Upon delivery, remove the form from the resealable bag, and sign (with date and time samples relinquished). The laboratory representative must also sign the form

(with date and time samples received). Keep the last copy of the form for our records (typically the pink copy).

3.6 If the samples are to be sent via courier, add the courier bill number to the chain-of-custody form. Sign the form with date and time samples were relinquished to the courier. The courier should not sign the chain of custody form. Place the chain-of-custody form in a resealable bag and tape the resealable bag to the inside of the cooler lid. Complete packing of the cooler as needed, and close the cooler. Tape the cooler closed with packing tape and tape the courier bill to the top of the cooler and a ARCADIS mailing label with the laboratory's address and phone number. Affix the two custody seals to the cooler, placing one on the front left side and one on the front right side of the cooler. The seals must cross the packing tape such that they would be broken if the cooler is opened. Place clear packing tape over the custody seal.

3.7 If the samples are sent via courier, call the laboratory and let them know the samples are being shipped. This is especially important for Saturday deliveries. After the samples are shipped, call the laboratory to verify receipt (when receipt expected).

4. Attachments

Example Sample label
Example Chain of Custody Form
Example Custody Seal

ARCADIS

EQUIPMENT DECONTAMINATION
STANDARD OPERATING PROCEDURE **No.T108**

By: P. Fuchsman
Approved by: T. Barber
Date: 6/20/02
Revision Number:
Date: 6/20/02

1. Applicability

1.1 ARCADIS Standard Operating Procedure (SOP) covers the decontamination equipment, field procedures, and documentation procedures necessary to properly decontaminate equipment and instruments used during the conduct of the Sampling and Analysis Plan (SAP) for the Study Area. Decontamination is the process of neutralization, washing, and rinsing exposed outer surfaces of equipment to minimize the potential for contaminant migration and/or cross-contamination. This procedure does not apply to personnel decontamination, which is described in the Health and Safety Plan (HASP).

1.2 The overall objective of multimedia sampling programs is to obtain samples, which accurately depict the chemical and physical conditions at the sampling site. Extraneous contaminated materials can be brought to a sampling location and/or introduced into the medium of interest during the sampling program with equipment previously used at another sampling site. Trace quantities of these materials can introduce indigenous concentrations into other samples and lead to false positive analytical results and, ultimately, to an incorrect assessment of the conditions associated with a site. Decontamination of sampling equipment (e.g., water and sediment samplers, containers, and other equipment) and field support equipment (e.g., sampling vessel) is required so that sampling cross-contamination is prevented, and on-site contaminants are not carried away from the Study Area.

2. Equipment/Supplies

The following equipment list contains materials, which may be needed in carrying out the procedures contained in this SOP. Not all materials on the equipment list may be required for each sampling investigation conducted under this SAP.

Bristle brushes
Wash/rinse tubs

Low phosphate detergent
10 percent nitric acid, ultrapure
Acetone or methanol plus hexane (pesticide grade or better in separate Teflon bottles)
Distilled/De-ionized ("analyte-free") water
Stainless steel bowl
Tap water (from any treated municipal water supply)
High-pressure/steam cleaner
Appropriate health and safety equipment
Sample container(s) for rinsate blank, if collected
Field log book

3. Procedure

3.1 Sampling apparatus that comes into contact with sample matrices will be decontaminated prior to use in the field to minimize cross-contamination. While performing the decontamination procedure, "phthalate-free gloves," such as nitrile or butyl rubber, must be used in order to prevent phthalate contamination of the sampling equipment or the samples. Sampling equipment will be decontaminated in the designated area for decontamination.

The decontamination procedure followed by EPA Region II (EPA Region II, CERCLA Quality Assurance Manual, October 1989, Revision 1) will be used prior to each sampling event for sampling equipment that will come in contact with the media to be sampled. The EPA Region II procedures are summarized below:

Wash and scrub with low phosphate detergent
Tap water rinse
Rinse with 10% nitric acid (HNO_3) ultrapure
Tap water rinse
An acetone only rinse or a methanol followed by hexane rinse (solvents must be pesticide grade or better)
Thorough de-ionized ("analyte-free") water rinse, air dry, and
Wrap in aluminum foil, shiny side out, for transport

If samples are collected only for geotechnical laboratory testing, (e.g., grain size) the sampling equipment will only be rinsed with tap water after washing with low-phosphate detergent solution.

Not all sampling equipment will require the full decontamination procedures listed in EPA Region II CERCLA Quality Assurance Manual. Equipment such as sediment samplers, surface water samplers, tissue homogenizers, and other tissue preparation devices will undergo decontamination as specified by EPA Region II.

The following steps will be used to decontaminate boat anchors, lines, ropes, submersible pump and hose, gill nets, and similar equipment:

Personnel will dress in suitable safety equipment to reduce personal exposure (see HASP).
Equipment will be rinsed with river water.
Rinse water will not be contained.

3.2 Field Instruments and Equipment

Instrumentation should be cleaned as per manufacture's instructions. Probes such as those used in dissolved oxygen, temperature, pH, Eh salinity, and conductivity meters will be carefully wiped clean using sponge and detergent water and rinsed with deionized water. Care will be taken to prevent damage to equipment. When possible, instruments that are difficult to decontaminate, such as cameras and logging instruments, may be protectively wrapped to reduce or eliminate the need for decontamination.

3.3 Rinsate Blanks

Rinsate blanks are a type of QA/QC sample collected to assess the adequacy of decontamination procedures. Rinsate blanks are collected by pouring deionized "analyte-free" water over the sampling equipment (e.g., sampler, knife, bowl, pan or blender) after it has been decontaminated in the field. The rinsate is collected in a stainless steel bowl and transferred to sample bottles for analysis with other samples. Rinsate blanks will be collected at the frequency specified in the QAPP.

3.4 Documentation

The field personnel are responsible for documenting decontamination activities related to their on-site activities. Observations and data will be recorded with ink in a field logbook with consecutively numbered pages. The information in the field logbook will include the following as a minimum:

Responsible person's name
Date and time of activity
Information concerning items decontaminated and the procedure utilized
Information related to taking of rinsate blank samples

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SURFACE SEDIMENT SAMPLING FOR
SEDIMENT CHEMISTRY AND TOXICITY TESTS
STANDARD OPERATING PROCEDURE **No. T113**

By: P. Fuchsman
Approved by: T. Barber
Date: 6/20/02
Revision Number:
Date: 6/20/02

1. Applicability

1.1 ARCADIS Standard Operating Procedure (SOP) describes the procedures to be followed for the collection of composite surface sediment samples from the Study Area and reference area. Surface sediment sampling will be performed to collect sediment chemistry data in conjunction with the ecological investigations being performed under the Sampling and Analysis Plan (SAP) for the Study Area, and to perform bulk sediment toxicity tests.

1.2 This SOP does not address health and safety, equipment decontamination, field parameter measurements, sample preservation, chain-of-custody, or laboratory analysis. Refer to other ARCADIS SOPs, the project work plans including the quality assurance project plan, sampling plan, and health and safety plan, as appropriate.

2. Equipment/Supplies

2.1 The SAP identifies sampling locations, frequency of sampling, sample type, and analytical procedures. The field team is responsible for reviewing the SAP prior to conducting field activities and ensuring that all field equipment, including sample containers with preservatives are available and in acceptable condition.

Equipment to be used during collection of sediment samples may include, but is not limited to the following:

Sampling vessel
Stainless steel Ponar or equivalent grab sampler
Stainless steel bowl or equivalent container
Sample containers
Ice

Insulated coolers
Sample identification labels/tags
Waterproof marking pens
Stainless steel spoons/spatula
Decontamination SOP (T108)
Chain-of-custody form (T107)
Field log book (T102)

3. Equipment Decontamination

3.1 Decontamination of surface sediment sampling equipment will be performed between each composite sampling location/event in accordance with procedures outlined in SOP No. 108 – Equipment Decontamination. Personnel decontamination procedures are contained in the HASP.

4. Sampling Locations

4.1 The position and depth of the sampling location will be established. The depth of the sampling location will be determined using either a fathometer or weighted demarcated line.

5. Surface Sediment Sampling Collection and Preparation

5.1 The following protocol shall be implemented, as practicable, for collecting composite surface sediment samples at the sampling locations. The analytical sample volume requirements for sediment chemistry samples are provided in the QAPP. The appropriate sample containers will be provided by the contract laboratory(s) prior to the field program.

5.2 Sediments will be homogenized following EPA Region II guidance using the "core and quarter" method. Rocks, shells, and other debris will be first removed from the sample. The sediment is removed from the sampling device and placed in a stainless steel pan, then gently mixed using a stainless steel spoon. The sediment in the pan is scraped from the sides, corners and bottom of the pan, rolled to the middle of the pan, and initially mixed. The sample should then be quartered and moved to the four corners of the pan. Each quarter of the sample is

mixed individually, and then rolled to the center of the container and the entire sample mixed again until fully homogenized. Sediment mixing should be kept to the minimum required to achieve homogenization, to limit effects on chemical partitioning and other sediment characterization.

5.3 Discrete samples will be collected to ensure that the resulting composite sample is spatially representative of the desired sampling area.

1 Label the appropriate sample containers with the appropriate pre-printed sample identification labels.

2 If sampling a wadable creek or shallow water body, attach the line or cable to the Ponar or equivalent grab sampler and spread the sampling jaws until they lock in the open position. Firmly hold the line and drop the open sampler into the surface water. After the sampler closes on the bottom, slowly and carefully retrieve the Ponar sampler to avoid the possibility of premature opening or loss of sample.

3 If sampling in deep water from a boat, attach the line or cable to the Ponar or equivalent grab sampler and spread the sampling jaws until they lock in the open position. Secure the free end of the sampler line to a firm support (e.g., the boat, raft or dock) and deploy the sampler into the surface water slowly. Provide the sampler with at least two feet of slack sampling line to facilitate settling of the sampling jaws into the sediment. After the sampler closes on the bottom, slowly retrieve the Ponar sampler to the surface. Care should be taken to retrieve the sampler as smoothly as possible to avoid the possibility of premature opening or loss of a portion of the sample.

4 Place the closed sampler into an appropriate clean container (e.g., stainless steel bowl) and open the jaws to release the sediment sample. The sample container should be sufficiently large to hold several discrete grab samples.

5 Repeat steps 2/3 through 4 for each discrete sample to be taken.

6 Place each of the discrete samples into the sample container with the previous samples until the approximate target volume has been obtained.

7 Homogenize the mixture of discrete samples in the stainless steel bowl. The resulting composite sample can be used for chemical analysis and/or toxicity testing.

8 Fill the appropriate sample containers with sediment using a stainless steel spatula or spoon.

9 Close and seal each container.

10 Check label on each sample container and cover the label with clear plastic tape. Wrap with bubble wrap and place in sealable plastic bag. Refrigerate or place in cooler containing ice. Ship to laboratories on ice.

11 Complete the appropriate chain-of-custody form for each sample container.

6. Sampling Containers, Preservation, Handling and Tracking

6.1 Sample containers, handling and preservation procedures for the sediment chemistry samples are described in SOP No. 107 – Field Chain-of-Custody.

6.2 For the toxicity testing, the sample containers will be shipped in coolers containing ice to the analytical laboratory for testing.

7. Quality Control Samples

7.1 To help identify potential sources of sample contamination and evaluate potential error introduced by sample collection and handling, field quality control samples (QC samples) will be collected during the sediment sample collection and processing. The QC samples will be labeled in accordance with SOP No. 107 – Field Chain-of-Custody, and sent to the laboratory with the other samples for analysis. QC samples for sediment sample collection will include rinsate samples, field duplicate samples, and matrix spike samples, and will be collected at the frequency specified in the QAPP.

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FIELD CLASSIFICATION OF SEDIMENTS/SOILS
ASTM D2488-93
STANDARD OPERATING PROCEDURE **No. T115**

By: M. Hamper
Approved by: G. Vanderlaan
Date: 2/24/97
Revision Number: 1
Date: 4/12/99

1. Applicability

1.1 ARCADIS Standard Operating Procedure (SOP) covers the field description and classification of soils following ASTM D2488-93, which is a qualitative, visual-manual method for materials that pass a 3-inch sieve. The ASTM D2488-93 practice is attached. This SOP indicates that the user should follow the ASTM practice and identifies which options should be utilized.

1.2 This SOP does not address health and safety, sample collection, or laboratory analysis. Refer to other ARCADIS SOPs, the project work plans including the quality assurance project plan, sampling plan, and health and safety plan, as appropriate.

2. Equipment/Supplies

Stainless steel knife or spatula
Water/squirt bottle
Tape measure
Grain-size chart/color chart (Forestry Suppliers #77325)
Jar with lid
Hand lens
Field Log Book SOP (T102)

3. Procedure

3.1 Obtain a representative sample of soil from a stratum and record the sampling location and depth.

3.2 Remove particles greater than 3-inches from the soil sample. Note the volume percentage of the greater than 3-inch particles.

3.3 Only the less than 3-inch fraction will be classified. Examine the less than 3-inch fraction of the sample and estimate the percentage of the gravel, sand and fines on a dry weight basis to the nearest 5%. Use the jar method, visual method or wash method in Appendix X4 of ASTM D2488 to estimate the relative percentages of each category. The percentages must add up to 100%. Components present at less than 5% are recorded as "trace, and are not included in the 100% sum total. Dry weight basis rather than volume percent is used for consistency with the laboratory method for soil classification (D2487). The estimate of the dry weight fraction of each category is done visually in the field.

3.4 Determine if the soil sample will be considered fine-grained or coarse-grained. A fine-grained soil contains 50% or more fines. A coarse-grained sample contains less than 50% fines. If the sample is fine-grained, follow the procedure in Section 14 of ASTM D2488. If the sample is coarse-grained, follow the procedure in Section 15 of ASTM D2488.

3.5 Examine the sample for other characteristics including:

- The color of the sample should be noted. The color should be determined on a moist sample.
- Note whether the soil sample is dry, moist or wet.
- Note odors observed if the odor is organic or unusual. Do not attempt to smell contaminated samples if a health threat exists. Describe unusual odors, such as petroleum, chemical, etc.
- Describe the consistency as very soft, soft, firm, hard or very hard. (Not appropriate for soils with significant amounts of gravel.)
- Describe the structure of intact soils.
- Describe the sand size as fine, medium or coarse.
- Describe the gravel size as the smallest sieve opening that the maximum gravel particle will pass.
- Note the shape of gravel, cobbles, and boulders.

- Note the angularity of the sand particles, gravel, cobbles, and boulders.

- Note the presence of roots, root holes, organic material, man-made materials, etc.

- HCL reaction will not be required

3.6 Note the percentages of the fractions as:

Trace -less than 5%
Few -5 to 10%
Little -15 to 25 %
Some -30 to 45%
Mostly 50 to 100%

3.7 Example Descriptions

- **Well Graded Gravel with Sand (GW)—**Mostly fine to coarse, hard subangular gravel; Little fine to coarse, hard subangular sand, trace of fines, maximum size, 75 mm, brown, dry,

- **Lean Clay (CL)—**Mostly clay, little silt, medium dry strength, no dilatancy, medium toughness, and medium plasticity, gray, moist, fissured, firm.

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Appendix B-3

Analytical Laboratory Quality Assurance Manuals

QUALITY ASSURANCE MANUAL

for

Columbia Analytical Services, Inc.
1317 South 13th Avenue
Kelso, Washington 98626
(360) 577-7222
May 31, 2002

Approved by:

Laboratory Director/Technical Director: Jeff Christian

Quality Assurance Manager/Technical Director: Lee Wolf

Technical Director - Metals: Jeff Coronado

Technical Director - Inorganics: Todd Poynair

Technical Director - Organics: Jeff Grindstaff

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DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

2.0 TABLE OF CONTENTS

Section	Revision	Heading	Page
1.0	12.0	Title Page with Provision Signatures	1
2.0	12.0	Table Of Contents	2
3.0	7.0	Introduction and Company Quality Assurance Policy	4
4.0	8.0	Program Description	5
5.0	6.0	Statement of Professional Conduct and Laboratory Practice	10
6.0	9.0	Project Organization and Responsibilities	11
7.0	8.0	Information Management	15
8.0	8.0	Sample Management	18
9.0	8.0	Quality Control Objectives (Precision, Accuracy, and MDLs)	32
10.0	9.0	Quality Control Procedures	35
11.0	9.0	Calibration Procedures and Frequency	43
12.0	8.0	Data Reduction, Validation, and Reporting	48
13.0	10.0	Performance and System Audits	63
14.0	9.0	Preventive Maintenance	66
15.0	8.0	Corrective Action	68
16.0	9.0	Quality Assurance Reports	70
17.0	6.0	Personnel Training	72
18.0	10.0	References for Analytical Procedures	76

Tables		Page
Table 6-1	Summary of Technical Experience and Qualifications	14
Table 8-1	Sample Preservation and Holding Times	22
Table 12-1	Descriptions of CAS Data Deliverables	62
Table 13-1	Current CAS Performance and System Audit Programs	65

Figures Page

Figure 4-1 CAS/Kelso Laboratory Floor Plan 9
Figure 8-1 Sample Container Label and Custody Seal 28
Figure 8-2 Chain of Custody Form 29
Figure 8-3 Cooler Receipt and Preservation Check Form 30
Figure 8-4 Tier V Project Specification Form 31
Figure 12-1 Evaluation of Method Calibration 53
Figure 12-2 Evaluation of Continuing Calibration 54
Figure 12-3 Evaluation of Method Blank and Instrument Blank Results 55
Figure 12-4 Evaluation of Sample Results for Inorganic Analyses 56
Figure 12-5 Evaluation of Sample Results for Organic Analyses 57
Figure 12-6 Evaluation of Surrogate Compound Recoveries 58
Figure 12-7 Evaluation of Duplicate Sample and/or Duplicate Matrix Spike Results 59
Figure 12-8 Evaluation of Matrix Spike Recoveries 60
Figure 12-9 Evaluation of Laboratory Control Sample (LCS) Results 61
Figure 15-1 Nonconformity and Corrective Action Report 69
Figure 17-1 Initial Demonstration of Proficiency Requirements 75

Appendices Page

Appendix A List of QA Program Documents A1
Appendix B Organizational Chart and Resumes of Key Personnel B1
Appendix C Major Analytical Equipment C1
Appendix D Data Quality Capabilities D1
Appendix E Preventive Maintenance Procedures E1

3.0 INTRODUCTION AND COMPANY QUALITY ASSURANCE POLICY

Columbia Analytical Services, Inc. (CAS) is an employee-owned professional analytical services laboratory which performs chemical and microbiological analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sludge, sediment, tissue, industrial and hazardous waste, and other material.

It is a policy at CAS that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. This goal is achieved by ensuring that adequate Quality Control (QC) procedures are used throughout the monitoring process, and by establishing a means to assess performance of these Quality Control and other QA activities. Policies and procedures are established in order to meet the quality objectives of clients, accrediting authorities, and certifying organizations. The Quality System is established to meet the requirements of the National Environmental Laboratory Accreditation Conference (NELAC).

CAS maintains control of analytical results by adhering to written standard operating procedures (SOPs) and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data.

We recognize that quality assurance requires a commitment to quality by everyone in the organization - individually, within each operating unit, and throughout the entire laboratory.

CAS is a network of laboratories. In addition to the Kelso, WA facility, to which this manual is applicable, CAS also operates laboratories in California, Florida, New York, Arizona, and Texas.

The information in this document has been organized according to the format described in *EPA Requirements for Quality Management Plans, EPA QA/R-2,* USEPA, 2001; and *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5,* USEPA, 2001.

4.0 PROGRAM DESCRIPTION

The purpose of the QA program at CAS is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality. The concept of Quality Assurance can be extended, and is expressed in the mission statement of CAS:

> "The mission of Columbia Analytical Services, Inc., is to provide high quality, cost-effective, and timely professional testing services to our customers. We recognize that our success as a company is based on our ability to maintain customer satisfaction. To do this requires constant attention to customer needs, maintenance of state-of-the-art testing capabilities and successful management of our most important asset - our people - in a way that encourages professional growth, personal development and company commitment."

In support of this mission, our QA program addresses all aspects of laboratory operations, including laboratory organization and personnel, standard operating procedures, sample management, sample and quality control data, calibration practices, standards traceability data, equipment maintenance records, method proficiency data (such as method detection limit studies and control charts), document control/storage and staff training records.

4.1 Facilities and Equipment

CAS features over 25,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system has been specially designed to meet the needs of the analyses performed in each work space. Also, CAS minimizes laboratory contamination sources by employing janitorial and maintenance staff to ensure that good housekeeping and facilities maintenance are performed. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types. These specialized areas (and access restrictions) include:

- Shipping and Receiving/Purchasing
- Sample Management Office
  - Controlled-access sample storage areas
- Inorganic/Metals Sample Preparation Laboratories (2)
- Inorganic/Metals "clean room" sample preparation laboratory
- ICP-AES Laboratory
- ICP-MS Laboratory
- AA Laboratory
- Water Chemistry & General Chemistry Laboratories

- Semi-volatile Organics Sample Preparation Laboratories (3)
- Gas Chromatography Laboratory
- Gas Chromatography/Mass Spectrometry Laboratory
- Petroleum Hydrocarbon Laboratory
- Volatile Organics Laboratory
  - Separate sample preparation laboratory
  - Access by semi-volatile sample preparation staff only after removing lab coat and solvent-contaminated gloves, etc.
- Microbiology Laboratory
- Laboratory Deionized Water System
- Laboratory Management, Client Service, Report Generation and Administration
- Data Archival, Data Review and support functions areas
- Information Technology (IT) and LIMS

In addition, the designated areas for sample receiving, refrigerated sample storage, dedicated sample container preparation and shipping provide for the efficient and safe handling of a variety of sample types. Figure 4-1 shows the facility floor plan. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation are appropriate for the procedures in use. Appendix C lists the major equipment, illustrating the laboratory's overall capabilities and depth.

4.2 Technical Elements of the Quality Assurance Program

The Quality Assurance Program provides a platform on which technical operations are based. The program provides laboratory organization, procedures, and policies by which the laboratory operates. The necessary certifications and approvals administered by external agencies are maintained. This includes method approvals and audit administration. In addition, internal audits are performed to assess compliance with policies and procedures. Standard Operating Procedures (SOPs) are maintained for technical and administrative functions. A document control system is used for SOPs, as well as laboratory notebooks, and this QA Manual. A list of QA Program documents is provided in Appendix A.

Acceptable calibration procedures are defined in the SOP for each test procedure. Calibration procedures for other laboratory equipment (balances, thermometers, etc.) are also defined. Quality Control (QC) procedures are used to monitor the testing performed. Each analytical procedure has associated QC requirements to be achieved in order to demonstrate data quality. The use of method detection limit studies, control charting, and preventative maintenance procedures further ensure the quality of data produced. Proficiency Testing (PT) samples are used as an external means of monitoring the quality and proficiency of the laboratory. PT samples are obtained from qualified vendors and are performed on a regular basis. In addition to method proficiency, documentation of analyst training is performed to ensure proficiency and competency of laboratory analysts and technicians. Sample handling and custody procedures are defined in SOPs. Procedures are also in place to monitor the sample storage areas.

The technical elements of the QA program are discussed in further detail in later sections of this QA manual.

4.3 Operational Assessments

There are a number of methods used to assess the laboratory and its daily operations. In addition to the routine quality control (QC) measurements to measure quality, the senior laboratory management staff at CAS examine a number of other performance indicators to assess the overall ability of the laboratory to successfully perform analyses for its clients. On-time performance, Analytical Report defect rate and Customer Invoice defect rate are a few of the measurements performed at CAS that are used to assess performance from an external perspective (i.e., client satisfaction). The use of these and other indicators is outlined in the *SOP for Nonfinancial Performance Measures* (SOP No. ADM-NFPM). A frequent, routine assessment must also be made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload.

CAS utilizes a number of different methods to insure that adequate resources are available in anticipation of the demand for service. Regularly scheduled senior staff meetings, tracking of outstanding proposals and an accurate, current synopsis of incoming work all assist the senior staff in properly allocating resources to achieve the required results. All Requests for Proposal (RFP) documents are reviewed by the Project Chemist and appropriate managerial staff to identify any project specific requirements that differ from the standard practices of the laboratory. Any requirements that cannot be met are noted and communicated to the client, as well as requesting the client to provide any project specific Quality Assurance Plans (QAPP's) if available. A weekly status meeting is also conducted with the laboratory staff by the Client Services Manager to inform the staff of the status of incoming work, future projects, or project requirements.

4.4 Document Control

Procedures for control and maintenance of documents are described in the *SOP for Document Control (ADM-DOC_CTRL).* The procedures described in the SOP include distribution, tracking, filing, and copyrighting of CAS controlled documents. The requirements of the SOP apply to all standards preparation logbooks, instrument maintenance logbooks, run logbooks, standard operating procedures (SOPs), quality assurance manuals (QAMs), quality assurance project plans (QAPPs), safety manuals (SFM), and other controlled CAS documents.

Each controlled copy of a controlled document will be released only after a document control number is assigned and the recipient is recorded on a document distribution list. Filing and distribution is performed by the Quality Assurance Manager, or designee, and ensure that only the most current version of the document is distributed or is in use. A document control number is assigned to logbooks. Completed logbooks that are no longer in use are archived in a master logbook file.

CAS maintains a records system which ensures that all laboratory records (including raw data, reports, and supporting records) are retained and available. The archiving system is described in the *SOP for Data Archiving (ADM-ARCH).*

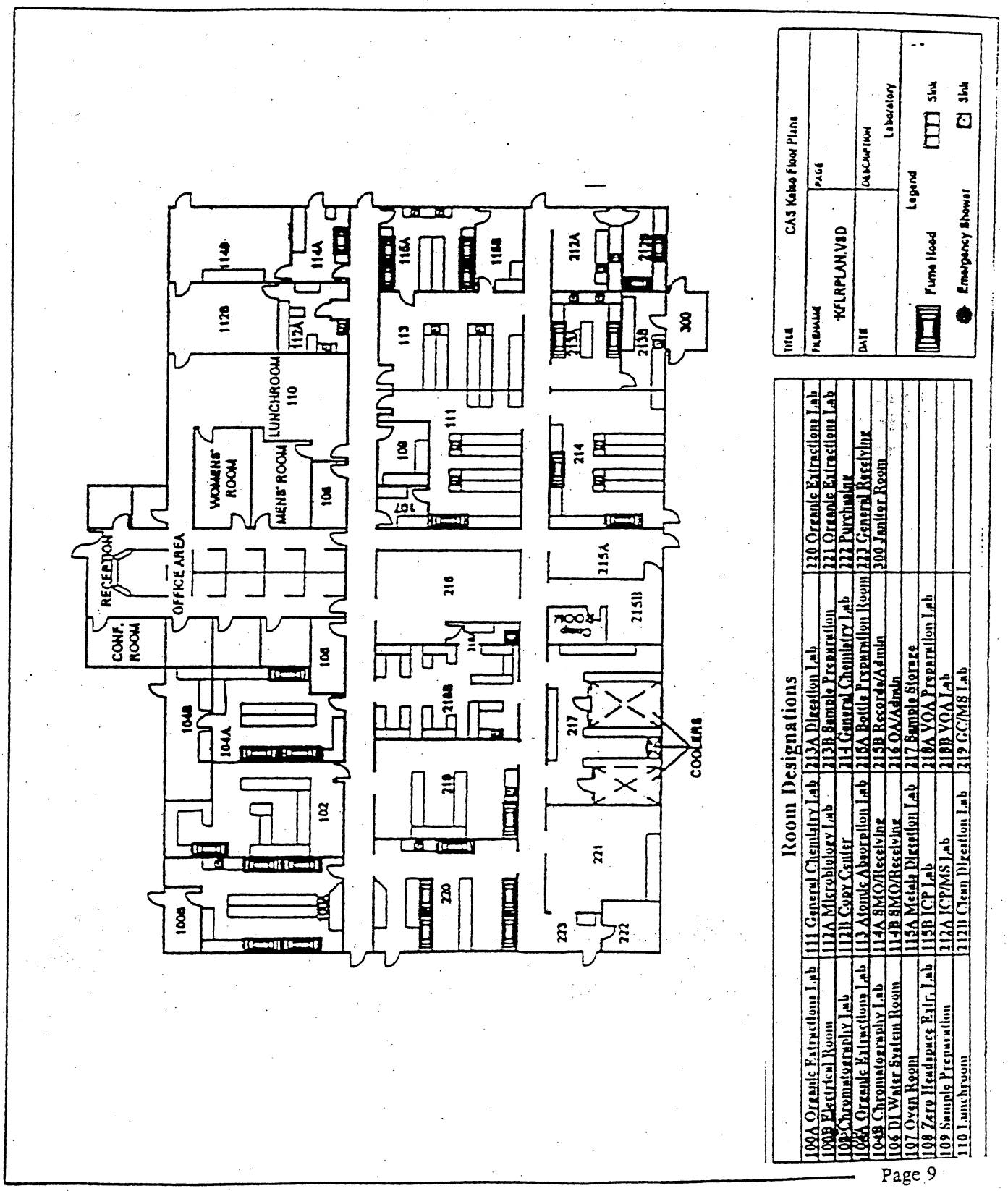
4.5 Subcontracting

Analytical services are subcontracted when CAS/Kelso needs to balance workload and/or when the requested analyses are not performed by CAS/Kelso. However, subcontracting is only done with the knowledge and approval of the client. Subcontracting to another CAS laboratory is preferred over external-laboratory subcontracting. Further, subcontracting is done to capable and qualified laboratories. Established procedures are used to qualify external subcontract laboratories. These procedures are described in the *SOP for Qualification of Subcontract Laboratories (ADM-SUBLAB).* The Quality Assurance Director is responsible for qualifying and oversight of subcontract laboratories.

4.6 Procurement

The quality level of reagents and materials (grade, traceability, etc.) required is specified in analytical SOPs. Department supervisors ensure that the proper materials are purchased. Inspection and verification of material ordered is performed at the time of receipt by receiving personnel. The receiving staff labels the material with the date received. Expiration dates are assigned (by the laboratory user) as appropriate for the material. Storage conditions and expiration dates are specified in the analytical SOP. The procedures for purchasing and procurement are described in the *SOP for Purchasing Through CAS Purchasing Agent (SOP ADM-PUR).* Also, refer to section 10.18 for a discussion of reference materials.

Figure 4-1
CAS/Kelso Laboratory Floor Plan



5.0 STATEMENT OF PROFESSIONAL CONDUCT AND LABORATORY PRACTICE

One of the most important aspects of the success of CAS as a company is the emphasis placed on the integrity of the data that are provided and the services that are performed. To promote product quality, CAS requires certain standards of conduct and ethical performance among our employees. The following examples of documented CAS policy are representative of these standards, and are not intended to be limiting or all-inclusive:

Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action. Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.

Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible. Employee discipline is progressive in its severity and each situation is handled individually in that the discipline is designed to fit the circumstances. Potential disciplinary actions may include a verbal warning, written warning, a second written notice (more severe and more strongly worded than a warning), suspension without pay, demotion, or termination.

It is the responsibility of all CAS employees to safeguard sensitive company and client information. The nature of our business and the economic well-being of our company and of our clients is dependent upon protecting and maintaining proprietary company/client information. All information, data, and reports (except that in the public domain) collected or assembled on behalf of a client is treated as confidential. No information may be given to third parties without the consent of the client. Unauthorized release of confidential information about the company or its clients is taken very seriously and is subject to formal disciplinary action. As a condition of employment, all employees are required to sign and adhere to confidentiality requirements set forth in CAS' "Employee Agreement" at date of hire and upon termination.

CAS makes every attempt to ensure that employees are free from any commercial, financial, or other undue pressures which might affect their quality of work. Related policies and responsibilities are described in the CAS Employee Handbook. At the time of hire, each employee is also required to sign a Commitment to Excellence in Data Quality. All employees receive in-house ethics training (*Assuring Ethical Practices in the Laboratory*) and are periodically reminded of their data quality and ethical conduct responsibilities.

6.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The CAS/Kelso staff, consisting of approximately 110 employees, includes chemists, technicians and support personnel. They represent diverse educational backgrounds and experience, and provide the comprehensive skills that a modern, state-of-the-art analytical laboratory requires. During seasonal workload increases, additional temporary employees may be hired to perform specific tasks.

CAS is committed to providing an environment that encourages excellence. Everyone within CAS shares responsibility for maintaining and improving the quality of our analytical services. The responsibilities of key personnel within the laboratory are described below. Table 1-1 lists the CAS/Kelso personnel assigned to these key positions. Managerial staff members are provided the authority and resources needed to perform their duties. An organizational chart of the laboratory, as well as the resumes of these key personnel, can be found in Appendix B.

- The role of the **Laboratory Director** is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program and is responsible for overall laboratory efficiency and the financial performance of the Kelso facility. The Laboratory Director has the authority to stop work in response to quality problems. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.

- The responsibility of the **Quality Assurance Manager** (QAM) is to oversee implementation of the quality program and to coordinate overall QA activities within the laboratory. The QAM works with individual laboratory production units to establish effective quality control and assessment plans. The QAM has the authority to stop work in response to quality problems. The QAM is also responsible for maintaining this QA Manual and performing an annual review of it, updating it if necessary; reviewing, approving, and controlling SOPs and coordinating the annual review of each SOP (Section 4.2.1); maintaining QA records such as metrological records, archived logbooks, PT sample results, etc.; oversight and review of QC data; coordinating PT sample analyses and approving nonconformity and corrective action reports for any unacceptable PT sample results (Section 15.0); reviewing data (Section 12.0); maintaining the laboratory's certifications and approvals (Section 13.0); performing internal QA audits (Section 13.0); preparing QA activity reports (Section 16.0); etc. The QAM reports directly to the Laboratory Director. The QAM also interacts with the CAS Quality Assurance Director, who is responsible for the CAS laboratory-wide QA program. It is important to note that when evaluating data, the QAM does so in an objective manner and free of outside, or managerial, influence.

The Quality Assurance Director is responsible for the overall QA program at all the CAS laboratories. The QA Director is responsible for performing an annual on-site audit at each CAS laboratory and preparing a written report; maintaining a data base of information about state certifications and accreditation programs; writing laboratory-wide SOPs; maintaining a data base of CAS-approved subcontract laboratories; providing assistance to the laboratory QA staff and laboratory managers; preparing a quarterly QA activity report; etc.

- In the case of absence of the Laboratory Director or QA Manager, deputies are assigned to act in that role. Default deputies for these positions are the Client Services Manager or Organics Department Manager (for the Laboratory Director) and the QA Director or Laboratory Director (for the QA manager).

- The **Environmental Health and Safety Officer** (EH&S) is responsible for the administration of the laboratory health and safety policies. This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections. The EH&S officer is also designated as the Chemical Hygiene Officer. The EH&S Officer has a dotted-line reporting responsibility to CAS' EH&S Director.

- The **Client Services and Sample Management Office Manager** is responsible for the Client Services Department (customer services/project chemists, and Electronic Data Deliverables group) and the sample management office/bottle preparation sections. The Client Services Department provides a complete interface with clients from initial project specification to final deliverables. The sample management office handles all the activities associated with receiving, storage, and disposal of samples. The Client Services Manager has the authority to stop subcontractor work in response to quality problems.

- The **Project Chemist** is a senior-level scientist assigned to each client to act as a technical liaison between the client and the laboratory. The project chemist is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements. This entails coordinating with the CAS laboratory and administrative staff to ensure that client-specific needs are understood, and that the services CAS provides are properly executed and satisfy the requirements of the client.

- The Analytical Laboratory is divided into operational units based upon specific disciplines. Each department is responsible for establishing, maintaining and documenting a quality control program based upon the unique requirements within that department's responsibilities. Each **Department Manager and Supervisor** has the responsibility to ensure that quality control functions are carried out as planned, and to guarantee the production of high quality data. Department managers and bench-level supervisors have the responsibility to monitor the day-to-day operations to ensure that productivity and data quality objectives are met. Each department manager has the authority to stop work in response to quality problems in their area. Each analyst in the laboratory has the responsibility to carry out testing according to prescribed methods, standard operating procedures and quality control guidelines particular to the laboratory in which he/she is working.

- The **Sample Management Office** plays a key role in the laboratory QA program by maintaining documentation for all samples received by the laboratory, and by assisting in the archival of all laboratory results. The sample management office staff is also responsible for the proper disposal of samples after analysis.
- **Information Technology** (IT) staff are responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, IT systems development and implementation, education of analytical staff in the use of scientific software, Electronic Data Deliverable (EDD) generation, and data back-up, archival and integrity operations.

Table 6-1
Summary of Technical Experience and Qualifications

Personnel	Years of Experience	Project Role
Jeff Christian, B.S.	23	Laboratory Director
Lee Wolf, B.S.	16	Quality Assurance Manager
Eileen Arnold, B.A.	21	Environmental, Health and Safety Officer
Lynda Huckestein, B.S.	16	Senior Project Chemist Client Services Manager Sample Management Office Manager
Jeff Coronado, B.S.	12	Inorganics Department Manager
Todd Poyfair, B.S.	10	Gas Chromatography and Petroleum Hydrocarbons Department Manager
Jeff Grindstaff, B.S.	13	Volatiles and Semivolatiles GC/MS Department Manager
Paul Gowan, B.A.	15	Technical Information Specialist
David Edelman, Ph.D.	22	CAS Corporate Technical Director
Lawrence Jacoby, Ph.D.	29	CAS Quality Assurance Director
Gary Ward, M.S.	27	CAS Information Technology Director CAS Chief Quality Officer
Steve Vincent, B.S.	26	CAS President

7.0 INFORMATION MANAGEMENT

The generation, compilation, reporting, and archiving of electronic data is a critical component of laboratory operations. In order to generate data of known and acceptable quality, the quality assurance systems and quality control practices for electronic data systems must be complete and comprehensive and in keeping with the overall quality assurance objectives of the organization. CAS management provides the tools and resources to implement electronic data systems and establishes information technology standards and policies. Appendix C lists major automated data processing equipment.

7.1 Software Quality Assurance Plan

CAS has defined practices for assuring the quality of the computer software used throughout all laboratory operations to generate, compile, report, and store electronic data. These practices are described in the CAS Software Quality Assurance Plan (SQAP). The purpose of the SQAP is to describe the policies and practices for the procurement, configuration management, development, validation and verification, data security, maintenance, and use of computer software. The policies and practices described in the plan apply to purchased computer software as well as to internally developed computer software. A key component of configuration management plan are policies for controlling the software version that is in use in the laboratory.

7.2 IT Support

The local CAS Information Technology (IT) department is established to provide technical support for all computing systems. The IT department staff continually monitor the performance and output of operating systems. The IT department oversees routine system maintenance and data backups to ensure the integrity of all electronic data. A software inventory is maintained. Additional IT responsibilities are described in the SQAP.

In addition to the local IT department, CAS corporate IT provides support for network-wide systems. CAS-Kelso also has personnel assigned to information management duties such as development and implementation of reporting systems; data acquisition, and Electronic Data Deliverable generation.

7.3 Information Management Systems

CAS has various systems in place to address specific data management needs. The CAS Laboratory Information Management System (LIMS) is used to manage sample information and invoicing. Access is controlled by password. This is a Unix-based network system, operating in an Oracle® database environment This system is used to establish and define sample identification, analysis specifications, and provide a means of sample tracking. This system is used during sample login to generate the internal Service Request. The Service Request provides a summary of client information, sample information, required analyses, work instructions, deliverable requirements and other necessary information provided on the chain of custody. The LIMS also is the basis for valuable sample tracking mechanisms used throughout the laboratory. Laboratory analysts generate worklists from the LIMS and the barcoding system used for internal chain of custody (Section 8) interfaces with this system.

Where possible, instrument data acquired locally is immediately moved to a server (Microsoft Windows2000® local area network) dedicated to this function. This provides a reliable, easily-maintained, high-volume acquisition and storage system for electronic data files. With password entry, users may access the system from many available computer stations, improving efficiency and flexibility. Another server is dedicated to data reporting, EDD generation, and administrative functions. Access to these systems is controlled by password. A standardized EDI (electronic data interchange) format is used as a reporting platform, providing functionality and flexibility for end users. With a common standardized communication platform, the EDI provides data reporting in a variety of hardcopy and electronic deliverable formats.

7.4 Backup and Security

CAS laboratory data is either acquired directly to the centralized acquisition server or acquired locally and then transferred to the server. All data is eventually moved to the centralized data acquisition server for reporting and archiving. Differential backups are performed on all file server information once per day, Sunday through Thursday. Full backups are performed on Friday and Saturday nights. Tapes are physically stored in a locked media cabinet within a locked, temperature controlled computer room.

Access to sample information and data is on a need-to-know basis. Access is restricted to the person's areas of responsibility. Passwords are required on all systems. No direct external, non-CAS access is allowed to any of our network systems.

The external e-mail system and internet access is set up via a single gateway to discourage unauthorized entry. CAS uses a closed system for company e-mail. Files, such as electronic deliverables, are sent through the external e-mail system only via a trusted agent. The external messaging system operates through a single secure gateway. All attachments sent in and out of the gateway are subject to a virus scan. Because the Internet is not regulated, we use a limited access approach to provide a fire-wall for added security. Virus screening is performed every week on all network systems.

8.0 SAMPLE MANAGEMENT

8.1 Sampling and Sample Preservation

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples. CAS recommends that clients follow sampling guidelines described in 40 CFR 136, USEPA SW-846, and state-specific sampling guidelines, if applicable. Sampling factors that must be taken into account to insure accurate, defensible analytical results include:

- Amount of sample taken
- Type of container used
- Type of sample preservation
- Sample storage time
- Proper custodial documentation

CAS uses the sample preservation, container, and holding-time recommendations published in a number of documents. The primary documents of reference are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III for hazardous waste samples, and USEPA 600/4-79-020, 600/4-91-010 and Supplement I, 600/4-82-057, 600/R-93/100, 600/4-88-039 and Supplements I and II, and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. The complete citation for each of these references can be found in Section 18.0 of this document. The container, preservation and holding time information is summarized in Table 8-1 for soil and water. Where allowed by the project sampling and analytical protocols (such as Puget Sound Protocols) the holding time for sediment, soil, and tissue samples may be extended for a defined period when stored frozen at -20°C.

CAS routinely provides sample containers with appropriate preservatives for our clients. The containers are purchased as "precleaned" to a level 1 status, and conform to the requirements for analytical sample established by the USEPA. Certificates of analysis for the sampling containers are available to clients if requested. Reagent water used for sampling blanks (trip blanks, etc.) and chemical preservation reagents are tested by the laboratory to ensure that they are free of interferences and documented. Our sample kits typically consist of foam-lined, precleaned shipping coolers, (cleaned inside and out with appropriate cleaner, rinsed thoroughly and air-dried), specially prepared and labeled sample containers individually wrapped in protective material, (VOC vials are placed in a specially made, foam holder), chain-of-custody (COC) forms, and custody seals. An example of a sample container label and a custody seal is shown in Figure 8-1. Figure 8-2 is a copy of the chain-of-custody form routinely used at CAS. For large sample container shipments, the containers may be shipped in their original boxes. Such shipments will consist of several boxes of labeled sample containers and sufficient

materials (bubble wrap, COC forms, custody seals, shipping coolers, etc.) to allow the sampling personnel to process the sample containers and return them to CAS. The proper preservative is added to the sample containers prior to shipment, unless otherwise instructed by the client.

If any returning shipping cooler exhibits an odor or other abnormality after receipt and subsequent decontamination by laboratory personnel, a second, more vigorous decontamination process is employed. Containers exhibiting an odor or abnormality after the second decontamination process are promptly and properly discarded. CAS keeps client-specific shipping requirements on file and utilizes major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. CAS also provides courier service that makes regularly scheduled trips to the Greater Portland, Oregon Metropolitan area.

When environmental samples are shipped by CAS to other laboratories for analysis each sample bottle is wrapped in protective material and placed in a plastic bag (preferably Ziploc®) to avoid any possible cross-contamination of samples during shipping. The sample management office (SMO) follows formalized procedures for maintaining the chain of custody of the sample(s) (SOP *for Chain of Custody for Sample Transfer between Laboratories* [SOP ADM-COC]), proper packaging and shipment, specification of proper methodology, etc. Blue or gel ice is the only temperature preservative used by CAS, unless otherwise specified by the client or receiving laboratory.

8.2 Sample Receipt and Handling

Standard Operating Procedures have been established for the receiving of samples into the laboratory. These procedures ensure that samples are received and properly logged into the laboratory, and that all associated documentation, including chain of custody forms, is complete and consistent with the samples received. Complete documentation of all sample storage is maintained in order to preserve the integrity of the samples.

Samples are delivered to the CAS sample management office (SMO) and are received by a Sample Custodian. A Cooler Receipt and Preservation Check Form (CRF - See Figure 8-3 for an example) is used to assess the shipping cooler and its contents as received by the laboratory personnel. Verification of sample integrity by the Sample Custodian includes the following activities:

- Assessment of custody seal presence/absence, location and signature;
- Temperature of sample containers upon receipt;
- Chain of custody documents properly used (entries in ink, signature present, etc.);
- Sample containers checked for integrity (broken, leaking, etc.);
- Sample is clearly marked and dated (bottle labels complete with required information);
- Appropriate containers (size, type) are received for the requested analyses;
- The minimum amount of sample material is provided for the analysis.

- Sample container labels and/or tags agree with chain of custody entries (identification, required analyses, etc.);
- Assessment of proper sample preservation (if inadequate, corrective action is employed); and
- VOC containers are inspected for the presence/absence of bubbles. (No assessment of proper preservation is performed for VOC containers by SMO personnel).

Samples are logged into a Laboratory Information Management System (LIMS). Any anomalies or discrepancies observed during the initial assessment are recorded on the CRF and chain of custody documents. All potential problems with a sample shipment are addressed by contacting the client and discussing the pertinent issues. When a satisfactory resolution has been reached by the Project Chemist and client, the log-in process may continue and analysis may begin. During the log-in process, each sample is given a unique laboratory code and a service request form is generated. The LIMS generates a Service Request that contains client information, sample descriptions, sample matrix information, required analyses, sample collection dates, analysis due dates and other pertinent information. The service request is reviewed by the appropriate Project Chemist for accuracy, completeness, consistency of requested analyses and for client project objectives.

Samples are kept refrigerated until they undergo analysis, unless otherwise specified. CAS stores samples in one of twelve various refrigerators or freezers, depending on the type of analysis and the matrix of the sample. CAS has four walk-in refrigerators which house the majority of sample containers received at the laboratory. In addition to the four walk-in refrigerators, there are three additional refrigerators, including dedicated refrigerated storage of VOC samples. These refrigerators are segregated by matrix type (soil or water) and method of analysis. The dedicated storage areas for VOC samples are monitored using storage blanks, as described in the *SOP for VOA Storage Blanks (VOC-BLAN)*. CAS also has four sub-zero freezers capable of storing samples at -20° C; these are primarily used for tissue and sediment samples requiring specialized storage conditions. One additional freezer provides additional frozen storage capacity for miscellaneous and archived samples. The temperature of each sample storage unit used at CAS is monitored daily and the data recorded in a bound logbook. Continuous-graph temperature recorders have also been placed in the walk-in refrigerators to provide a permanent record of the storage conditions to which samples are exposed.

CAS adheres to the method-prescribed or project-specified holding times for all analyses. In order to comply with holding time requirements, the sampling date and time are entered into the LIMS system at the time of sample receipt and login. Each analyst then monitors holding times by obtaining analysis-specific worklists (typically daily, or more often) from the LIMS. These worklists provide holding time information on all samples for the analysis, calculated from the sample date and the holding time requirement. In order to report adherence to holding time requirements, the date analyzed is printed or written on the analytical raw data. For analyses with holding time prescribed in hours, the time analyzed is also recorded.

Upon completion of all analyses and submittal of the final report, aqueous samples and sample extracts are retained at ambient temperature on holding shelves for 30 days (unless other arrangements have been made in advance), and soil samples are retained at ambient temperature on holding shelves for 60 days. Upon expiration of these time limits, the samples are either returned to the client or disposed of according to approved disposal practices. All samples are characterized according to hazardous/non-hazardous waste criteria and are segregated accordingly. All hazardous waste samples are disposed of according to formal procedures outlined in the *CAS Environmental Health and Safety Manual.* All waste produced at the laboratory, including the laboratory's own various hazardous waste streams, is treated in accordance with applicable local and Federal laws. Documentation is maintained for each sample from initial receipt through final disposal. This ensures that an accurate history of the sample from "cradle to grave" is generated.

8.3 Sample Custody

Sample custody transfer at the time of sample receipt is documented using chain-of-custody (COC) forms accompanying the samples. During sample receipt, it is also noted if custody seals were present. This is described in the *SOP for Sample Receiving (SMO-GEN).* Figure 8-2 is a copy of the chain-of-custody form routinely used at CAS.

Facility security and access is important in maintaining the integrity of samples received at CAS/Kelso. Access to the laboratory facility is limited by use of locked exterior doors with a coded entry, except for the reception area and sample receiving doors, which are manned during business hours and locked at all other times. In addition, the sample storage area within the laboratory is a controlled access area with locked doors with a coded entry. The CAS facility is equipped with an alarm system and CAS employs a private security firm to provide night-time and weekend security.

A barcoding system is used to document internal sample custody. Each person removing or returning samples from/to sample storage while performing analysis is required to document this custody transfer. The system uniquely identifies the sample container and provides an electronic record of the custody of each sample. For sample extracts and digestates the analyst documents custody of the sample extract or digestate by signing on the benchsheet, or custody record, that they have accepted custody. The procedures are described in the *SOP for Sample Tracking and Internal Chain of Custody (SMO-SCOC).*

8.4 Project Setup

The analytical method(s) to be used for sample analysis are chosen based on the COC information and project requirements. Unless specified otherwise, the most recent version of reference methods are used. LIMS codes are chosen to identify the analysis method used for analysis. The Project Chemist ensures that the correct methods are selected for analysis, deliverable requirements are identified, and due dates are specified on the LIMS generated Service Request. To communicate and specify project-specific requirements, a Tier V form (Figure 8-4) is used and accompanies the service request form.

Table 8-1
Sample Preservation and Holding Times

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Bacterial Tests				
Coliform, Fecal and Total	W	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Fecal Streptococci	W	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Inorganic Tests				
Acidity	W	P,G	Cool, 4°C	14 days
Alkalinity	W	P,G	Cool, 4°C	14 days
Ammonia	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Biochemical Oxygen Demand (BOD)	W	P,G	Cool, 4°C	48 hours
Bromide	W	P,G	None Required	28 days
Chemical Oxygen Demand (COD)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Chloride	W	P,G	None Required	28 days
Chlorine, Total Residual	W	P,G	None Required	24 hours
Color	W	P,G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	W	P,G	Cool, 4°C, NaOH to pH>12, plus 0.6 g Ascorbic Acid	14 days
Cyanide, Weak Acid Dissociable	W	P,G	Cool, 4°C, NaOH to pH >12	14 days
Fluoride	W	P,G	None Required	28 days
Hardness	W	P,G	HNO_3 to pH<2	6 months
Hydrogen Ion (pH)	W	P,G	None Required	24 hours
Kjeldahl and Organic Nitrogen	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrate	W	P,G	Cool, 4°C	48 hours
Nitrate	Chlorinated DW	P,G	Cool, 4°C	14 days
Nitrate-Nitrite	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrite	W	P,G	Cool, 4°C	48 hours
Orthophosphate	W	P,G	Filter Immediately, Cool, 4°C	48 hours
Oxygen, Dissolved (Probe)	W	G, Bottle and Top	None Required	Analyze immediately
Oxygen, Dissolved (Winkler)	W	G, Bottle and Top	Fix on Site and Store in Dark	8 hours
Phenolics, Total	W	G Only	Cool, 4°C, H_2SO_4 to pH<2	28 days
Phosphorus, Elemental	W	G Only	Cool, 4°C	48 hours
Phosphorus, Total	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Residue, Total	W	P,G	Cool, 4°C	7 days
Residue, Filterable (TDS)	W	P,G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	W	P,G	Cool, 4°C	7 days
Residue, Settleable	W	P,G	Cool, 4°C	48 hours

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Residue, Volatile	W	P,G	Cool, 4°C	7 days
Silica	W	P Only	Cool, 4°C	28 days
Specific Conductance	W	P,G	Cool, 4°C	28 days
Sulfate	W	P,G	Cool, 4°C	28 days
Sulfide	W	P,G	Cool, 4°C, Add Zinc Acetate plus Sodium Hydroxide to pH>9	7 days
Sulfite	W	P,G	None Required	24 hours
Surfactants (MBAS)	W	P,G	Cool, 4°C	48 hours
Tannin and Lignin	W	P,G	Cool, 4°C	28 days
Temperature	W	P,G	None Required	Analyze immediately
Turbidity	W	P,G	Cool, 4°C	48 hours
Metals				
Chromium VI	W	P,G	Cool, 4°C	24 hours
Mercury	W	P,G	HNO_3 to pH<2	28 days
	S	P,G	Cool, 4°C	28 days
Metals, except Chromium VI and Mercury	W	P,G	HNO_3 to pH<2	6 months
	S	G, Teflon-Lined Cap	Cool, 4°C	6 months
Organic Tests				
Oil and Grease/Hexane Extractable Material (EPA 1664)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Carbon, Total (TOC)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Halogens, Total (TOX)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2, No headspace	28 days
Organic Halogens, Adsorbable (AOX)	W	G, Teflon-Lined Cap	Cool, 4°C, HNO_3 to pH<2	6 months
Petroleum Hydrocarbons, Total	W	G, Teflon-Lined Cap	Cool, 4°C, HCl or H_2SO_4 to pH<2	7 days until extraction; 40 days after extraction
	S	G, Teflon-Lined Cap	Cool, 4°C	14 days until extraction; 40 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Volatile Organics				
Purgeable Organics	DW	G, Teflon-Lined Septum Cap,	Ascorbic Acid, HCl to pH<2, Cool, 4°C, No Headspace	14 days
Petroleum Hydrocarbons, Volatile (Gasoline-Range Organics)	W	G, Teflon-Lined Septum Cap	Cool, 4°C, HCl to pH<2 No Headspace	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C Minimize Headspace	14 days
Purgeable Halocarbons	W	G, Teflon-Lined Septum Cap, No Headspace	**No Residual Chlorine Present:** HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present:** 10% $Na_2S_2O_3$, HCl to pH<2, Cool, 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE)	W	G, Teflon-Lined Septum Cap, No Headspace	**No Residual Chlorine Present:** HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present:** 10% $Na_2S_2O_3$, HCl to pH<2, Cool 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.
Acrolein, Acrylonitrile, Acetonitrile	W	G, Teflon-Lined Septum Cap	Adjust pH to 4-5, Cool, 4°C, No Headspace	14 days

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
EDB and DBCP	W,S	G, Teflon-Lined Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	28 days
Semivolatile Organics				
Petroleum Hydrocarbons, Extractable (Diesel-Range Organics)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Alcohols and Glycols	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Phenols	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Phthalate Esters	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Nitrosamines	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Organochlorine Pesticides and PCBs	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Nitroaromatics and Cyclic Ketones	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Polynuclear Aromatic Hydrocarbons	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Haloethers	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Hydrocarbons	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Organophosphorus Pesticides	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Nitrogen- and Phosphorus-Containing Pesticides	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Herbicides	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Phenolics	W	G, Teflon-Lined Cap	H_2SO_4 to pH<2, Cool, 4°C[g]	30 days until extraction; 30 days after extraction
Resin and Fatty Acids	W	G, Teflon-Lined Cap	NaOH to pH ≥10, Cool, 4°C[g]	30 days until extraction; 30 days after extraction
Toxicity Characteristic Leaching Procedure (TCLP)				
Mercury	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	28 days until extraction; 28 days after extraction
Metals, except Mercury	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	180 days until extraction; 180 days after extraction
Volatile Organics	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C Minimize Headspace TCLP extract: Cool, 4°C, HCl to pH<2, No Headspace	14 days until extraction; 14 days after extraction
Semivolatile Organics	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C, Store in Dark[g] TCLP extract: Cool, 4°C, Store in Dark[g]	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Organochlorine Pesticides	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Chlorinated Herbicides	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Contract Laboratory Program (CLP)				
Cyanide, Total and Amenable to Chlorination	W	P,G	Cool, 4°C, NaOH to pH 12, plus 0.6 g Ascorbic Acid	12 days[h]
	S	P,G	Cool, 4°C	12 days[h]
Mercury	W	P,G	HNO_3 to pH<2	26 days[h]
	S	P,G	Cool, 4°C	26 days[h]
Metals, except Mercury	W	P,G	HNO3 to pH<2	6 months[h]
	S	P,G	Cool, 4°C	6 months[h]
Volatile Organics	W	G, Teflon-Lined Cap	HCl to pH <2, Cool, 4°C, Minimize Headspace	10 days[h]
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	10 days[h]
Semivolatile Organics	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	5 days until extraction;[h,i] 40 days after extraction
Organochlorine Pesticides and PCBs	W,S	G, Teflon-Lined Cap	Cool, 4°C	5 days until extraction;[h,i] 40 days after extraction

a See Section 18.0 for sources of holding time information.
b DW = Drinking Water, W = Water; S = Soil or Sediment; HW = Hazardous Waste
c P = Polyethylene; G = Glass
d For chlorinated water samples
e The recommended maximum holding time is variable, and is dependent upon the geographical proximity of sample source to the laboratory.
f Fourteen days until extraction for soil, sediment, and sludge samples.
g If the water sample contains residual chlorine, 10% sodium thiosulfate is used to dechlorinate.
h Number of days following sample receipt at the laboratory.
i Ten days until extraction for soil, sediment, and sludge samples.

Figure 8-1
Sample Container Label and Custody Seal

Project Name: _____

Sample Name: _____

Date_____ Time_____ Sampler _____

Analysis _____

_____

_____

Preservative: _____

Comments:

Lab Label #2

Custody Seal

Date_____ Project_____

Signature _____Container#_____ of_____

Figure 8-2
Chain of Custody Form

Columbia Analytical Services, Inc.

CHAIN OF CUSTODY

SR#: _____

1317 South 13th Ave. • Kelso, WA 98626 • (360) 577-7222 • (800) 695-7222x07 • FAX (360) 636-1068

PAGE _____ OF _____ COC # _____

PROJECT NAME
PROJECT NUMBER
PROJECT MANAGER
COMPANY/ADDRESS
PHONE # FAX #
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 Gas☐	8021☐ BTEX☐ (see below) Diesel☐ Oil☐	☐ Fuel Fingerprint (FIO) ☐ NW-HCID Screen	Oil & Grease/TRPH 1664 HEM☐	1664 SGT☐	PCB's Aroclors☐	Congeners☐	Pesticides/Herbicides 608☐ 8081A☐	Chlorophenolics - 8151M Tri☐ Tetra☐	8141A☐ 8151A☐ PCP☐	PAHS 8310☐ SIM☐	Metals, Total or Dissolved (See list below)	Cyanide☐	Hex-Chrom☐	pH, Cond, Cl, SO_4, PO_4, 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☐					REMARKS

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)
___ IV. CLP Deliverable Report
___ V. EDD

INVOICE INFORMATION

P.O. # _____
Bill To: _____

TURNAROUND REQUIREMENTS

____ 24 hr. ____48 hr.
____5 Day
____ 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 NORHTWEST OTHER: ____ (CIRCLE ONE)

SPECIAL INSTRUCTIONS/COMMENTS:

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

Figure 8-3
Cooler Receipt and Preservation Check Form

Project/Client_____ Work Order K22_____

Cooler received on_____ and opened on _____ by _____

1.	Were custody seals on outside of cooler? If yes, how many and where?_____	Y	N
2.	Were seals intact and signature & date correct?	Y	N
3.	COC # ____ ____ ____ ____ ____		
	Temperature of cooler(s) upon receipt: ____ ____ ____ ____ ____		
	Temperature Blank: ____ ____ ____ ____ ____		
4.	Were custody papers properly filled out (ink, signed, etc.)?	Y	N
5.	Type of packing material present _____		
6.	Did all bottles arrive in good condition (unbroken)?	Y	N
7.	Were all bottle labels complete (*i.e.* analysis, preservation, etc.)?	Y	N
8.	Did all bottle labels and tags agree with custody papers?	Y	N
9.	Were the correct types of bottles used for the tests indicated?	Y	N
10.	Were all of the preserved bottles received at the lab with the appropriate pH?	Y	N
11.	Were VOA vials checked for absence of air bubbles, and if present, noted below?	Y	N
12.	Did the bottles originate from CAS/K or a branch laboratory?	Y	N
13.	Are CWA Microbiology samples received with > ½ the 24 hr. hold time remaining from collection?	Y	N
14.	Was CL2/Residual negative?	Y	N

Explain any discrepancies: _____

_____

_____

RESOLUTION: _____

_____

Samples that required preservation or received out of temperature:

Sample ID	Reagent	Volume	Lot Number	Bottle Type	Rec'd out of Temperature	Initials

CRFREV.DOC12/24/01

Figure 8-4
Tier V Form

Columbia Analytical Services Inc.

Client :

Project Name :

Project Number :

Project Description :

Project Chemist :

Service Request :

SMO LimsTemplate ID :

QAPP/SOW Information :

Reporting

Tier Level :

In result field use :

Flagging Requirements :

Other Requirements :

Report to :

EDD :

For multiple runs use :

Sample Considerations

Sample Limitations :

Sample Prep/Analysis :

Non-Standard Holdtimes :

Historical Data :

Comments :

9.0 QUALITY CONTROL OBJECTIVES (PRECISION, ACCURACY, AND MDLS)

A primary focus of Columbia Analytical Services Quality Assurance (QA) Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis of field samples, acceptable method performance is established by performing demonstration of capability analyses and performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. CAS has established Quality Control (QC) objectives for precision and accuracy that are used to determine the acceptability of the data that is generated in its laboratories. These QC limits are either specified in the methodology or are statistically derived based on the laboratory's actual historical data obtained from the various QC measurements for each analytical method. The Quality Control objectives are defined below and the numeric values are shown in Appendix D.

9.1 Demonstration of Capability

Where required by mandatory test method, regulation, or accreditation protocols, a demonstration of capability (DOC) is made prior to using any test method. This demonstration is made following regulatory, accreditation, or method specified procedures. In general, this demonstration does not test the performance of the method in real world samples, but in the applicable clean matrix free of target analytes and interferences.

A quality control reference material or quality control sample is obtained. The analyte(s) is(are) diluted in a volume of clean matrix (for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples). Where specified, the method-required concentration levels are used. Four aliquots are prepared and analyzed according to the test procedure. The mean recovery and standard deviations are calculated and compared to the corresponding acceptance criteria for precision and accuracy in the test method or laboratory-generated acceptance criteria (if there are not established mandatory criteria). All parameters must meet the acceptance criteria. Where spike levels are not specified, actual Laboratory Control Sample results or MDL study results may be used to meet this requirement, provided acceptance criteria is met.

9.2 Accuracy

Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of standard reference materials, standard solutions and laboratory-fortified blanks.

In addition, laboratory-fortified (i.e. matrix-spiked) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value. If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis (e.g., extraction efficiencies) or caused by an artifact of the measurement system (e.g., contamination). CAS utilizes several quality control measures to eliminate analytical bias, including systematic analysis of method blanks, laboratory control samples and independent calibration verification standards. Because bias can be positive or negative, and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement

9.3 Precision

Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: repeatability - the random error associated with measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions, and reproducibility - the random error associated with measurements made by different test operators, in different laboratories, using the same method but different equipment to analyze identical samples of test material.

At CAS, our "within-batch" precision is measured through the use of replicate sample or QC analyses and is expressed as the relative percent difference (RPD) between the replicate measurements. The "batch-to-batch" precision is calculated from the variance observed in results from analysis of standard solutions or laboratory control samples from multiple analytical batches.

9.4 Control Limits

The acceptance limits for accuracy and precision originate from two different sources: For analyses having enough QC data, control limits are calculated at the 99% confidence limits. New control limits are generated using the data generated in the previous year or when a representative population of new values is acquired. After review of the data by the Quality Assurance Manager, the new acceptance criteria replace the previous criteria and method conformity is assessed using the new values. For analyses not having enough QC data, or where the method is prescriptive, control limits are taken from the method on which the procedure is based. If the method does not have control limits stated in it, then control limits are assigned method-default or reasonable values. These control limits are updated when new statistical limits are generated for the appropriate surrogate, laboratory control sample, and matrix spike compounds (typically once a year) or when method prescribed limits change.

The acceptance limits for accuracy and precision for selected analytes are shown in the table in Appendix D. For accuracy limits, the values listed are for laboratory control samples unless otherwise indicated. For inorganics, the precision limit values listed are for laboratory duplicates. For organics, the precision limit values listed are for duplicate laboratory control samples or duplicate matrix spike analyses.

9.5 Representativeness

Representativeness is the degree to which the field sample represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed gives results identical to analysis of the entire field sample. CAS has sample handling procedures and protocols to ensure that the sample used for analysis is representative of the entire sample. These include the *SOP for Sample Preparation, Compositing, and Subsampling*, the *SOP for Solid Sample Preparation*, and the *SOP for Tissue Sample Preparation.* Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample.

9.6 Completeness

Completeness is a measure of the amount of valid data that is obtained, compared to the amount that is expected. For the purposes of this plan, completeness is calculated by dividing the number of samples having valid data by the total number of samples in the project, expressed as a percentage. The CAS objective for completeness is 100%.

9.7 Comparability

Comparability expresses the confidence with which one data set can be compared to another. To ensure comparability, standard operating procedures are used for the preservation, handling, and analysis of all samples. Data is reported in units specified by the customer.

9.8 Method Detection Limits

Method Detection Limits (MDL) for analytical methods routinely performed at CAS/Kelso are determined annually. The MDLs are determined by following the *Standard Operating Procedure for the Determination of Method Detection Limits* (SOP No. ADM - MDL) which is based on the procedure outlined in 40 CFR Part 136, Appendix B. The Method Reporting Limits (MRLs) used at CAS are the routinely reported lower limits of quantitation which take into account day-to-day fluctuations in instrument sensitivity as well as other factors. These MRLs are the levels to which CAS routinely reports results in order to minimize false positive or false negative results. The MRL is normally two to ten times the method detection limit.

10.0 QUALITY CONTROL PROCEDURES

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below. In addition, a number of other quality control processes which may impact analytical results are also described below.

10.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks.

CAS maintains a database of SOPs for use in both technical and administrative functions. SOPs are written following the format and content requirements described in the *SOP for Preparation of Standard Operating Procedures* (SOP No. ADM-SOP). Each SOP has been reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the Quality Assurance Manager). All SOPs undergo a documented annual review to make sure current practices are described. A comprehensive list of current SOPs is maintained by the QA Manager. The document control process ensures that only the most currently prepared version of an SOP is being used for guidance and instruction. The QA Manual, QAPPs, SOPs, standards preparation logbooks, maintenance logbooks, et al., are controlled documents. The procedures for document control are described in the *SOP for Document Control* (SOP No. ADM-DOC_CTRL). In addition to SOPs, each laboratory department maintains a current file, accessible to all laboratory staff, of the promulgated methodology used to perform analyses. Laboratory notebook entries have been standardized following the guidelines in the *Making Entries into Logbooks and onto Benchsheets* SOP (SOP No. ADM-DATANTRY). The entries made into laboratory notebooks are reviewed and approved by the appropriate supervisor at a regular interval (e.g., weekly, monthly, etc.).

10.2 Deviation from Standard Operating Procedures

When a customer requests a modification to an SOP (such as a change in reporting limit, addition or deletion of target analyte(s), etc.), the project chemist handling that project must discuss the proposed deviation with the department manager in charge of the analysis and obtain their approval to accept the project. The project chemist is responsible for documenting the approved or allowed deviation from the standard operating procedure by placing a detailed description of the deviation attached to the quotation or in the project file and also providing an appropriate comment on the service request when the samples are received.

For circumstances when a deviation or departure from company policies or procedures involving any non-technical function is found necessary, approval must be obtained from the appropriate supervisor, manager, the laboratory director, or other level of authority.

Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the possible need for a change in policy will be addressed by the laboratory director.

10.3 Modified Procedures

CAS strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating procedures are available to analysts and are also available to our clients for review, especially those for "Modified" methods. Client approval is obtained for the use of "Modified" methods prior to the performance of the analysis.

10.4 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that CAS has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples, are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner.

The minimum requirements of an analytical batch are:

1) The number of (field) samples in a batch is not to exceed 20.

2) All (field) samples in a batch are of the same matrix.

3) The QC samples to be processed with the (field) samples include:

   a) Method Blank (a.k.a. Laboratory Reagent Blank)

      Function: Determination of laboratory contamination.

   b) Laboratory Control Sample (a.k.a. Laboratory Fortified Blank)

      Function: Assessment of method performance

   c) Matrix Spiked (field) Sample (a.k.a. Laboratory Fortified Sample Matrix)

      Function: Assessment of matrix problems

      NOTE: A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked.

d) Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample (a.k.a. Laboratory Duplicate)

Function: Assessment of batch precision

NOTE: A sample identified as a field blank, an equipment blank, or a trip blank is not to be duplicated.

4) A single lot of reagents is used to process the batch of samples.

5) Each operation within the analysis is performed by a single analyst, technician, chemist, or by a team of analysts/technicians/chemists.

6) The time frame is not to exceed a 24 hour period. "Open batches" extending over more than one 24 hour period are not allowed.

7) (Field) samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic constituents, it begins when the samples are extracted.

8) The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch (digestion, extraction, etc.), the QC samples in the batch do not require analysis each time a field sample within the preparation batch is analyzed (multiple instrument sequences to analyze all field samples in the batch need not include re-analyses of the QC samples).

9) The batch is is to be assigned a unique identification number that can be used to correlate the QC samples with the field samples.

10) Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.

11) Specific project, program, or method SOP requirements may be exceptions. If project, program, or method SOP requirements are more stringent than these laboratory minimum requirements, then the project, program, or method SOP requirements will take precedence. However, if the project, program, or method SOP requirements are less stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

10.5 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is either analyte-free water or analyte-free soil (when available), subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute may be used instead. The method blank is analyzed to demonstrate that the analytical system itself is not contaminated with the analyte(s) being measured.

The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, below half of the MRL for the analyte(s) being tested. Otherwise, corrective action must be taken. A method blank is included with the analysis of every sample preparation batch, every 20 samples, or as stated in the method, whichever is more frequent.

10.6 Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve.

10.7 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of either analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed. The frequency of CCB analysis is either once every ten samples or as indicated in the method, whichever is greater.

10.8 Calibration Standards

Calibration standards are solutions of known concentration prepared from primary standard solutions which are, in turn, prepared from stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

10.9 Initial (or Independent) Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are standards that are analyzed *after* calibration with newly prepared standard(s) but *prior to* sample analysis, in order to verify the validity of the standards used in the calibration. Once it is determined that there is no systematic error in preparation of the calibration standard(s), they are considered valid standards and may be used for subsequent calibrations (as expiration dates and methods allow). The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards. ICVs are also analyzed in accordance with method-specific requirements.

10.10 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

10.11 Internal Standards

Internal standards consist of known amounts of specific compounds that are added to each sample following sample preparation or extraction. Internal standards are generally used for GC/MS and ICP-MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by certain matrix effects. The integrated area of the internal standard compared to the continuing calibration check standard should vary by no more than the limits specified in each method.

10.12 Surrogates

Surrogates are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate, and the recovery is a measurement of the overall method performance. The acceptance criteria for these various analytes are listed in Appendix D, along with other data quality capabilities.

10.13 Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix)

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) has been added. The samples are then prepared and analyzed in the same analytical batch, and in exactly the same manner as are routine samples. The stock solutions used for spiking the sample(s) are purchased and prepared independently of calibration standards. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

$$Recovery\ (\%) = (S - A) \times 100 \div T$$

Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

For the appropriate methods, matrix spiked samples are prepared and analyzed at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples.

10.14 Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed. The relative percent difference between duplicate analyses or between an MS and DMS is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

Relative Percent Difference (RPD) = (S1 - S2) x 100 ÷ S_{ave}

Where S1 and S2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and

S_{ave} = The average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, either duplicates (and/or matrix spikes) or MS/DMS analyses are performed at a minimum frequency of one set per 20 samples.

10.15 Laboratory Control Samples (a.k.a. Laboratory Fortified Blanks or Quality Control Samples)

The laboratory control sample (LCS) is an aliquot of analyte-free water or analyte-free soil (or anhydrous sodium sulfate or equivalent) to which known amounts of the method analyte(s) is(are) added. A standard reference material (SRM) of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. The LCS sample is prepared and analyzed in the same analytical batch, and in exactly the same manner, as the other routine samples. Stock solutions used for LCSs are purchased or prepared independently of calibration standards. The percent recovery (% REC.) of the target analytes in the LCS assists in determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy. Acceptance criteria for LCS analyses are obtained through the use of control charts. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent.

If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

10.16 Interference Check Samples

An interference check sample (ICS) is a solution containing both interfering and analyte elements of known concentration that can be analyzed to verify background and interelement correction factors in metals analyses. The ICS is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. The ICS is spiked with the elements of interest at concentrations of approximately ten times the instrument detection limits. The ICS is analyzed at the beginning and end of an analytical run or every eight hours, whichever is more frequent, and the results must be within ± 20% of the true values.

10.17 Post Digestion Spikes

Post digestion spikes are samples prepared for metals analyses that have an analyte spike added to determine if matrix effects may be a factor in the results. The spike addition should produce a method-specified minimum concentration above the instrument detection limit. A post digestion spike is analyzed with each batch of samples and recovery criteria are specified for each method.

10.18 Source and Preparation of Standard Reference Materials

All analytical measurements generated at CAS are performed using materials and/or processes that are traceable to a Standard Reference Material (SRM). Metrology equipment (analytical balances, thermometers, etc.) is calibrated using SRMs traceable to the National Institute of Standards and Technology (NIST). These primary SRMs are themselves recertified on an annual basis. All sampling containers provided to the client by the laboratory are purchased as precleaned (Level 1) containers, with certificates of analysis available for each bottle type. This information is provided to the client when requested.

Consumable SRMs routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally-recognized, reputable vendors. All vendors have fulfilled the requirements for ISO 9001 certification and/or are accredited by A_2LA. CAS relies on a primary vendor for the majority of its analytical supplies. In addition, consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Supelco, Ultra Scientific, AccuStandard, Chem Services, Inc., Aldrich Chemical Co., Baker, Spex, E. M. Science, etc. are examples of the vendors used by CAS. All reference materials that are received at CAS are recorded by the technical staff in the appropriate notebook(s) and are stored under conditions that provide maximum protection against deterioration and contamination. The notebook entry includes such information as an assigned logbook identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. In addition, the date that the standard is received in the laboratory is marked on the container. When the SRM container is used for the first time, the date of usage and the initials of the applicable technician are also recorded on the container.

Stock solutions and/or calibration standard solutions are prepared fresh as often as necessary according to their stability. After preparation, all standard solutions are properly labeled as to analyte concentration, solvent, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook(s) following the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP No. ADM-DATANTRY). Prior to introduction into the analytical system/process, all reference materials are verified with a second, independent source of the material (see section 10.9 above). Once the reference material has been verified to be accurate, it may then be used for instrument calibration and subsequent quantitative purposes. In addition, the independent source of reference material is also used to check the calibration standards for signs of deterioration.

10.19 Control Charting

The generation of control charts is routinely performed at CAS. Surrogate, Matrix Spike and LCS recoveries are all monitored and charted. In addition, the laboratory also monitors the Relative Percent Difference (RPD) measurement of precision. Control charts are available to each individual laboratory unit to monitor the data generated in its facility using control charts which have been programmed to identify various trends in the analytical results. If trends in the data are perceived, various means of corrective action may then be employed in order to prevent future problems with the analytical system(s). Finally, data quality reports using control charts are generated for specific clients and projects pursuant to contract requirements. The control charting procedure is described in the SOP for *Control Charting Quality Control Data* (ADM-CHRT).

10.20 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at CAS undergoes a rigorous cleansing procedure prior to every usage. A number of SOPs have been generated that outline the various procedures used at CAS; each is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

11.0 CALIBRATION PROCEDURES AND FREQUENCY

All equipment and instruments used at CAS are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below. Records are maintained to provide traceability of reference materials.

Any item of the equipment which has been subjected to overloading or mishandling, or has been shown by verification or otherwise to be defective; is taken out of service until it has been repaired. The equipment is placed back in service only after verifying by calibration that the equipment performs satisfactorily. An evaluation of the effect of this defect on previous calibrations or tests is made and documented appropriately.

Calibration verification is performed according to in the applicable analytical methodology. Calibration verification procedures and criteria are listed in laboratory Standard Operating Procedures. Documentation of calibration verification is maintained in appropriate reference files. For NELAP accredited methods, the concentration of calibration verification standards are varied within the calibration range periodically.

11.1 Temperature Control Devices

Temperatures are monitored and recorded for all of the temperature-regulating support equipment such as sample refrigerators, freezers, and standards refrigerators. Bound record books are kept which contain daily recorded temperatures, identification and location of equipment, acceptance criteria and the initials of the technician who performed the checks. The procedure for performing these measurements is provided in the *SOP for Support Equipment Monitoring and Calibration (SOP ADM-SEMC).* The SOP also includes the use of acceptance criteria and correction factors.

Where the operating temperature is specified as a test condition (such as ovens, incubators, evaporators) the temperature is recorded on the raw data. All thermometers are identified according to serial number, and the calibration of these thermometers is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. The NIST thermometer is recertified by a professional metrology organization on an annual basis.

11.2 Analytical Balances

Analytical balances are serviced on a semi-annual basis by a professional metrology organization. New certificates of calibration for each balance are issued to the laboratory on a semi-annual basis. The calibration of each analytical balance is checked daily with three class S or S-1 weights, which assess the accuracy of the balance at low, mid-level and high levels within the working range. The weights are recertified using NIST traceable standards by a professional metrology organization on an annual basis.

As needed, the balances are recalibrated using the manufacturers recommended operating procedures. Bound record books are kept which contain the recorded measurements, identification and location of equipment, acceptance criteria and the initials of the technician who performed the checks. The procedure for performing these measurements and use of acceptance criteria is described in the SOP ADM-SEMC.

11.3 Water Purification System

The water purification system used at CAS is designed to produce deionized water of 18 megohms resistivity or better, meeting specifications for Type I water, as described in *Standard Methods for the Examination of Water and Wastewater (SM1080)*. The system is monitored continuously with an on-line meter, which is recorded daily in a bound record book. Deionizers are rotated and replaced when the first unit in the series produces water of 0.5 megohms, which is monitored by a light on the unit. The status of the deionizers is also checked (resistivity reading and light status) and recorded daily in a bound record book. Activated carbon filters are also in series with the demineralizers to produce "organic-free" water. Finally, the water is checked at a point downstream of the original source - typically a spigot in one of the laboratory operating units. This information is also recorded on a weekly basis.

11.4 Inductively Coupled Plasma-Atomic Emission Spectrograph (ICP-AES)

Each emission line on the ICP is calibrated daily against a blank and against standards. Analyses of calibration standards, initial and continuing calibration verification standards, and inter-element interference check samples are carried out as specified in the EPA CLP Statement of Work for Inorganic Analysis, SOW No ILM04.0.

11.5 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)

Each element of interest is calibrated for using a blank and a single standard. Prior to calibration, a short-term stability check is performed on the system. Following calibration, an independent check standard is analyzed, and a continuing calibration verification standard (CCV) is analyzed with every ten samples.

11.6 Atomic Absorption Spectrophotometers (AAS)

These instruments are calibrated daily using a minimum of four standards and a blank. Calibration is validated using reference standards, and is verified at a minimum frequency of once every ten samples. Initial calibration points cannot be "dropped" from the resulting calibration curve.

11.7 GC/MS Systems

All GC/MS instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise) using procedures outlined in Standard Operating Procedures and/or appropriate USEPA method citations. All reference materials used for this function are vendor-certified standards. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method. Compounds selected as system performance check compounds (SPCCs) must show a method-specified response factor in order for the calibration to be considered valid. Calibration check compounds (CCCs) must also meet method specifications for percent difference from the multipoint calibration. For isotope dilution procedures, the internal standard response(s) and labeled compound recovery must meet method criteria. Method-specific instrument tuning is regularly checked using bromofluorobenzene (BFB) for volatile organic chemical (VOC) analysis, or decafluorotriphenylphosphine (DFTPP) for semi-volatile analysis. Mass spectral peaks for the tuning compounds must conform both in mass numbers and in relative intensity criteria before analyses can proceed. Calibration policies for organics chormatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

11.8 Gas Chromatographs and High Performance Liquid Chromatographs

Calibration and standardization follow SOP guidelines and/or appropriate USEPA method citations. All GC and HPLC instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise). The lowest standard is equivalent to the method reporting limit; additional standards define the working range of the GC or LC detector. Results are used to establish response factors (or calibration curves) and retention-time windows for each analyte. Calibration is verified at a minimum frequency of once every ten samples, unless otherwise specified by the reference method. *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

11.9 UV-Visible Spectrophotometer (manual colorimetric analyses)

Routine calibrations for colorimetric and turbidimetric analyses involve generating a 5-point calibration curve including a blank. Initial calibration points cannot be "dropped" from the resulting calibration curve. Correlation coefficients must meet method or SOP specifications before analysis can proceed.

Independent calibration verification standards (ICVs) are analyzed with each batch of samples. Continuing calibration is verified at a minimum frequency of once every ten samples. Typical UV-Visible spectrophotometric methods at CAS include total phenolics, phosphates, surfactants and tannin-lignin.

11.10 Flow Injection Analyzer (automated colorimetric analysis)

A minimum of three standards and a blank are used to calibrate the instrument for cyanide analysis. A blank and five (or six) standards are used to calibrate the instrument for all other automated chemistries. Initial calibration points cannot be "dropped" from the resulting calibration curve. Standard CAS acceptance limits are used to evaluate the calibration curve prior to sample analysis.

11.11 Ion Chromatographs

Calibration of the ion chromatograph (IC) involves generating a 5-point calibration curve. Initial calibration points cannot be "dropped" from the resulting calibration curve. A correlation coefficient of ≥ 0.995 for the curve is required before analysis can proceed. Quality Control (QC) samples that are routinely analyzed include blanks and laboratory control samples. The target analytes typically determined by the IC include nitrate, nitrite, chloride, fluoride, sulfate and bromide. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method.

11.12 Turbidimeter

Calibration of the turbidimeter requires analysis of three Nephelometric Turbidity Unit (NTU) formazin standards. Quality Control samples that are routinely analyzed include blanks, Analytical Products Group® QC samples (or equivalent) and duplicates.

11.13 Ion-selective electrode

Two standards are used to calibrate the electrodes before analysis. The slope of the curve must be within acceptance limits before analysis can proceed. Quality Control samples that are routinely analyzed include blanks, LCSs and duplicates.

11.14 Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following the *SOP for Checking Pipet Calibration.* Both accuracy and precision verifications are performed, at intervals applicable to the pipet and use. The results of all calibration verifications are recorded in bound logbooks.

11.15 Other Instruments

Calibration for the total organic carbon (TOC), total organic halogen (TOX), and other instruments is performed following manufacturer's recommendations and applicable SOPs.

12.0 DATA REDUCTION, VALIDATION, AND REPORTING

CAS reports the analytical data produced in its laboratories to the client via the certified analytical report (CAR). This report includes a transmittal letter, a case narrative, client project information, specific test results, quality control data, chain of custody information, and any other project-specific support documentation. The following procedures describe our data reduction, validation and reporting procedures.

12.1 Data Reduction and Review

Results are generated by the analyst who performs the analysis and works up the data. All data is initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation, etc.). The resulting data set is either manually entered (e.g., titrimetric or microbiological data) into an electronic report form or is electronically transferred into the report from the software used to process the original data set (e.g., chromatographic software). Once the complete data set has been transferred into the proper electronic report form(s), it is then printed. The resulting hardcopy version of the electronic report is then reviewed by the analyst for accuracy. Once the primary analyst has checked the data for accuracy and acceptability, the hardcopy version of the data is forwarded to the supervisor or second qualified analyst, who reviews the data for errors. Where calculations are not performed a validated software system, the reviewer rechecks a minimum of 10% of the calculations. When the entire data set has been found to be acceptable, a final copy of the report is printed and signed by the laboratory supervisor, departmental manager or senior laboratory staff. The entire data package is then placed into the appropriate service request file, and an electronic copy of the final data package is forwarded to the appropriate personnel for archival. Data review procedures are described in the SOP for *Laboratory Data Review Process*.

Policies and procedures for manual editing of data are established. The analyst making the change must initial and date the edited data entry, without obliteration of the original entry. The policies and procedures are described in the *SOP for Making Entries Into Logbooks and Onto Benchsheets* (SOP ADM-DATANTRY).

Policies and procedures for electronic manual integration of chromatographic data are established. The analyst performing the integration must document the integration change by printing both the "before" and "after" integrations and including them in the raw data records. The policies and procedures are described in the *SOP for Manual Integration of Chromatographic Peaks* (SOP ADM-INT).

12.2 Confirmation Analysis

12.2.1 Gas Chromatographic and Liquid Chromatographic Analyses

For gas chromatographic (GC) and liquid chromatographic (LC) analyses, all positive results are confirmed by a second column, a second detector, a second wavelength (HPLC/UV), or by GC/MS analysis, unless exempted by one of the following situations:

- The analyte of interest produces a chromatogram containing multiple peaks exhibiting a characteristic pattern, which matches appropriate standards. This includes polychlorinated biphenyls and hydrocarbon fuels (e.g., gasoline and diesel).

- The sample is analyzed for benzene, toluene, ethylbenzene, xylenes, and naphthalene (BTEXN), and the sample is found, by a separate analysis, to contain gasoline. In a sample containing no gasoline, the presence of BTEXN compounds will be confirmed.

- The sample meets all of the following requirements:

    1. All samples (liquid or solid) come from the same source (e.g., groundwater samples from the same well) for continuous monitoring. Samples of the same matrix from the same site, but from different sources (e.g., different sampling locations) are not exempt.

    2. All analytes have been previously analyzed in sample(s) from the same source (within the last year), identified and confirmed by a second column or by GC/MS. The chromatogram is largely unchanged from the one for which confirmation was carried out, and the documents indicating previous confirmation must be available for review.

12.2.2 Confirmation Data

Confirmation data will be provided as specified in the method. Identification criteria for GC, LC or GC/MS methods are summarized below:

- GC and LC Methods

    1. The analyte must fall within plus or minus three times the standard deviation (established for the analyte/column) of the retention time of the daily midpoint standard in order to be qualitatively identified. The retention-time windows will be established and documented, as specified in the appropriate Standard Operating Procedure (SOP).

2. When sample results are confirmed by two dissimilar columns or detectors, the agreement between quantitative results must be evaluated. The relative percent difference between the two results is calculated and evaluated against SOP and/or method criteria.

- GC/MS Methods - Two criteria are used to verify identification:

    1. Elution of the analyte in the sample will occur at the same relative retention time (RRT) as that of the analyte in the standard.

    2. The mass spectrum of the analyte in the sample must, in the opinion of a qualified analyst or the department manager, correspond to the spectrum of the analyte in the standard or the current GC/MS reference library.

12.3 Data Review and Validation

The integrity of the data generated in the laboratory is assessed through the evaluation of the results of the analysis of method blanks, laboratory control samples, sample duplicates, matrix spiked samples, QC samples, trip blanks, et al. The numerical criteria for evaluation of these QC samples are listed within each method-specific Standard Operating Procedure. These various QC sample analyses are evaluated using the flow diagrams found in Figures 12-1 through 12-9. Other validation measures of the data include a check of the linearity of the calibration curve, an accuracy check of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed.

12.4 Data Reporting

When an analyst determines that a data package has met the data quality objectives (and/or any client-specific data quality objectives) of the method and has qualified any anomalies in a clear, acceptable fashion, the data package is reviewed by a trained chemist. Prior to release of the report to the client, the project chemist reviews and approves the entire report for completeness and to ensure that any and all client-specified objectives were successfully achieved. The original raw data, along with a copy of the final report, is filed in project files by service request number for archiving. CAS maintains control of analytical results by adhering to standard operating procedures and by observing sample custody requirements. All data are calculated and reported in units consistent with project specifications, to enable easy comparison of data from report to report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). A case narrative may be written by the project chemist to explain any unusual problems with a specific analysis or sample, etc. Either the CAS-defined data qualifiers or project-defined data qualifiers are used depending on project requirements.

For subcontracted analyses, the Project Chemist verifies that the report received from the subcontractor is complete. Verification includes checking that the correct analyses were performed, the analyses were performed for each sample as requested, a report is provided for each analysis, and the report is signed. The Project Chemist accepts the report if all verification items are complete. Acceptance is demonstrated by forwarding the report to the CAS client.

12.5 Documentation

CAS maintains a records system which ensures that all laboratory records of analysis data retained and available. Analysis data is retained for 5 years from the report date unless contractual terms specify a longer retention time. The archiving system is described in the *SOP for Data Archiving*.

12.5.1 Documentation and Archiving of Routine Analysis Data

The archiving system includes all of the following items for each set of analyses performed:

- Benchsheets describing sample preparation (if appropriate);
- Instrument parameters;
- Sample analysis sequence;
- Analysis benchsheets and instrument printouts;
- Chromatograms and peak integration reports for all samples, standards, blanks, spikes and reruns;
- Log book ID number for the appropriate standards;
- Copies of report sheets submitted to the work request file; and
- Copies of Nonconformity and Corrective Action Report (NCAR) forms, if necessary.

Individual sets of analyses are indexed by analysis date and service request number. Since many analyses are performed with computer-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed, they are written on the integration report or securely stapled to the chromatogram, if done on a separate sheet.

12.5.2 Documentation of QC and Data

To summarize the recovery data for surrogates and matrix spikes, a separate documentation system has been established. The results are segregated according to the sample matrix. Additional information is included, indicating those results affected by matrix interferences, etc. Surrogate and matrix spike acceptance limits are listed in Appendix D. This system also includes results for the most recent calibration curves, as well as method validation results.

12.6 Deliverables

In order to meet individual project needs, CAS provides several levels of analytical reports. Basic specifications for each level of deliverable are described in Table 12-1. Variations may be provided based on client or project specifications. This includes (but is not limited to) to following specialized deliverables:

- ADEC – Alaska Department of Conservation specified data package
- ACOE/HTRW – Army Corps of Engineers HTRW specified data package and reporting requirements
- AFCEE – Air Force Center for Environmental Excellence project-specific reporting

When requested, CAS provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. The EDD is prepared by report production staff using the electronic version of the laboratory report to minimize transcription errors. User guides and EDD specification outlines are used in preparing the EDD. The EDD is reviewed and compared to the hard-copy report for accuracy.

Figure 12-1
Evaluation of Method Calibration

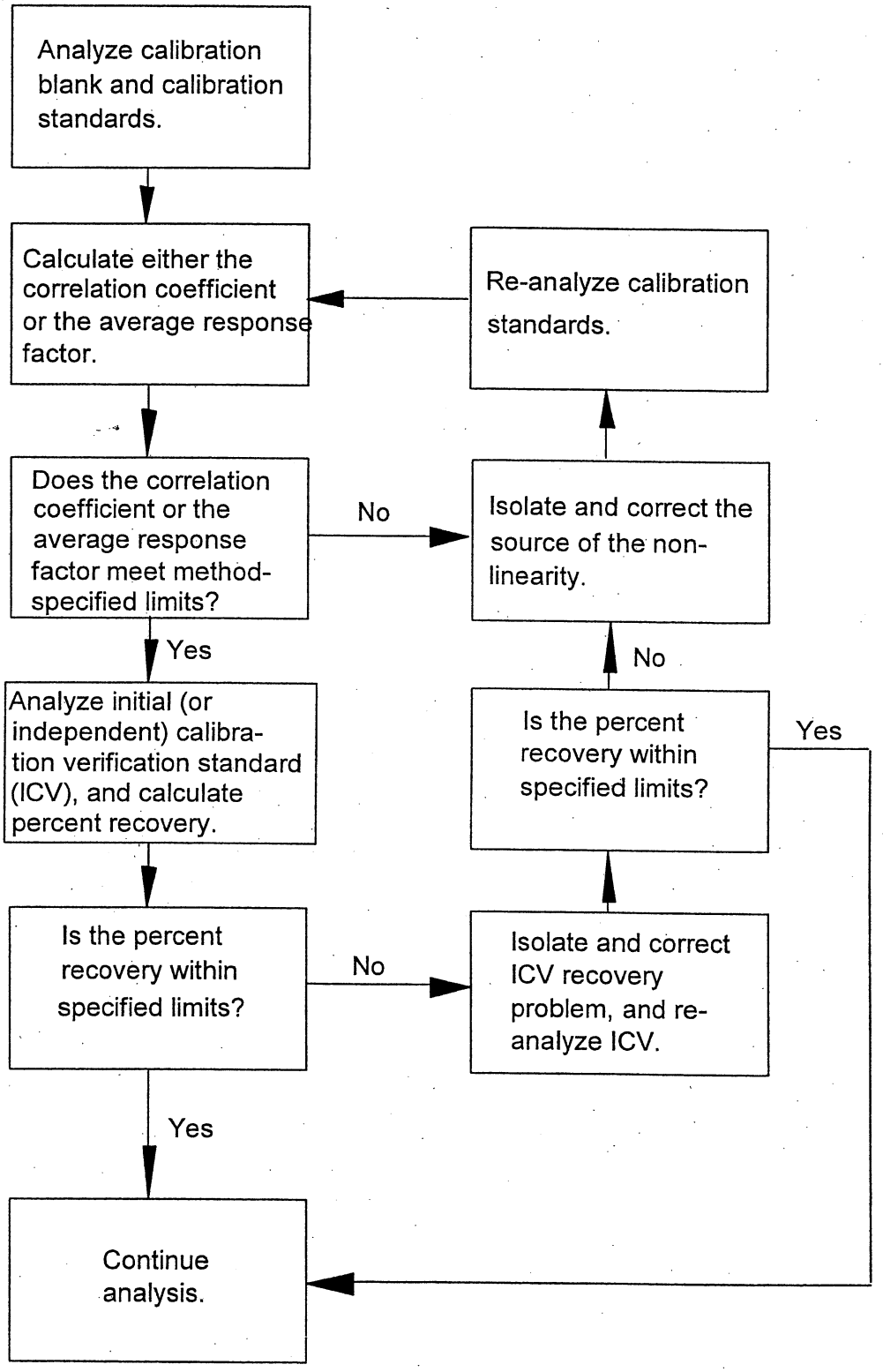


Figure 12-2
Evaluation of Continuing Calibration

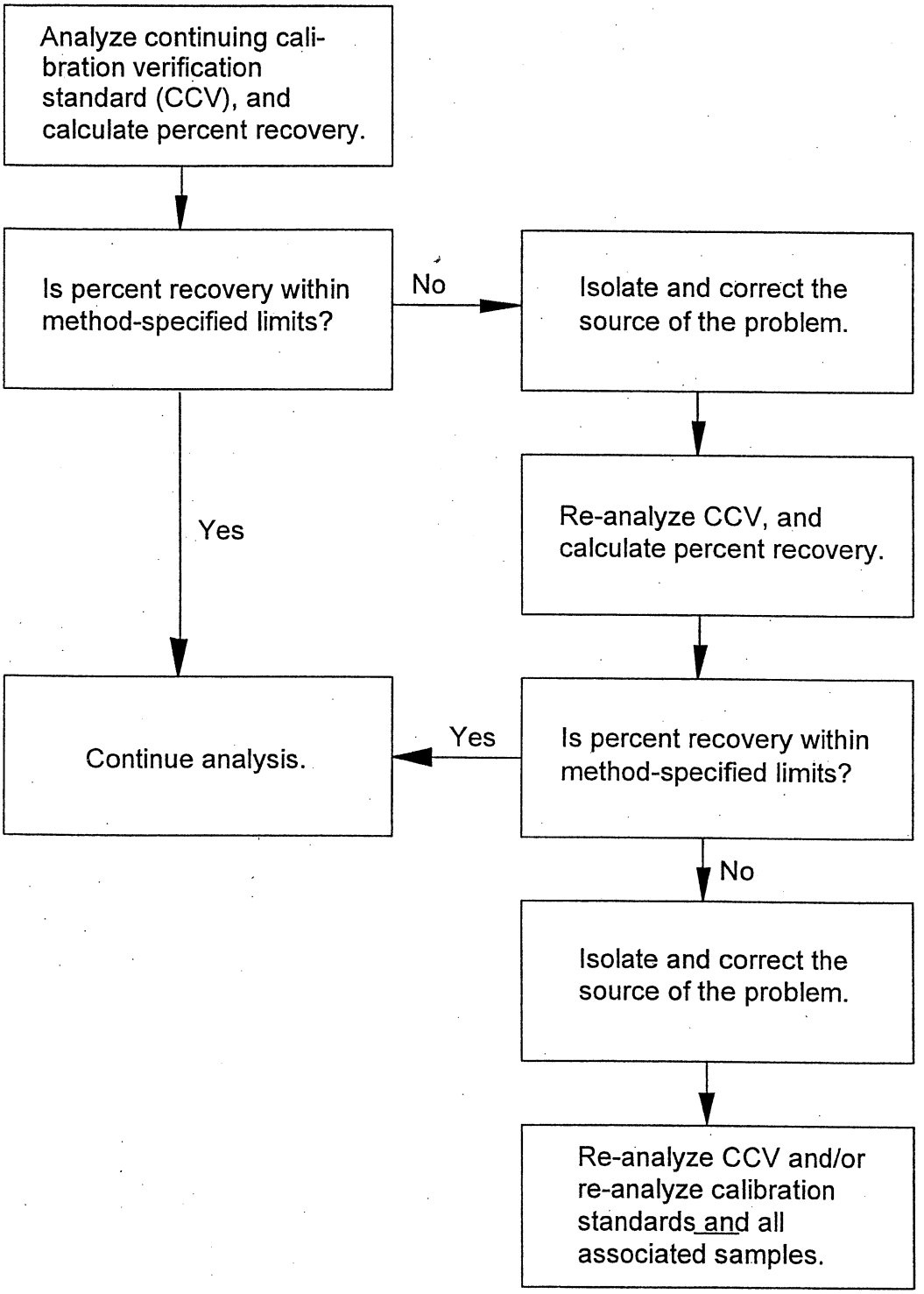


Figure 12-3
Evaluation of Method Blank and Instrument Blank Results

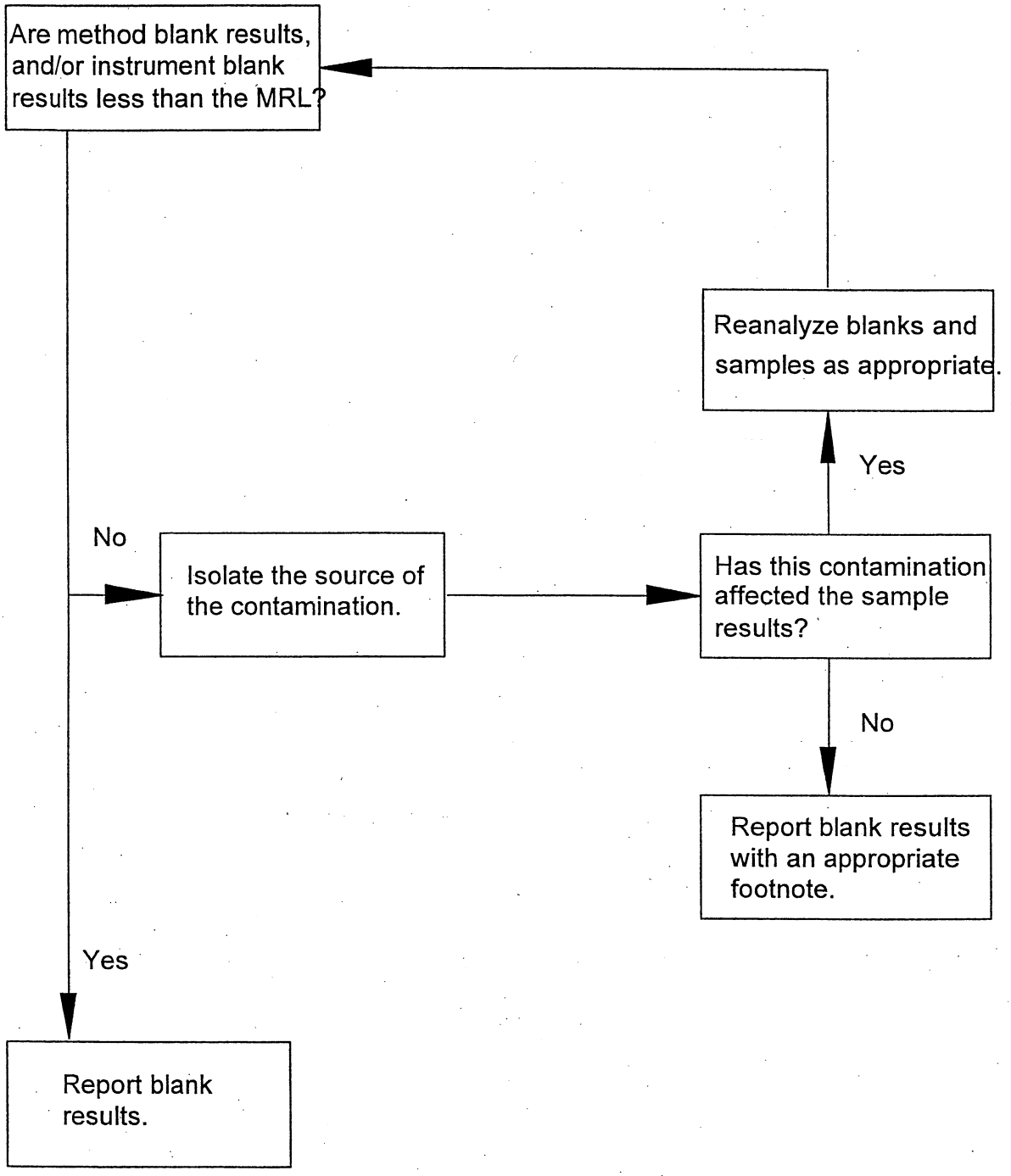


Figure 12-4
Evaluation of Sample Results for Inorganic Analyses

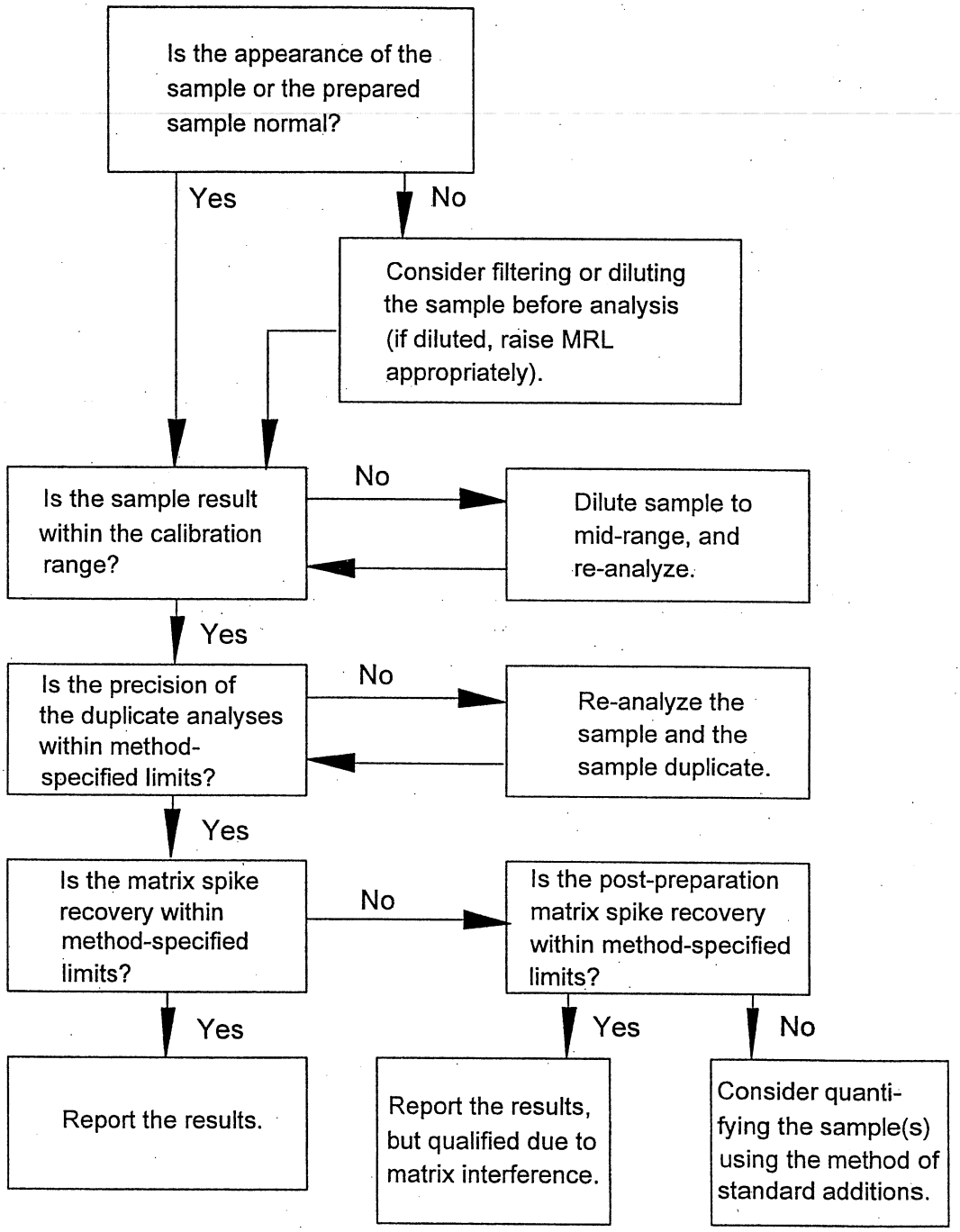


Figure 12-5
Evaluation of Sample Results for Organic Analyses

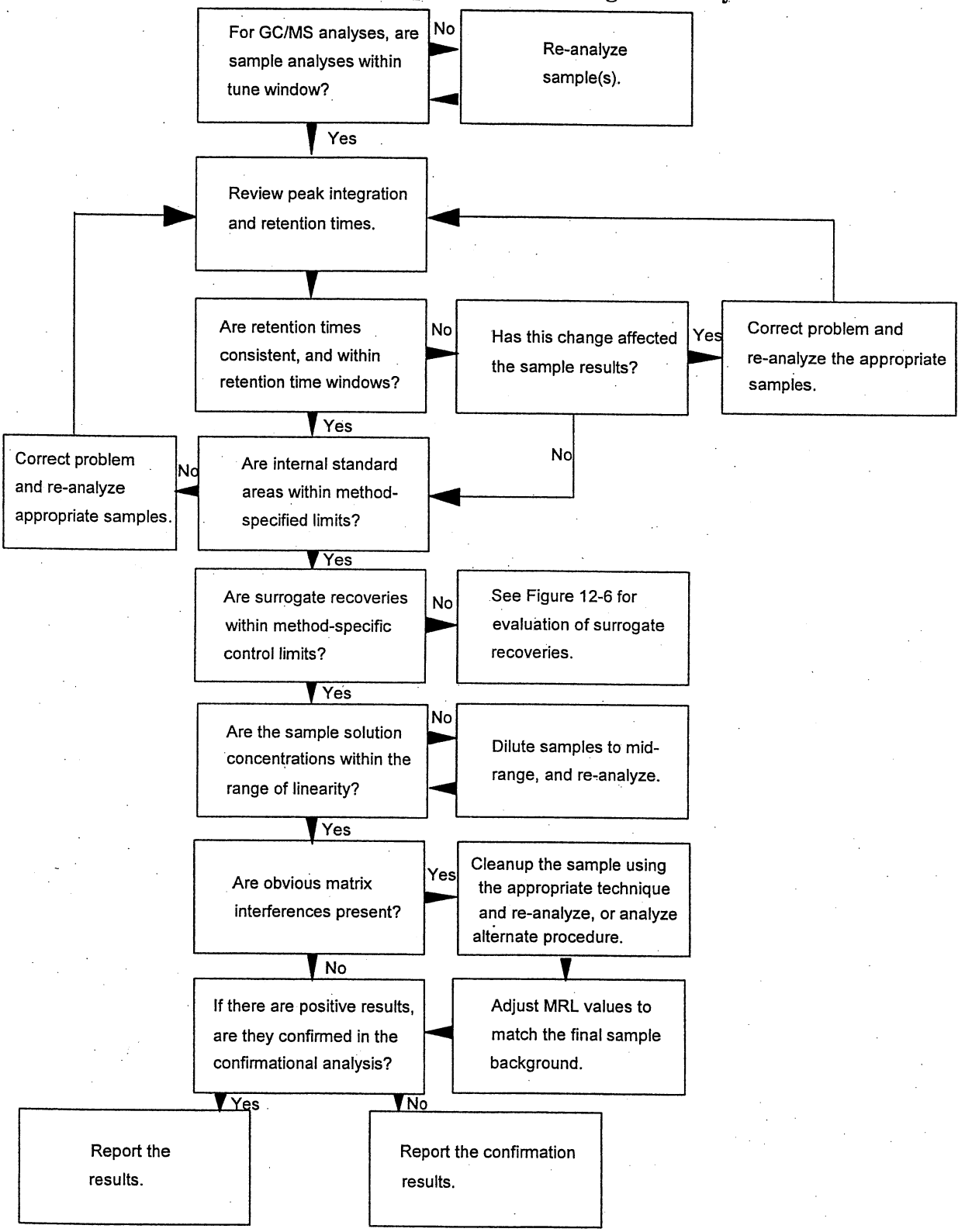


Figure 12-6
Evaluation of Surrogate Compound Recoveries

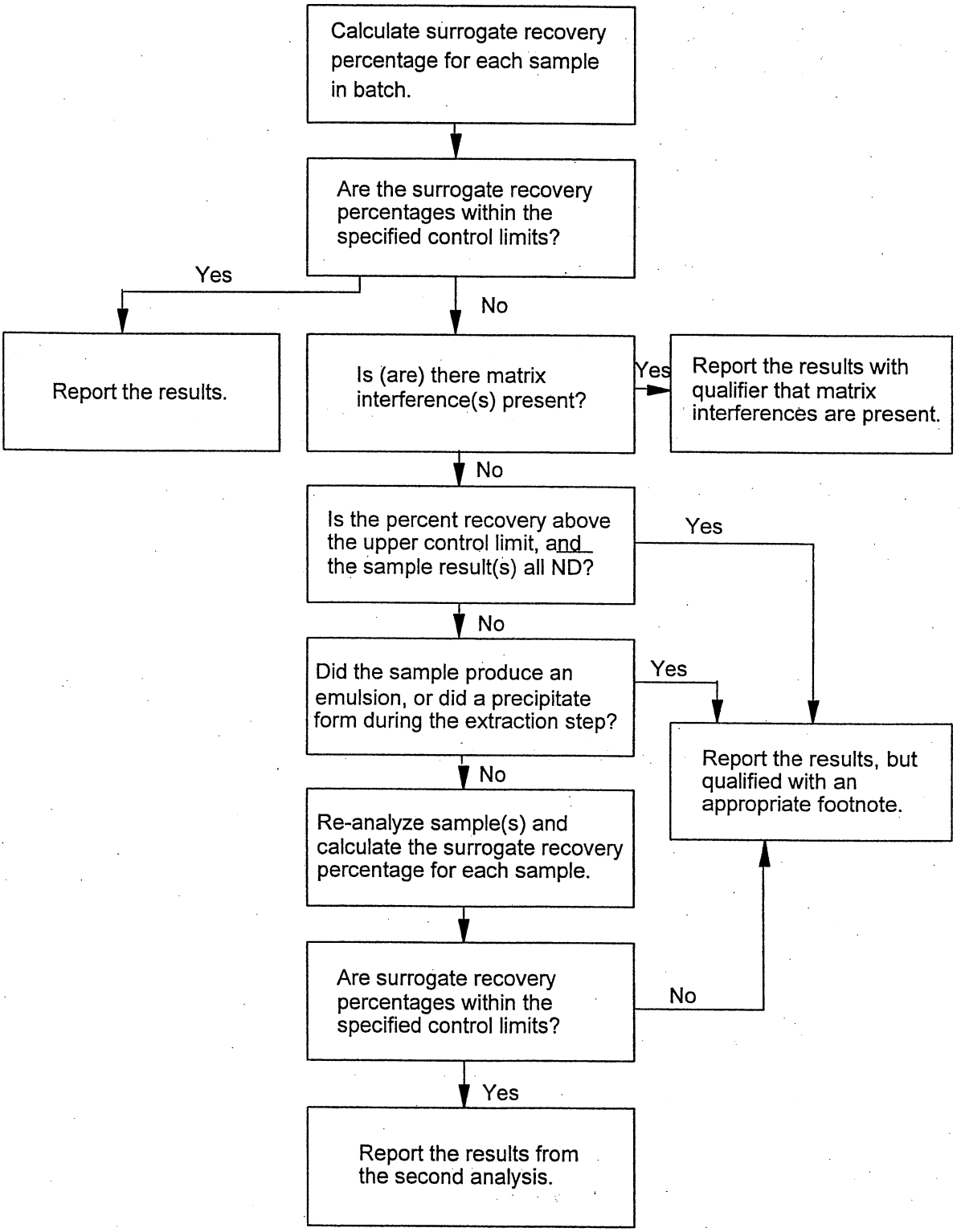


Figure 12-7
Evaluation of Duplicate Sample and/or Duplicate Matrix Spike Results

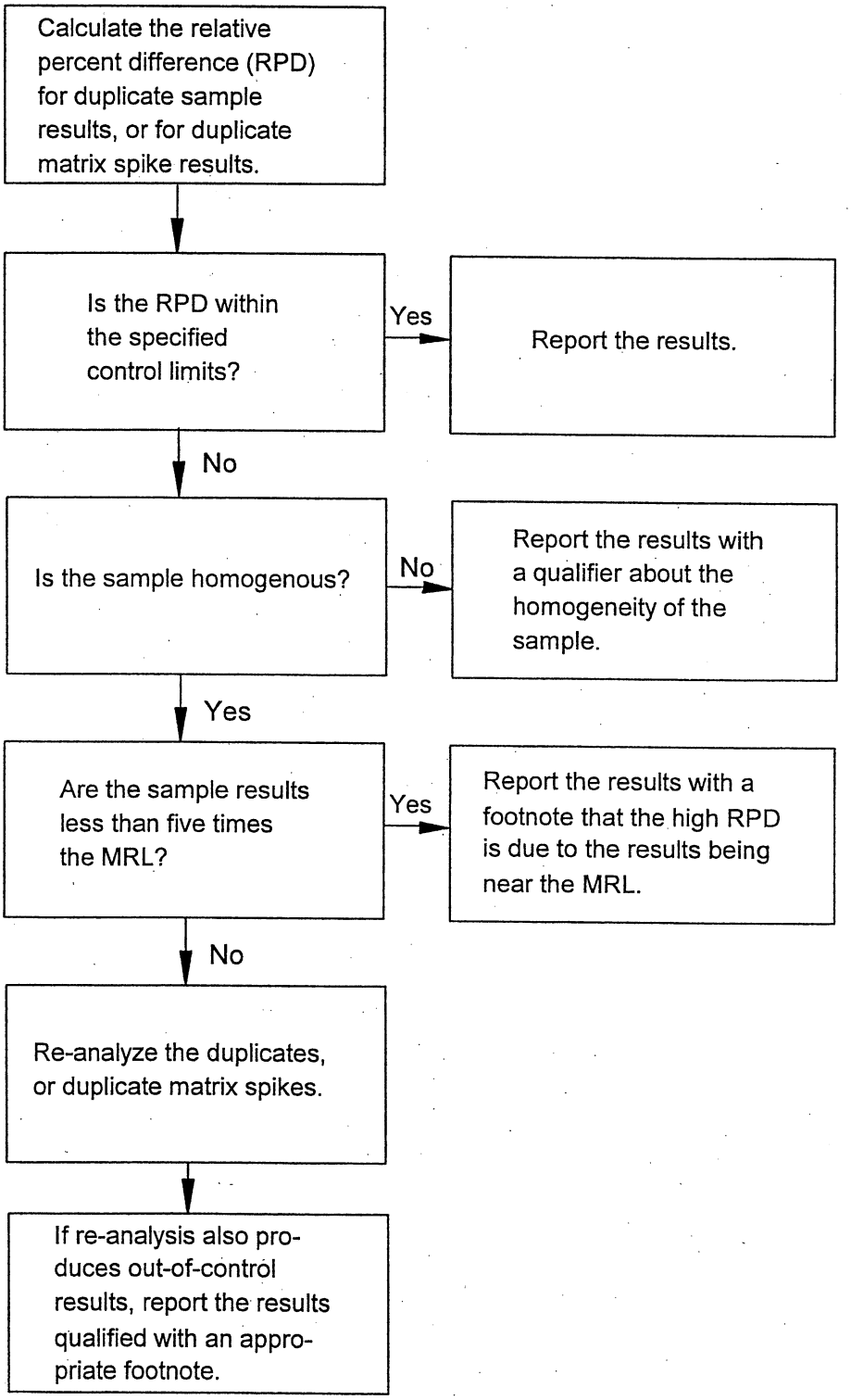


Figure 12-8
Evaluation of Matrix Spike Recoveries

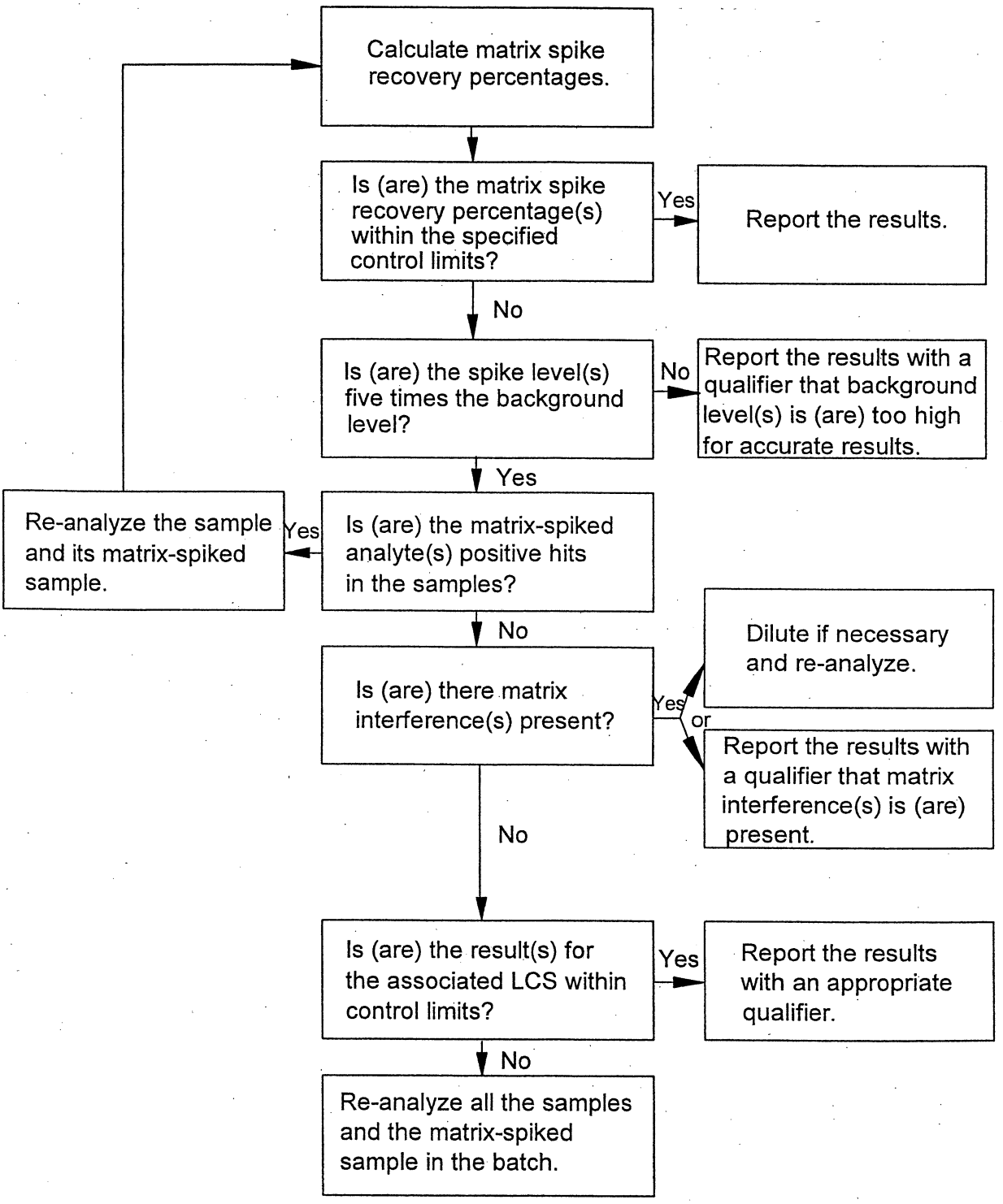


Figure 12-9
Evaluation of Laboratory Control Sample (LCS) Results

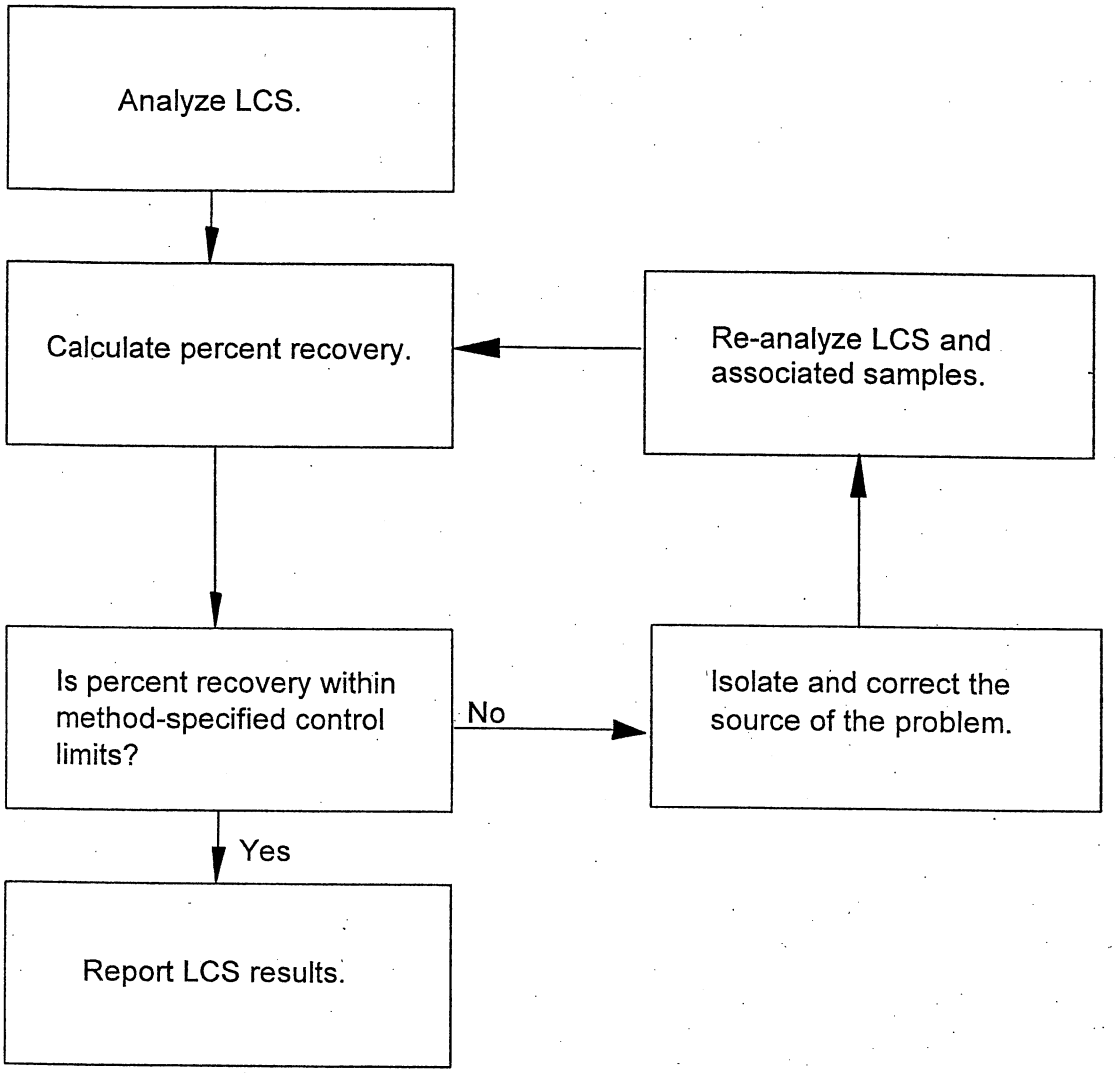


Table 12-1
Descriptions of CAS Data Deliverables

Tier I. Routine Certified Analytical Report (CAR) includes the following:

1. Transmittal letter
2. Sample analytical results
3. Method blank results
4. Surrogate recovery results and acceptance criteria for applicable organic methods
5. Chain of custody documents
6. Dates of sample preparation and analysis for all tests

Tier II and IIA. In addition to the Tier I Deliverables, this CAR includes the following:

1. Matrix spike result(s) with calculated recovery and including associated acceptance criteria
2. Duplicate or duplicate matrix spike result(s) (as appropriate to method), with calculated relative percent difference
3. Tier IIA also includes Laboratory Control Sample (LCS) result(s) with calculated recovery and including associated acceptance criteria

Tier III. Data Validation Package. In addition to the Tier II Deliverables, this CAR includes the following:

1. Case narrative
2. Calibration records and results of initial and continuing calibration verification standards, with calculated recoveries
3. Results of laboratory control sample (LCS) or Quality Control check sample, with calculated recovery and/or associated acceptance limit criteria
4. Results of calibration blanks or solvent blanks (as appropriate to method)
5. Summary forms for associated QC and calibration parameters
6. Copies of all raw data, including extraction/preparation bench sheets, chromatograms, and instrument printouts. For GC/MS, this includes tuning criteria and mass spectra of all positive hits. Results and spectra of TIC compounds will be included upon request.

Tier IV. CLP-Level Data Validation Package.

A complete Data Validation Package containing all sample results, quality control and calibration results, and raw data necessary to fulfill all deliverable requirements of an EPA Contract Laboratory Program (CLP) data package.

13.0 PERFORMANCE AND SYSTEM AUDITS

Quality audits are an essential part of CAS/Kelso's quality assurance program. There are two types of audits used at the facility: System Audits are conducted to qualitatively evaluate the operational details of the QA program, while Performance Audits are conducted by analyzing performance evaluation samples in order to quantitatively evaluate the outputs of the various measurement systems.

13.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of CAS/Kelso are conducted regularly by various regulatory agencies and clients. Table 13-1 summarizes some of the major programs in which CAS/Kelso participates. Programs and certifications are added as required. Additionally, internal system audits of CAS/Kelso are conducted regularly by the Quality Assurance Manager and by the CAS Quality Assurance Director. The internal audits are scheduled as seven auditing events as follows:

- Comprehensive lab-wide system audit – performed annually by the QA Director.
- Lab-wide systems audit – performed annually by the QA Manager.
- Comprehensive data package audits examining compliance with all QA program requirements as applied to selected projects – 1 per quarter.
- Focused audits examining the lab-wide implementation of selected QA program requirements - 1 per year.

The results of each audit are reported to the Laboratory Director for review and comment. All audit and review findings, and corrective actions are documented. Any deficiencies noted by the auditor are summarized in an audit report and corrective action is identified within a specified length of time to correct each deficiency. Should problems impacting data quality be found during an internal audit, any client whose data is adversely impacted will be given written notification if not already provided.

Electronic data audits are generally performed in conjunction with data package audits, but may be performed independently. The electronic audits focus on organic chromatographic data and include an examination of audit trails, peak integrations, calibration practices and files, GCMS tuning data, peak response data, use of appropriate files, and other components of the analysis. The audit also verifies that the electronic data supports the hardcopy reported data.

13.2 Performance Audits

CAS/Kelso also participates in the analysis of interlaboratory proficiency testing (PT) samples. Participation in PT studies is performed on a regular basis and are designed to evaluate all analytical areas of the laboratory. In addition to those PT studies required by programs listed in Table 13-1, CAS participates in additional studies as follows:

- Water Pollution (WP) and additional water parameters, 2 per year.
- Water Supply (WS) PT studies, 2 per year.
- Hazardous Waste/Soil PT studies, 2 per year.
- Underground Storage Tank PT studies, 2 per year.
- Microbiology (WSM and WP) PT studies, 2 per year.
- Other studies as required for specific certification needs.

PT samples are processed by entering them into the LIMS system as samples (assigned Service Request, due date, testing requirements, etc.) and are processed the same as field samples. The laboratory sections handle samples the same as field samples, performing the analyses following method requirements and performing data review. The laboratory sections prepare an analytical report, which is forwarded to the QA Manager for subsequent reporting to the appropriate agencies or study coordinator. Results of the performance evaluation samples and audits are reviewed by the Quality Assurance Manager, Laboratory Director, the laboratory staff, and the CAS Quality Assurance Director. For any results outside acceptance criteria, the analysis data is reviewed to identify a possible cause for the deficiency, and corrective action is taken and documented.

Table 13-1
Current CAS Performance and System Audit Programs

Federal and National Programs

- Naval Facilities Engineering Service Center Validated
  Laboratory for Drinking Water, Wastewater and Hazardous Waste
- U.S. Air Force, Air Force Center for Environmental Excellence (AFCEE)
  Approved Laboratory for Drinking Water, Wastewater and Hazardous Waste
- U.S. Army Corps of Engineers - MRD, HTRW Center of Expertise
  Validated for HTRW parameters
- National Environmental Laboratory Accreditation Conference (NELAC)

State and Local Programs

- State of Alaska, Department of Environmental Conservation
  UST Laboratory, Lab I.D. UST040
- State of Arkansas, Department of Environmental Quality
  Certified Environmental Laboratory
- State of Arizona, Department of Health Services
  License No. AZ0339
- State of California, Department of Health Services, Environmental Laboratory Accreditation Program
  Certification No. 2286
- State of Florida, Department of Health
  Primary NELAC Accreditation No. E87412
- State of Idaho, Department of Health and Welfare
  Certified Drinking Water Laboratory
- State of Indiana, Department of Health
  Certified Drinking Water Laboratory
- State of Louisiana, Department of Environmental Quality
  Approved Environmental Laboratory
- State of Maine, Department of Human Services
  Certified Environmental Laboratory, Lab I.D. WA901
- State of Minnesota, Department of Health
  Certified Environmental Laboratory, Lab I.D. 053-999-368
- State of Montana, Department of Health and Environmental Sciences
  Certified Drinking Water Laboratory, Lab I.D. 0047
- State of Nebraska, Health and Human Services System
  Certified Drinking Water Laboratory
- State of North Carolina, Department of Environment and Natural Resources
  Certified Environmental Laboratory, Lab I.D. 605
- State of Oklahoma, Department of Environmental Quality
  General Water Quality/Sludge Testing, Lab I.D. 9801
- State of Oregon, ORELAP Laboratory Accreditation Program
  Secondary NELAC Accreditation, WA200001
- State of South Carolina, Department of Health and Environmental Control
  Certified Environmental Laboratory, Lab I.D. 61002
- State of Utah, Department of Health, Division of Laboratory Services
  Certified Environmental Laboratory
- State of Washington, Department of Health
  Certified Drinking Water Laboratory No. 017
- State of Washington, Department of Ecology, Environmental Laboratory Accreditation Program
  Certification No. C001

14.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a crucial element of Columbia Analytical Services Quality Assurance program. Instruments at CAS (e.g., ICP/MS and ICP systems, GC/MS systems, atomic absorption spectrometers, analytical balances, gas and liquid chromatographs, etc.) are maintained under commercial service contracts or by qualified, in-house personnel. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at CAS contain extensive information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on **every** instrument used at CAS before it maybe used for sample analysis. If an instrument is modified or repaired, a return to analytical control is **required** before subsequent sample analyses can occur. When an instrument is acquired at the laboratory, the following information is noted in a bound maintenance notebook specifically associated with the new equipment:

- The equipment's serial number;
- Date the equipment was received.;
- Date the equipment was placed into service.;
- Condition of equipment when received (new, used, reconditioned, etc.); and
- Prior history of damage, malfunction, modification or repair (if known).

Equipment records also include a copy of the manufacturer's manual(s) and dates and results of calibrations.

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at CAS. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase.

Responsibility for ensuring that routine maintenance is performed lies with the section supervisor. The supervisor may perform the maintenance or assign the maintenance task to a qualified bench level analyst who routinely operates the equipment. In the case of non-routine repair of capital equipment, the section supervisor is responsible for providing the repair, either by performing the repair themselves with manufacturer guidance or by acquiring on-site manufacturer repair. Each laboratory section maintains a critical parts inventory. The parts inventories include the items needed to perform the preventive maintenance procedures listed in Appendix E.

This inventory or "parts list" also includes the items needed to perform any other routine maintenance and certain in-house non-routine repairs such as gas chromatography/mass spectrometry jet separators and electron multipliers and ICP/MS nebulizer. When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem;
- Repairs and/or maintenance performed;
- Description and/or part number of replaced parts;
- Source(s) of the replaced parts;
- Analyst's signature and date; and
- Demonstration of return to analytical control.

See the table in Appendix E for a list of preventive maintenance activities and frequency for each instrument.

15.0 CORRECTIVE ACTION

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). Failure to meet established analytical controls, such as the quality control objectives outlined in Section 9.0, prompts corrective action. In general, corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the supervisor, team leader, the department manager, and/or the Quality Assurance Manager may examine and pursue alternative solutions. In addition, the appropriate project chemist may be notified in order to ascertain if contact with the client is necessary.

Problems with analysis, as well as the corresponding corrective actions taken, are documented on Nonconformity and Corrective Action Reports (See Figure 15-1) following the requirements in the *SOP for Nonconformity and Corrective Action Documentation* (SOP No. ADM - NCAR). This form is utilized to document corrective actions in response to out-of-control situations. The Quality Assurance Manager reviews each problem, ensuring that appropriate corrective action has been taken by the appropriate personnel. The Nonconformity and Corrective Action Report (NCAR) is filed in the associated service request file and a copy is kept by the Quality Assurance Manager. The Quality Assurance Manager periodically reviews all NCARs looking for chronic, systematic problems that need more in-depth investigation and alternative corrective action consideration. In addition, the appropriate project chemist is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

Corrective action due to a performance audit or a check sample problem is initiated by the Quality Assurance Manager; the affected laboratory laboratory supervisors and managers are promptly informed of performance audit results requiring corrective action.

Figure 15-1

Columbia Analytical Services, Inc.

Nonconformity and Corrective Action Report

SAMPLES/SYSTEM/JOB/CLIENT AFFECTED N&CA Report No. _____

NONCONFORMITY

Analysis/Event:

Instrument/System: _____ Date: _____

Detailed Description of Nonconformity:

Originator (name) : _____ Date: _____

Supervisor Verification: _____ Date: _____

CORRECTIVE ACTION AND OUTCOME

Detailed Description: (Re-establishment of conformity must be demonstrated and documented. Describe the steps that were taken, or are planned to be taken, to correct the particular Nonconformity and prevent the reoccurrence of the Nonconformity.

Is the data to be flagged in the Analytical Report with an appropriate qualifier? **No** **Yes**

Person Responsible: _____ Date: _____

Supervisor Verification: _____ Date: _____

NOTIFICATION - CUSTOMER/CLIENT - INTERNAL/EXTERNAL

Project Chemist Notified by: _____ Date: _____

Customer Notified? **No** **Yes** If Yes, Notifier: _____ Date: _____

Project Chemist/Customer Comments (Retain record, e.g. telephone record, email) _____

_____

ACCEPTANCE OF CORRECTIVE ACTION

Comments:

Corrective Action(s) have been implemented. QA Pgm Mgr: _____ Date: _____

Original: Client File Copies: Supervisor and QA Pgm Mgr NCARFORM.DOC 9/28/01

16.0 QUALITY ASSURANCE REPORTS

Quality assurance requires an active, ongoing commitment by CAS personnel at all levels of the organization. Information flow and feedback mechanisms are designed so that analysts, supervisors and managers are aware of quality assurance issues in the laboratory.

Analysts performing routine tests in the laboratory are aware of the various method acceptance criteria and in-house control limits that must be met in order to generate acceptable results. The analysts are also responsible for generating a Data Quality Report (DQR), or similar, form with every analytical batch they process; this report contains explicit documentation of the various controls that must be met during the analysis. This report also allows the analyst to provide appropriate notes and/or a case narrative if problems were encountered with the analyses. A Non-Conformity and Corrective Action Report (NCAR) (see Section 15.0) may also be attached to the data prior to review. Supervisors or qualified analysts review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and corrected if possible.

It is the responsibility of each laboratory unit to provide the project chemist with a final report of the data, accompanied by signature approval. Footnotes and/or narrative notes must also accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate project chemist, who in turn reviews the entire collection of analytical data for completeness. The project chemist must also review the entire body of data to ensure that any and all client-specified objectives were successfully achieved. A case narrative may be written by the project chemist to explain any unusual problems with a specific analysis or sample, etc.

The Quality Assurance Manager (QAM) provides overview support to the project chemists if required to do so (e.g., contractually specified, etc.). The QAM is also responsible for the oversight of all internal and external audits, for all proficiency testing sample and analysis programs, and for all laboratory certification/accreditation responsibilities.

The QAM also prepares quarterly reports for the Laboratory Director which summarize the various QA/QC activities that have occurred during the previous quarter. The typical report will address such topics as the following:

- Status, schedule, and results of internal and external audits (e.g., deficiency resolution status);
- Status, schedule, and results of internal and external proficiency testing studies;
- Status of certifications, accreditations, and approvals;
- Status of QA Manual and SOP review and revision;

- Status of MDLs studies;
- Discussion of QC problems in the laboratory;
- Discussion of corrective action program issues;
- Status of staff training and qualification; and
- Other topics as appropriate.

Any problems noted by the Laboratory Director are then discussed during the regularly-scheduled senior staff operations meetings with all appropriate department managers. The Laboratory Director performs an annual documented review of the quality system to identify any necessary changes or improvements to the quality system.

17.0 PERSONNEL TRAINING

Technical position descriptions are available for all employees, regardless of position or level of seniority. These documents are maintained by the Human Resources personnel and are available for review. In order to assess the technical capabilities and qualifications of a potential employee, all candidates for employment at CAS are evaluated, in part, against the appropriate technical description.

Training begins the first day of employment at CAS when the company policies are presented and discussed. Safety and QA/QC requirements are integral parts of all technical SOPs and, consequently, are integral parts of all training processes at CAS. Safety training begins with the reading of the *Safety Manual.* All employees are also required to attend safety meetings during which the safety program is discussed and safety training is presented by the Environmental, Health and Safety Officer. Each employee is responsible for complying with the requirements of the QA Manual and QA/QC requirements associated with their function(s).

CAS requires each employee to participate in Ethics training, which is part of the CAS Improper Practices Prevention Program. CAS also encourages its personnel to continue to learn and develop new skills that will enhance their performance and value to the Company. Ongoing training occurs for all employees through a variety of mechanisms. The "CAS University" education system, external and internal technical seminars and training courses, and laboratory-specific training exercises are all used to provide employees with professional growth opportunities.

17.1 Initial Demonstration of Proficiency

Training in analytical procedures typically begins with the reading of the Standard Operating Procedure (SOP) for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the applicable Demonstration of Proficiency analysis qualifies the analyst to perform the method independently. Demonstration of Proficiency is performed by one of the following:

- Successful completion of a Initial Precision and Recovery (IPR) study (required where mandated by the method).
- Analysis of 4 consecutive Laboratory Fortified Blanks, with acceptable accuracy and precision.

- Where spiking is not possible but QC standards are used ("non-spiked" Laboratory Control Samples), analysis of 4 consecutive Laboratory Control Samples with acceptable accuracy and precision.

- Where one of the three above is not possible, special requirements are as follows:

    - Total Settleable Solids: Successful single-blind PT sample analysis and duplicate results with RPD<10%.
    - Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
    - Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

A flowchart identifying the Demonstration of Proficiency requirements is given in Figure 17-1. The flowchart identifies allowed approaches to assessing Demonstration of Capability when a 4-replicate study is not mandated by the method, when spiking is not an option, or when QC samples are not readily available.

17.2 Continuing Demonstration of Proficiency

A periodic demonstration of proficiency is required to maintain continuing qualification. Continuing Demonstration of Proficiency is required each year, and may be performed one of the following ways:

- Successful performance on external (independent) single-blind PT sample analyses using the test method, or a similar test method using the same technology.

- Performing Initial Demonstration of Proficiency as described above, with acceptable levels of precision and accuracy.

- Performing an acceptable Method Detection Limit study for the method.

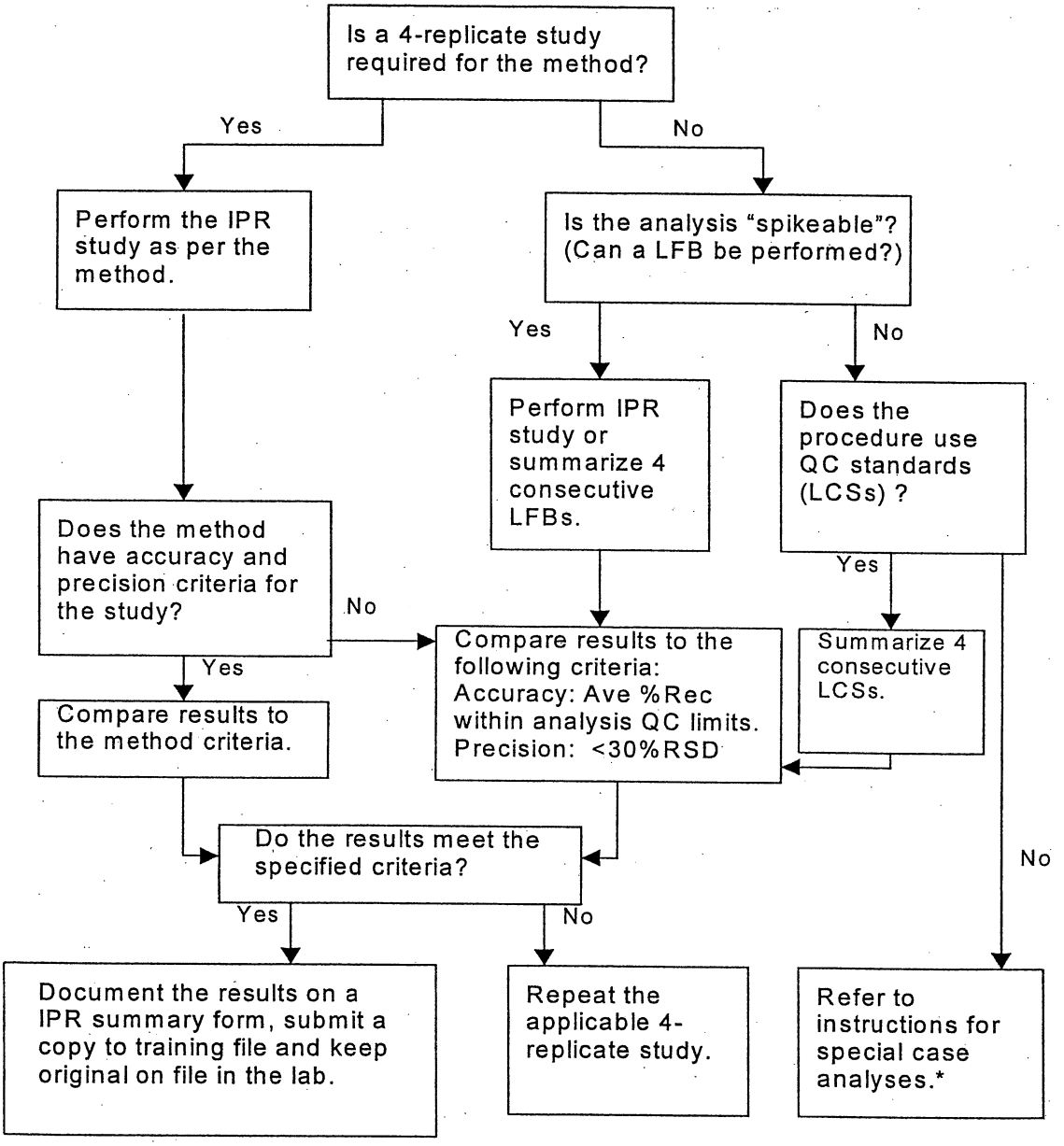
- For methods for which PT samples are not available and a spiked analysis (LFB, MDL, etc.) is not possible, analysis of field samples that have been analyzed by another analyst with statistically indistinguishable results.

17.3 Documentation of Training

Records are maintained to indicate the employee has the necessary training, education, and experience to perform their functions. Information of previously acquired skills and abilities for a new employee is maintained in Human Resources personnel files and CAS resumes.

A database is used to record the various technical skills and abilities acquired and maintained by an employee while employed by CAS. Information in the database includes the employee's name, a description of the skill including the appropriate method and SOP reference, the mechanism used to document proficiency, and the date the training was completed. General procedures for documenting technical training are described in the *SOP for Documentation of Technical Personnel Training (SOP No. ADM-TRANDOC).*

Figure 17-1
Initial Demonstration of Proficiency Requirements



*Total Settleable Solids: Successful PT sample analysis and duplicate results with RPD<10%.
*Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
* Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

18.0 REFERENCES FOR ANALYTICAL PROCEDURES

The analytical methods used at CAS generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection limit, the concentration of the analyte being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives. Typical methods used at CAS are taken from the following references:

- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, (September 1986) and Updates I (July 1992), II (September 1994), IIA (August 1993), IIB (January 1995), III (December 1996), and Proposed Update IVA (January 1998). See Chapters 1, 2, 3, and 4.

- *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, (Revised March 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) and Supplement I, EPA/600/R-94/111 (May 1994).

- *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, EPA 600/4-82-057 (July 1982) and 40 CFR Part 136, Appendix A.

- *Methods for the Determination of Organic Compounds in Drinking Water*, EPA/600/4-88/039 (December 1988) and Supplement I, EPA/600/4-90/020 (July 1990) and Supplement II, EPA/600/R-92/129 (August 1992) and Supplement III, EPA/600/R-95/131 (August 1995).

- *Standard Methods for the Examination of Water and Wastewater*, 16th Edition (1985); 17th Edition (1989); 18th Edition (1992); 19th Edition (1995). See Introduction in Part 1000.

- 40 CFR Part 136, Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act.

- 40 CFR Part 141, National Primary Drinking Water Regulations.

- *Analytical Methods for Petroleum Hydrocarbons*, ECY 97-602, Washington State Department of Ecology, June 1997.

- State-specific total petroleum hydrocarbon methods for the analysis of samples for gasoline, diesel, and other petroleum hydrocarbon products (Alaska, Arizona, California, Oregon, Washington, Wisconsin, etc.).

- Annual Book of ASTM Standards, Part 31, Water.

- EPA Contract Laboratory Program, Statement of Work for Organic Analysis, SOW Nos. OLM01.8, OLM02.0, OLM03.1, OLM03.2, and OLM04.2.

- EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, SOW No. ILM04.0.

- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, EPA-540/R-94/012 (February 1993).

- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, EPA-540/R-94/013 (February 1994).

- National Institute for Occupational Safety and Health (NIOSH) *Manual of Analytical Methods*, Third Edition (August 1987); Fourth Edition (August 1994).

- *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*, for USEPA and USACE (March 1986), with revisions through April 1997.

- WDOE 83-13, *Chemical Testing Methods for Complying with the State of Washington Dangerous Waste Regulations* (March 1982) and as Revised (July 1983 and April 1991).

- *Identification and Listing of Hazardous Waste,* California Code of Regulations, Title 22, Division 4.5, Chapter 11.

- *Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Wastewater*, EPA 821-R-93-017 (October 1993).

- *Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters*, EPA 821-B-98-016 (July 1998).

- National Council of the Pulp and Paper Industry for Air and Stream Improvement (NCASI).

- *Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185 (August 1995).

- *Manual for the Certification of Laboratories Analyzing Drinking Water*, 4th Edition, EPA 815-B-97-001 (March 1997).

- National Environmental Laboratory Accreditation Conference (NELAC) Quality Standards, 1999, 2000, 2001.

APPENDIX A

LIST of QA PROGRAM DOCUMENTS

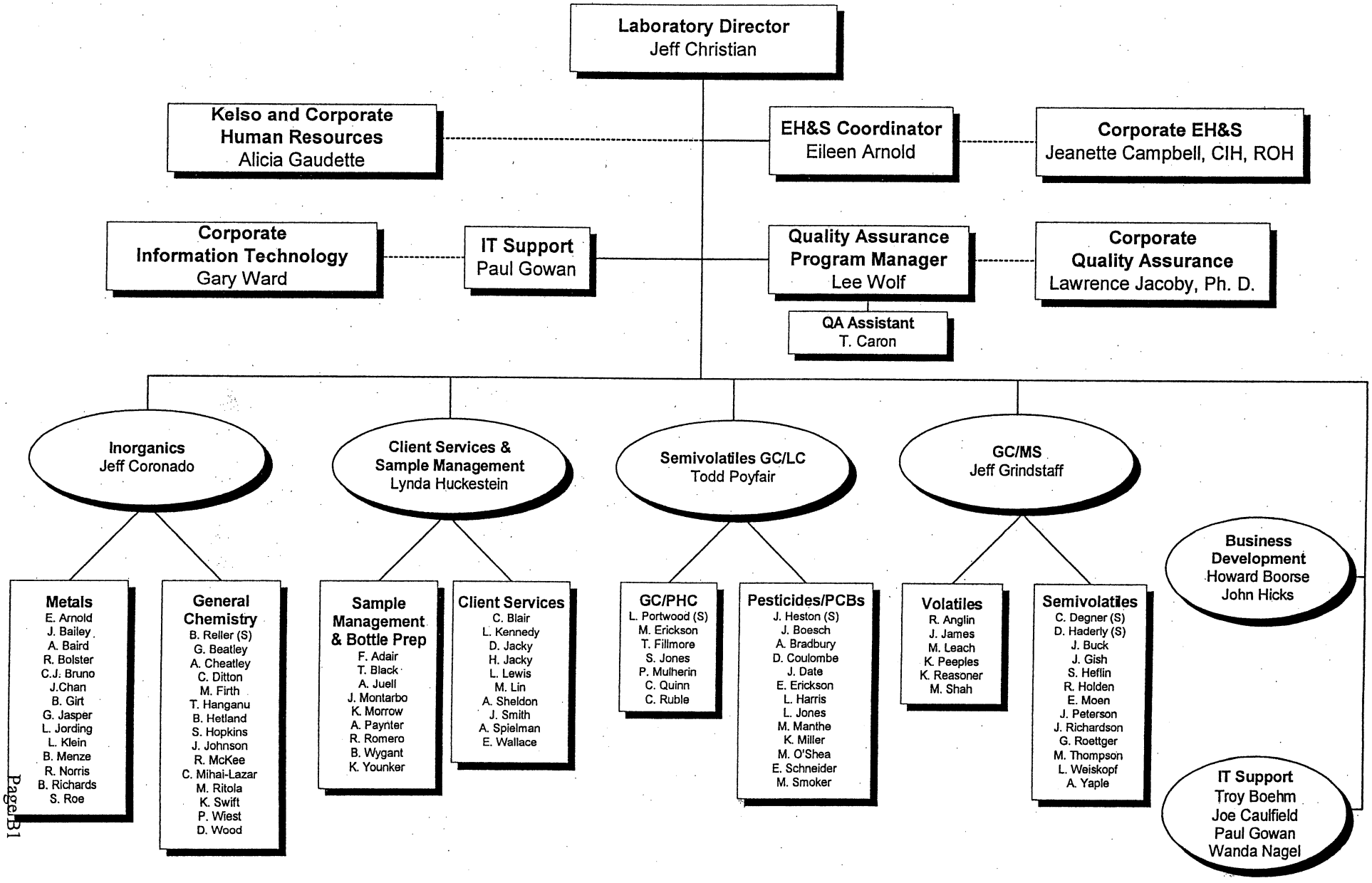
Quality Assurance Manual	6/6/02
Software Quality Assurance Plan	8/7/01
SOP Table of Contents	Soplist.doc
CAS-Kelso Certifications/Accreditations	Cert_kel.xls
Columbia Analytical Services MDL Tracking Spreadsheet	Mdl_list.xls
Technical Training Summary Database	TrainDat.mdb
Approved Signatories List	QA Dept Files
Personnel resumes/qualifications	(all staff)
Personnel Job Descriptions	Human Resources
Quality Control Criteria	Qclimits.xls
Master Logbook of Laboratory Logbooks	Masterlog-001
ADMINISTRATIVE STANDARD OPERATING PROCEDURES	
ADMINISTRATIVE - LOCAL	FILE NAME
CHECKING PIPETTE CALIBRATION	ADM-CPIP
CONTINGENCY PLAN FOR LABORATORY EQUIPMENT FAILURE	ADM-ECP
CONTROL CHARTING QUALITY CONTROL DATA	ADM-CHRT
DATA ARCHIVING	ADM-ARCH
DATA REPORTING AND REPORT GENERATION	ADM-RG
INTERNAL QUALITY ASSURANCE AUDITS	ADM-IAUD
LABORATORY DATA REVIEW PROCESS	ADM-DREV
MANUAL PEAK INTEGRATION FO AFCEE PROJECTS	ADM-AFINT
METHOD DETECTION LIMIT DOCUMENTATION AND CONTROL	ADM-MDLC
PROJECT CHEMIST REVIEW OF SERVICE REQUEST FILES	ADM-PCR
REAGENT LOGIN AND TRACKING	ADM-RLT
SUPPORT EQUIPMENT MONITORING AND CALIBRATION	ADM-SEMC
THERMOMETER CALIBRATION	ADM-TCAL

ADMINISTRATIVE - CORPORATE	FILE NAME
CHAIN OF CUSTODY FOR SAMPLE TRANSFER BETWEEN LABORATORIES	ADM-COC
CHECKING NEW LOTS OF CHEMICALS FOR CONTAMINATION	ADM-CTMN
CONTROL LIMITS	ADM-CTRL_LIM
DEALING WITH COMPLAINTS	ADM-CMPLT
DOCUMENT CONTROL	ADM-DOCCTRL
DOCUMENTATION OF TECHNICAL PERSONNEL TRAINING	ADM-TRANDOC
ELECTRONIC DATA AUDITING	ADM-E_DATAUDIT
MAKING ENTRIES INTO LOGBOOKS AND ONTO BENCHSHEETS	ADM-DATANTRY
MANUAL INTEGRATION OF CHROMATOGRAPHIC PEAKS	ADM-INT
NONCONFORMITY AND CORRECTIVE ACTION	ADM-NCAR
NONFINANCIAL PERFORMANCE MEASURES	ADM-NFPM
PREPARATION OF STANDARD OPERATING PROCEDURES	ADM-SOP
PURCHASING THROUGH CAS PURCHASING AGENT	ADM-PUR
QUALIFICATION OF SUBCONTRACT LABORATORIES	ADM-SUBLAB
SAMPLE BATCHES	ADM-BATCH
SIGNIFICANT FIGURES	ADM-SIG.FIG
THE DETERMINATION OF METHOD DETECTION LIMITS	ADM-MDL
SAMPLE MANAGEMENT SOPS	FILE NAME
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT
SAMPLE DISPOSAL	SMO-SDIS
SAMPLE RECEIVING	SMO-GEN
SAMPLE TRACKING AND LABORATORY CHAIN OF CUSTODY	SMO-SCOC

APPENDIX B

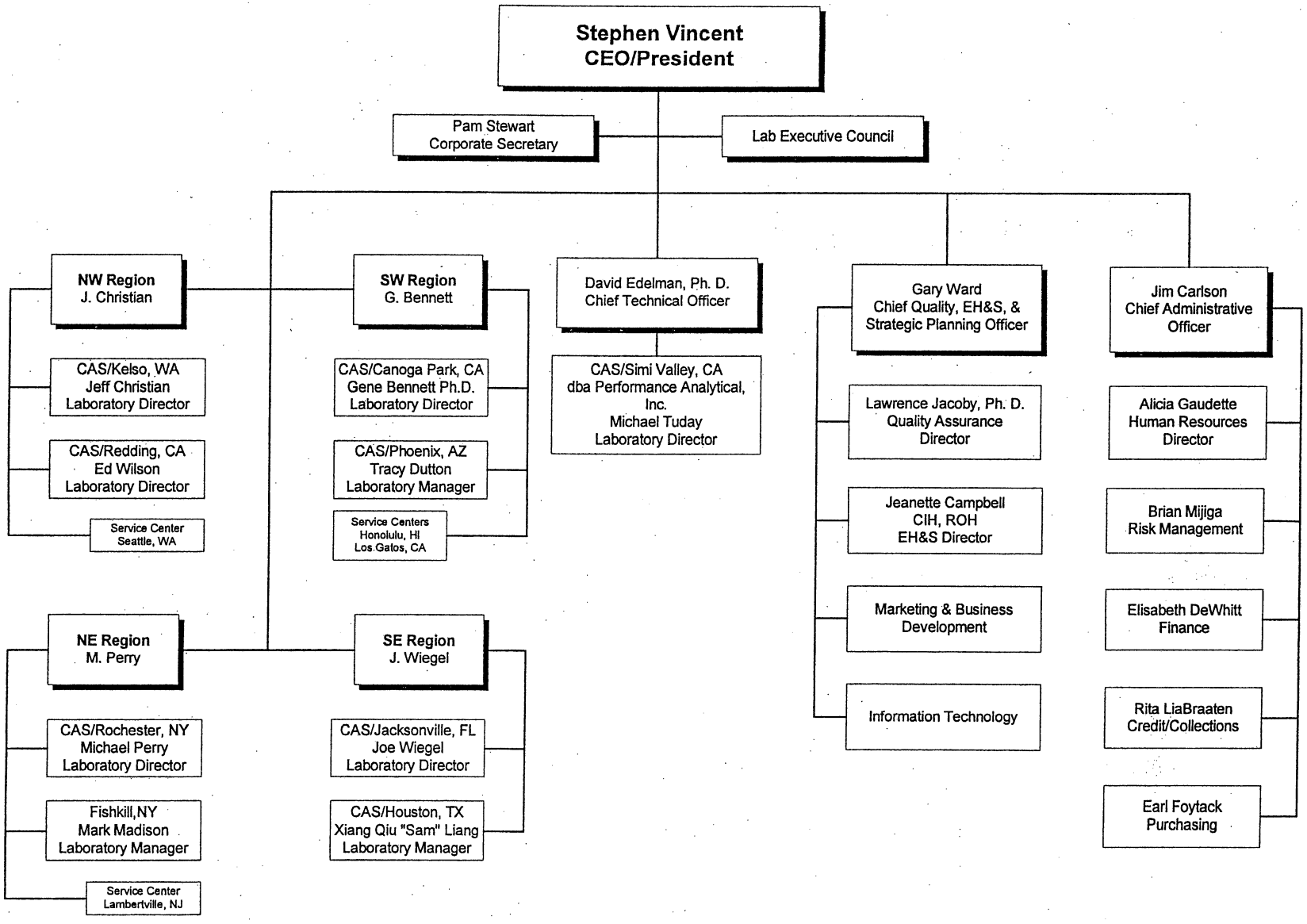
ORGANIZATIONAL CHARTS and RESUMES OF KEY PERSONNEL

Columbia Analytical Services, Inc.
Kelso, Washington Laboratory Organization



Revised on 05/10/02

Columbia Analytical Services, Inc.
Laboratory Division Organization



Revised on 05/16/0

JEFFREY D. CHRISTIAN AUGUST 1989-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position

Vice President/Kelso Laboratory Director. January 1996-Present.

Responsibilities

All phases of laboratory operations, including project planning, budgeting, and quality assurance. Also responsible for additional duties acquired as a member of the Columbia Analytical Services, Inc. Board of Directors.

Experience

Kelso Laboratory Director - Columbia Analytical Services, Inc., Kelso, Washington. All phases of laboratory operations, including project planning, budgeting, and quality assurance. 1993-1995.

Kelso Laboratory Operations Manager - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibility was directing the daily operation of the Kelso Laboratory. Other responsibilities and duties included functioning as a technical consultant to clients, providing assistance in developing and planning analytical schemes to match client objectives, and writing and developing analytical procedures/methods. Also, Project Manager for State of Alaska Department of Environmental Conservation contract, and Coordinator for EPA Special Analytical Services (SAS). 1992-1993.

Project Chemist and Manager, Metals Analysis Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Responsible for directing the daily operation of the Metals Laboratory, including the sample preparation, AAS, ICP-OES, and ICP-MS Laboratories. 1989-1992.

Scientist - Weyerhaeuser Technology Center, Federal Way, Washington. Supervised atomic spectroscopy laboratory which included flame and furnace AAS, ICP-OES, and sample preparation capabilities to handle a wide variety of sample types. Interfaced with internal and external clients to provide technical support. Wrote and developed analytical procedures/methods. 1986-1989.

Lead Technician, Metals Lab - Weyerhaeuser Technology Center, Federal Way, Washington. Primary ICP and AAS analyst for EPA-CLP contract work. Extensive experience in wide variety of environmental and product-related testing. 1981-1986.

Research Assistant - ITT Rayonier, Olympic Research Division, Shelton, Washington. Performed water quality tests, product-related analytical tests, corrosion tests (i.e., potentiometric polarization techniques), and operated pilot equipment specific to the pulp and paper industry. 1978-1981

Education

Evergreen State College, Olympia, Washington. B.S. Chemistry. 1993.
VG-Elemental ICP-MS Training Course. 1992.
Pacific Lutheran University, Tacoma, Washington. 1988-1989.
Tacoma Community College, Tacoma, Washington. 1970-1971, 1988-1989.
Perkin-Elmer Advanced Furnace, Norwalk, Connecticut. 1986.
L.H. Bates Technical, Tacoma, Washington - Chemistry Certification. 1976-1978.
Central Washington University, Ellensburg, Washington. 1969-1970.

Publications/ Presentations

ICP-MS Analysis of Marine Tissue, AOAC Meeting, Olympia, Washington, June 1994.

Analyzing Seawater by ICP-MS, Environmental Lab, October/November 1993, Vol. 5. No. 5, page 10-13.

Reductive Precipitation as a Pre-Concentration Technique for the Analysis of Seawater by ICP-MS, June 1993, AOAC, Olympia, Washington.

The Analysis for Metals at PPT Levels in Environmental Samples Using Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) Techniques, HazMat West, November, 1992, Long Beach, California.

Analysis of Pulping Liquor by ICP Emission, January, 1984. International Conference on Plasma Spectrochemistry, San Diego, California. (Co-Author).

Modification of ICP for Sulfur Determination in Black Liquors, January, 1984. International Conference on Plasma Spectrochemistry, San Diego, California. (Co-Author)

LEE E. WOLF *OCTOBER 1988-PRESENT*

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position Responsibilities

Kelso Quality Assurance Manager. 1996-Present.

Responsible for the overall coordination of the laboratory QA program and for ensuring that established quality objectives are met. Responsible for Quality Assurance functions including the Quality Assurance Manual, certifications, documenting standard operating procedures, and maintaining performance evaluation records. Oversee balance calibration and sample storage temperature control. Maintain certifications/accreditations for regulatory agencies and client certifications or approval programs. Act as primary point of contact during laboratory audits. Provides audit responses and initiates any changes in procedures resulting from an audit. Coordinate the analysis of performance evaluation samples required for certification/accreditation programs. Report and review results for these analyses. Conduct internal audits and make recommendations for corrective action.

Experience

Project Chemist/Principal Organic Scientist - Columbia Analytical Services, Inc., Kelso, Washington. Perform GC and GC/MS method development, and special projects coordination. Oversees and acts as technical advisor to the GC and GC/MS laboratories. Acts as GC/MS interpretation specialist and CLP organics specialist. Also responsible for Project Chemist functions including managing and coordinating projects for clients, identifying client's needs, and preparing data reports. 1994-1996.

Semivolatile Organics Department Manager - Columbia Analytical Services, Inc., Kelso, Washington. Overall management of the semivolatile organics analysis section. Oversee the operation of semivolatile GC/MS instrumentation, data review and reporting, and related QA/QC functions. Responsible for the supervision of staff, including scheduling, training, and other personnel issues. Also responsible for Project Chemist functions for organics EPA-SAS and other clients. Involved coordinating and scheduling projects for clients, identifying client's needs, and preparing data reports (1992-1994). 1988-1994.

GC/MS Chemist - U.S. Testing Co., Richland, Washington. Responsible for GC and GC/MS analysis of water and soil samples for volatiles and semivolatiles by EPA protocol, including Methods 8240, 8270 and CLP. Coordinated extraction and GC-GC/MS areas to manage sample/data flow through the lab. Experience also with pesticide/PCB analysis by EPA Methods 8080 and CLP. Responsible for development of analysis methods for non-routine pesticides and herbicides and performed HPLC analysis. 1985-1988.

Laboratory Assistant - Eastern Washington University, Cheney, Washington. Responsible for supervision and instruction of organic chemistry labs. Experience with GC and IR operation. Responsible for lab safety. 1985.

Chemist Assistant - Spokane County Air Pollution Control Authority, Spokane, Washington. Responsible for gathering and analyzing air samples for CO content using IR equipment. 1984.

Education

Documenting Your Quality System, A2LA Short Course, Las Vegas, Nevada, 1998
Internal Laboratory Audits, A2LA Short Course, Las Vegas, Nevada, 1998
Mass Spectra Interpretation, ACS Short Course, Denver, Colorado. 1992.
Eastern Washington University, Cheney, Washington - B.S. in Chemistry. 1985.
Eastern Washington University, Cheney, Washington - B.A.(minor) in Geology. 1985.

Publications/ Presentations

Selected Ion Monitoring: Issues for Method Development, Panel Discussion, Association of Official Analytical Chemists, (AOAC) Pacific Northwest Regional Meeting, 1995.
Method Enhancement Techniques for Achieving Lowlevel Detection of Butyl Tin in Marine Sediments and Tissues, Association of Official Analytical Chemists (AOAC Pacific Northwest Regional Meeting, 1994.
The Determination of Low-Level Concentrations of Polynuclear Aromatic Hydrocarbons (PAHs) in Soil and Water Using Gas Chromatography/Mass Spectroscopy Selected Ion Monitoring (GC/MS SIM), HazMat West, November, 1992, Long Beach, California.

Memberships

American Chemical Society, 1989 to present.

LYNDA A. HUCKESTEIN JANUARY 1989-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position **Client Services Manager**

Responsibilities

Management of client services departments including responsibilities for Project Management, Electronic Data Deliverables and Sample Management. 1998-Present.

Employee representative to the board of directors for the Columbia Analytical Services Holding Company. 1999-2002.

Experience

Senior Project Chemist- Responsible for approximately $1.5 million dollars of client work annually, Lynda's primary responsibilities include technical project management and client service. She is responsible for meeting her clients' needs and providing appropriate analysis and timely reporting. Her area of expertise includes pulp & paper, marine, mining and DOD project management. 1992-2001

Report Generation Supervisor- Lynda also leads a group of electronic data deliverable staff responsible for the generation of the client specific electronic data deliverables. Her role is to manage the coordination of client specifications with the current laboratory data reporting platform. She also facilitates continuous quality improvement projects within the report generation group.

Project Chemist, Georgia Pacific - For over five years Lynda has been the primary liaison between the Georgia Pacific facilities in Camas Washington and Clatskanie Oregon. Columbia provides analytical testing and consultation in support of NPDES analyses, waste stream identification and FDA paper testing.

Project Chemist, Teck-Cominco Alaska, Inc. - Lynda has been project manager for work at their Red Dog Mine in Alaska for over ten years. She provides technical and regulatory interpretation assistance as well as project organization to their work sent to the laboratory.

Project Chemist and Department Manager, General and Water Chemistry and Biology Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibilities included the management of General and Water Chemistry, and the Biology Laboratory, which consisted of routine wastewater, bioassay, and microbiological analyses, supervision, data review, and reporting. 1989-1992.

Laboratory Analyst III - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibilities included coliform testing, total recoverable petroleum hydrocarbon extractions and analysis, BODs, ammonias, and Total Kjeldahl Nitrogen (TKN), in addition to miscellaneous wet chemistry. 1989.

Microbiologist/Chemist - Coffey Laboratories, Portland, Oregon. Coliform analysis; water chemistry. 1983.

Laboratory Assistant - Oregon State University, Corvallis, Oregon. Wheat spike dissection and tissue culture. 1983.

Education

Oregon State University, Corvallis, Oregon - B.S. in Microbiology. 1983.

JEFFREY A. CORONADO APRIL 1989-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position **Inorganics Department Manager. June 2001-Present.**

Responsibilities Oversees the operation of the inorganic groups which consists of two separate laboratories: Metals Laboratory and the General Chemistry Laboratory. Responsible for the quality and timeliness of the inorganic laboratory's analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation.

Experience

Metals Department Manager. October 1992-June 2001. Management duties included management of all aspects of the metals laboratory operation, including personnel training and evaluation, review of all metals data, and report generation. Also responsible for client service on a number of ongoing CAS accounts. Technical duties included primary analytical responsibility for trace level metals analysis by ICP-MS. Analyses ranged from routine water and soil analysis, to marine tissues, as well as industrial applications such as ultra-trace QA/QC work for various semiconductor clients. Also was responsible for a number of specialized sample preparation techniques including trace metals in seawater by reductive precipitation, and arsenic and selenium speciation by ion-exchange chromatography. Developed methodology for performing mercury analysis at low part per trillion levels by cold vapor atomic fluorescence.

Supervisor, GFAA Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Supervision of metals analysis by graphite furnace atomic absorption following SW-846 and EPA CLP methodologies. Duties include workload scheduling, data review, instrument maintenance, personnel training and evaluation. April 1989-October 1992.

Education

EnSys Inc. Field Immunoassay Training Course. 1995.

Winter Conference on Plasma Spectrochemistry, San Diego, CA 1994.

VG-Elemental ICP-MS Training Course. 1992.

Western Washington University, Bellingham, Washington - B.S. in Chemistry. 1988.
Western Washington University, Bellingham, Washington - B.A. in Business Administration. 1985.

JEFF A. GRINDSTAFF OCTOBER 1991-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position

Manager, GC/MS Volatile and Semivolatile Organics Laboratory. 1997-Present.

Responsibilities

Primary responsibilities include supervising GC/MS volatile and semivolatile organics staff, new method development, training, reviewing GC/MS volatile organics data, tracking department workload, scheduling and performance of volatile organics analyses, and general maintenance and troubleshooting of GC/MS systems.

Experience

Manager, GC/MS Volatile Organics Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibilities include supervising GC/MS volatile organics staff, new method development, training, reviewing GC/MS volatile organics data, tracking department workload, scheduling and performance of volatile organics analyses, and general maintenance and troubleshooting of GC/MS systems. 1994-1997.

Scientist III, GC/MS Volatile Organics Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. 1991-1994

Chemist - Enseco-CRL, Ventura California. Responsible for establishing GC/MS department, including maintenance of consumable inventory; preparing a state certification data package; and for developing and implementing analytical methods, SOPs, and extended data programs. Performed daily maintenance and troubleshooting of GC and GC/MS instrumentation. Scheduled and performed routine and nonroutine volatile organic analyses to meet holding times and due dates. 1990-1991.

GC/MS Chemist - Coast-to-Coast Analytical Services, San Luis Obispo, California. Prepared daily analytical standards for volatile organic analyses, and performed daily calibration and tuning of GC/MS instrumentation, along with general maintenance of hardware. Implemented and further developed EPA methods for quantitative analysis of pesticides and priority pollutants. 1988-1990.

Education

Mass Selective Detector Maintenance, Hewlett-Packard Education Center. December 1993.
Interpretation of Mass Spectra I, Hewlett-Packard Analytical Education Center. October 1992.
California Polytechnic State University, San Luis Obispo, California - BS in Chemistry. 1989.
Allan Hancock College, Santa Maria, California - AA in Liberal Arts. 1986.

Publications/ Presentations

Alternate Method to Lower Detection Limits to Satisfy Regulatory Action Levels for Volatiles in Groundwater, with David Edelman, Kairas Parvez, and Paul Laymon. TAPPI National Meeting, Orlando, Florida. 1996.

Affiliations

American Chemical Society. 1989.

TODD POYFAIR AUGUST 1991-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position

Manager, GC/HPLC Semivolatile Organics Laboratories - 2001-Present.

Responsibilities

Primary responsibilities include supervising GC, HLPC, and Fuels semivolatile organics laboratories and staff, oversee training, reviewing GC and HPLC data, tracking department workload, scheduling and performance of GC and HPLC analyses.

Experience

General Chemistry Department Supervisor/Manager - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibilities included the management, supervision, training, workload coordination, data review, reporting, and maintenance of the General Chemistry Laboratory. 1995-2001.

Project Chemist, Client Services Group - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibilities included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to act as liaison for all client-related activities within CAS. 1993-1995

Scientist II, General Chemistry Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Primary responsibilities include the review and summarization of pH, alkalinity, conductivity, turbidity, hardness, and CODs. 1992-1993

Scientist I, General Chemistry Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Responsible for analysis of Total Organic Halogens, Chemical Oxygen Demand, Sulfides, Ammonia, Total Kjeldahl Nitrogen, Nitrate/Nitrite by Lachat, and Cyanide. 1992

Analyst III, General Chemistry Laboratory - Columbia Analytical Services, Inc., Kelso, Washington. Responsible for the analysis of pH, Conductivity, Alkalinity, Turbidity, and Oil and Grease. 1991-1992

Education

Portland State University, Portland, Oregon - B.S. in Chemistry. 1991.
Portland State University, Portland, Oregon B.A. in German. 1990.
Brigham Young University, Provo, Utah. 1982-1983 & 1985-1986.

PAUL GOWAN JUNE 1994-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position **Technical Information Specialist. 1994-Present.**

Responsibilities Primary responsibilities include support automation of HP chemstation and Enviroquant; support continued development of unified organic laboratories; support and development of project-specific data deliverables; technical assistance in Information Technology at Kelso to meet CAS IT objectives as related to LIMS.

Experience

Organics Section Manager - Anametrix, Inc., San Jose, California. Managed the GC/MS and GC/Pesticide Departments, following protocols of the Department of Defense NEESA contracts (demanding stringent QA/QC and "Level D" data package submittals). Primary responsibilities included supervisor training and development, budget preparation and maintenance, performance reviews, data review, continuing research on environmental trends, Standard Operating Procedure generation and updates, method development, capital equipment evaluation for laboratories, project management. 1992-1994.

GC/MS Program Manager - Anametrix, Inc., San Jose, California. Supervised five chemists, two Finnigan 4000 GC/MS systems, and three HP 5971 GC/MS systems. Primary responsibilities were to maintain high productivity and insure that legally defensible data was generated by the GC/MS department, sample scheduling and tracking, instrument maintenance and troubleshooting, analyst training and review, client interfacing, purchasing of consumables. 1988-1992.

GC/MS Analyst - Anametrix, Inc., San Jose, California. Primary responsibilities included analyzing for volatile and semivolatile priority pollutants using EPA Methods 624/625 and 8240/8370. 1986-1988.

Education San Jose State University, San Jose, California - B.A. in Biochemistry. 1986.

EILEEN M. ARNOLD DECEMBER 1987-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position

Kelso Laboratory Health and Safety Officer, Scientist IV, Metals Laboratory. 1987-Present.

Responsibilities

Kelso Laboratory Health and Safety Officer: Responsible for the development and implementation of the CAS Health and Safety Program. Duties include accident investigation and incident review, and maintenance of all safety-related equipment and documents. Responsible for supervising the Laboratory Waste Management Plan. Chemical Hygiene Officer. 1987-Present.

Scientist IV, Metals Laboratory: Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. 1987-1992 and 1994-Present.

Experience

Project Chemist, Client Services Group - Columbia Analytical Services, Inc., Kelso, Washington. Duties included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to act as liaison for all client-related activities within Columbia Analytical Services, Inc. 1992- 1994.

Chemist - Dow Corning Corporation, Springfield, Oregon. ICP and atomic absorption work in silicon manufacturing. Methods development for ICP analysis of minor impurities found in silicon. 1986-1987.

Chemist - Ametek, Inc., Harleysville, Pennsylvania. Product research and development chemist involved in production of thin-film semiconductors for use as solar cells. Work involved AA and SEM techniques. 1982-1985.

Chemist - Janbridge, Inc., Philadelphia, Pennsylvania. Maintained electroplating process lines through wet chemical analysis techniques, and performed Quality Assurance testing on printed circuit boards. 1978-1982.

Education

Immaculata College, Immaculata, Pennsylvania - B.A. in Chemistry. 1977.

Affiliations

American Chemical Society. 1987.

DAVID L. EDELMAN APRIL 1988-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position **Chief Technical Officer for CAS Laboratory Network, Vice President and member of the Board of Directors for CAS Holdings,** - 2000 to present.

Responsibilities Responsible for facilitating implementation of new technology throughout the laboratory network. This includes establishing and implementing long-range objectives, plans, and policies. Major project through 2003 is implementation of state-of-the-art LIMS at all CAS locations. Responsibilities also include major customer problem solving, method development activities, and representing the company with its major customers, technical community, and the public.

Experience

Vice President-CAS, Corporate Technical Director, Information Technology Director, and East Coast Laboratories Director - Company Operations Committee Member, Member of Board of Directors. Technical Director and Senior Project Manager. Responsible for implementing new technology, TQM, and laboratory mergers. Responsible for the all operations in the Southeastern United States. 1992-2000

Laboratory Director - Columbia Analytical Services, Inc., Kelso, Washington. Responsible for the overall operation of the Kelso laboratory. 1991-1992.

Organics Laboratory Manager - Columbia Analytical Services, Inc., Kelso, Washington. Responsible for operating/managing the organic and bioassay laboratories to produce high quality, cost-effective, timely analytical services to comply with Federal RCRA, NPDES, CERCLA, SDWA, UST and State Regulatory requirements. 1988-1991.

Laboratory Manager - James River Corporation, Environmental Services. Responsible for managing laboratory operations, using GC/MS/DS, ICP, AA, TOC, GC/FID/ECD/TCD, UV/VIS, and bioassay techniques on effluents, sludges, wastes, ground water, and process streams. Responsible for obtaining accreditations from three state regulatory agencies. 1986-1988.

Laboratory Coordinator - Crown Zellerbach Corporation, Environmental Services. Performed/supervised analytical sampling services for RCRA Remedial Investigation/Feasibility Studies, NPDES permits, ground water contamination studies, and solid waste disposal site investigations. Performed GC/MS, HPLC, ICP, and wet chemistry techniques. Developed specialized skills for analyzing materials from pulp/paper/packing industry. 1979-1986.

Education

University of Washington, Seattle, Washington - Ph.D. in Forest Resources. 1979.
University of Washington, Seattle, Washington - M.S. in Organic Chemistry. 1976.
University of Washington, Seattle, Washington - B.S. in Chemistry. 1973.

Publications/ Presentations

Update on Effluent Guidelines Implementation, Challenges in Laboratory Certification. 2001 NCASI West Coast Regional Meeting, September 2001, Portland, OR

EPA Method 4425: Screening Method for Dioxin and Dioxin-like Compounds with Jack Anderson. American Chemical Society 222nd National Meeting, August 2001, Chicago, IL

Passive Diffusion Bag Samplers for Sampling Groundwater for Volatile Organic Compounds with Howard Boorse, Walt Scheible, Mark Wilson and Karen Daniels. ACS 222nd National Meeting, August 2001, Chicago, IL

Cost-Effective Dioxin Characterization using the P450 Reporter Gene System (RGS), with Bob Wilkinson, Mark Butler, and Jennifer Jones. 4th Annual Meeting of NAC Society of Environmental Toxicology and Chemistry, May 1998, Saratoga Springs, NY.

Cluster Rule Method Flexibility, Contracting for Analytical Data -Contract Lab Perspective, NCASI, Southern Regional Meeting, April 1997 and NCASI, Western Regional Meeting September, 1997.

Utilization of Lignin Degradation Compounds to Determine Groundwater Contamination from a Pulp and Paper Mill Landfill, with Daniel Diehl, Jeff Pickrell, and Craig Myers. TAPPI National Meeting, Minneapolis/St. Paul, Minnesota, 1997.

Alternate Method to Lower Detection Limits to Satisfy Regulatory Action Levels for Volatiles in Groundwater, with Kairas Parvez, Jeff Grindstaff, and Paul Laymon. TAPPI National Meeting, Orlando, Florida, 1996.

P450 RGS: A Biomarker for Assessing the Toxicity of Environmental Samples, with Jack Anderson, Kristen Bothner, and Steve Vincent. TAPPI National Meeting, Atlanta, Georgia, 1995.

*Investigation of Hydrocarbons Detected Downgradient from a Woodwaste Disposal Site.*with Kirk Girard, TAPPI National Meeting, Portland, Oregon 1994.

Hazardous Waste Analyses, The Pollution Prevention Network, 01/27/93.

Affiliations

American Chemical Society. 1976-Present. *Standard Methods* Committees Participation
Technical Association of the Pulp and Paper Industry. 1972-Present
Association of Official Analytical Chemists. 1979-Present.

LAWRENCE J. JACOBY AUGUST 1990-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue, Kelso, WA 98626 (360)577-7222

Current Position

Quality Assurance Director, Vice President. 1992-Present.

Responsibilities

Responsible for the conduct of the quality assurance (QA) program and activities for CAS; for conducting systems and data audits at each CAS laboratory to ensure that the QA objectives established by management and by the various certifications, accreditations, project plans, and regulations under which CAS operates are satisfactorily met, to ensure that the QA programs are functioning as stated in QA Manuals, and to make appropriate recommendations for corrective actions and improvements; for management of performance evaluation and round-robin samples analyses programs; for evaluating data quality; for maintaining CAS-wide standard operating procedures; for preparing quarterly QA reports to management; for facilitating quarterly ethics training; and for providing technical assistance to project chemists.

Experience

Quality Assurance Coordinator - Columbia Analytical Services, Inc., Kelso, Washington. Responsible for CAS/Kelso's quality assurance program and projects, and for evaluating data quality. 1990-1992.

Client Services Manager - CH_2M Hill Laboratory, Redding, California. Management of client services and sample custody groups; customer service and maintenance; project management; proposal and quotation preparation; project-engineer/laboratory liaison. 1989-1990.

Inorganic Division Manager - CH_2M Hill Laboratory, Redding, California. Responsible for managing the operation of the inorganic analyses section including wet chemical, soil, and metals analyses; project management; customer service; proposal and quotation preparation. 1988-1989.

Laboratory Manager - CH_2M Hill Laboratory, Corvallis, Oregon. Responsible for managing the operation of the laboratory and coordinating the activities of project support staff; project management; quality assurance; proposal and quotation preparation; laboratory safety; engineering project consulting. 1986-1988.

Analytical Chemist - Teledyne Wah Chang, Albany, Oregon. Responsibilities included methods development; instrument maintenance; non-routine analyses; workload scheduling and coordination; and task force assignments. 1976-1986.

Assistant Professor/Instructor - Portland State University, Portland, Oregon. 1969-1971. Chemeketa Community College, Salem, Oregon. 1971-1976. Taught college courses in general, organic and analytical chemistry.

Education

Colorado State University, Ft. Collins, Colorado - Ph.D. in Organic Chemistry. 1969.
Oregon State University, Corvallis, Oregon - B.S. in Chemistry. 1965.

Affiliations

American Society for Quality Control.
American Chemical Society.
AOAC International.

GARY WARD 2001-PRESENT

CAS Holdings Inc., 1 Mustard St., Suite 250, Canoga Park, CA 91303-3102 (360) 253-2210

Current Position

Chief Quality, Safety, & Ethics Officer - 2001 to present.

Responsibilities

Responsibilities include directing and managing the overall corporate-wide quality systems, and ethics and safety programs for all CAS facilities, as well as strategic planning, marketing and business development. Responsible for all interaction and liaison with government entities involving quality, technical and operational issues.

Experience

Deputy Director, Laboratory Standards – Intertek Testing Services, Houston, TX. Responsibilities included professional standards/quality assurance for 240 laboratories in 93 countries, involving laboratory tests ranging from petroleum products and environmental samples to toys, textiles, and building products. Resolution of issues with a variety of governments, agiences, and companies with particular focus on interactions with the US EPA. Was previously responsible for all operations of over 100 labs in the Americas, ranging from Canada to South America, including duties to improve quality, raise profits and revenues, and implement a LIMS. 1998-2001.

Director, Technical Operations – Environmental Health Laboratories, South Bend, IN. Responsibilities included operations and quality assurance of the laboratory. Directed, administered and coordinated activities of the lab in accordance with goals and objectives of the company. Responsible for the R&D program, laboratory throughput and financial performance, and implementation of the new LIMS system. 1995-1998.

Executive Scientist – Quanterra (Enseco), Arvada, CO. Responsibilities included providing expertise and experience in laboratory analysis and operations to the entire laboratory system. Duties included implementation of network-wide LIMS as well as coordination of the Technology, QA, IS, and Operations groups. As Director of Technology and Quality Assurance was responsible for management of the R&D program, Quality Assurance program, and Environment, Health and Safety program throughout the Enseco lab system. Direct reports were all QA managers, safety managers, and chief scientists from each of the 13 laboratories. 1987-1992.

Deputy Branch Chief – U.S. Environmental Protection Agency. Responsibilities included providing expertise to entire Superfund program ranging from lab analytical services to sampling. Duties involved managing the CLP program as well as the Superfund R&D program. As CLP National Program Manager was responsible for development and implementation of CLP analytical protocols, administration of contracts for over 100 laboratories throughout the country, and liasion with contract divisions, other EPA programs, and enforcement. Responsible for development and implementation of disk deliverables, automated contract screening, as well as writing new protocols for specific methods such as ICP/MS and for EPA methods such as included in SW846, 3rd Edition. Duties also included coordination of the annual CLP conferences. 1983-1987.

Education

M.S., Chemical Oceanography, RSMAS, University of Miami, FL 1973
B.S., Chemistry, Loyola University, LA 1970

Publications/ Presentations

Ward, G.K. *Laboratory Experience – Role of Auditing and Accreditation*, Invited Paper, US EPA 15th Annual Waste Testing and Quality Assurance (WTQA) Symposium, Washington, DC, July, 1999.

Ward, G.K., Wang, H., George III, J. and Thoma, J., *Cost Effective Analysis Of Semi-volatile Organic Compounds In Water By Micro-Solid Phase Extraction With Large Volume Injection GC/MS*, WQTC, Denver, CO, November, 1997.

Ward, G.K. *Innovative Broad Spectrum Tests for Screening Drinking Water*, Invited Speaker, Drinking Water Monitoring Meeting, Washington, DC, 1997.

Ward, G.K., George III, J., Wang, L., and Thoma, J. *Evaluation of Temperature and System Conditions on DBP Formation in Surface Water Using the Rapid Bench Scale Membrane Test for ICR Treatment Studies*, WQTC, Denver, CO, November, 1997.

Ward, G.K. *A Model for International Cooperation In Environmental Remediation Using Four New Cost-Effective Technologies*, Invited Speaker, Russian-United States International Symposium on Environmental Technologies for a Healthier World, March, 1997.

Ward, G.K., George III, J., Payne, G., and Conn, D. *Cost-Effective Low-Level Detection of Geosmin and MIB in Water by Large Volume Purge-and-Trap GC/MS*, WQTC, Denver, CO, November, 1997.

Ward, G.K. *Bench-Scale Treatment and ICR Studies, Taste & Odor Analyses, and Services for Validating Water Quality*, WQTC, Denver, CO, November, 1997.

Ward, G.K., Wang, H., George III, J., and Thoma, J. *Analysis of UltraTrace Level Dioxins in Drinking Water Using Large Volume Injection with GC/MS-MS*, Pittcon Book of Abstracts, 1997.

Ward, G.K., Brown, J., and Thoma, J. *Drinking Water Analysis: Method Performance and Detection Limits*, Environmental Testing and Analysis, 5: 13-17, 1996.

Over 100 additional publication references are available upon request.

Affiliations

ACS, ASTM, AAAS, IOBWA, TBWA, AWWA, A2LA

STEPHEN W. VINCENT JULY 1986-PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position

President, CAS Holdings Inc. 1986-Present.

Responsibilities

Responsible for the overall growth and profitability of the CAS laboratory network. This includes establishing and implementing long-range objectives, plans, and policies, and representing the company with its major customers, technical community, and the public.

Experience

Laboratory Manager - Weyerhaeuser Company, Federal Way, Washington. 1979-1986. Responsibilities: Involved all phases of technical and administrative management. This included management of organic, inorganic, and microbiological analyses and management of capital; an annual operating budget of approximately $2 million; management of thirty staff members; contract procurement, and project management. Projects included an EPA Inorganic CLP contract; an EPA acid rain deposition contract; a contract with the Fish and Wildlife Service to measure trace organic contaminants in animal tissues; and others.

Analytical Chemist - Weyerhaeuser Company, Longview, Washington. 1975-1979. Responsibilities: Method development, routine analysis and supervision for the Weyerhaeuser Multi-Region Support Lab. Responsible for setting up a company-wide laboratory audit, round robin, and quality assurance program.

Education

Stanford University - Executives Program. Market Strategy for Technology-Based Companies. 1994.

University of California at Los Angeles - Department of Business, Engineering and Management. Advanced Technical Management Program. 1991.

University of Washington, Seattle, Washington - Completion of course work for MS Pulp and Paper Technology. 1984.

University of California at Los Angeles - Graduate School of Engineering and Applied Science. Graduate School of Management. Engineering and Management Program. 1981.

University of Washington, Seattle, Washington - B.S. in Oceanography. 1974.

Publications/ Presentations

The State of the Environmental Testing Industry, ACIL Western Division 1995 Spring Meeting, Port Ludlow, Washington

Session Chairman, *New Low-Level Procedures and Field Techniques for the Assessment of Chemical Pollutants in the Environment*, HazMat West, November, 1992, Long Beach, California.

Changing Requirements for Chemical and Biological Analysis of Point Source Discharges, EMCON Geology Conference, Stanford University, Palo Alto, California. August 1989.

Laboratory Certification in the Northwest, Annual Conference, American Water Works Association, Eugene, Oregon. May 1989.

Basic Laboratory Skills, NCASI Central Lakes Meeting, Chicago, Illinois. 1982.

Weyerhaeuser Company's Corporate Quality Assurance Program, NCASI, New Orleans, Louisiana. June 1981.

The Impact of Pulp and Paper Effluents on the Water Quality of the Lower Columbia River, with W. G. Hines and S. R. Young. TAPPI Environmental Conference, New Orleans, Louisiana. April 1981.

Weyerhaeuser Company's Effluent Monitoring Program for Toxic Metals, National Council for Air and Stream Improvement, Portland, Oregon. 1977.

Affiliations

American Chemical Society.

Technical Association of the Pulp and Paper Industry.

APPENDIX C

MAJOR ANALYTICAL EQUIPMENT

MAJOR ANALYTICAL EQUIPMENT

GENERAL CHEMISTRY/WATER CHEMISTRY SAMPLE PREPARATION			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Digestion Systems (5): COD (4) Kjeldahl, Lachat 46-place (1)	 1987, 1989 1999	 LM LM	 8 5
Shatter Box - GP 1000	1989	LM	5
Thomas-Wiley Laboratory Mill, Model 4	1989	LM	7

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balances (9): Precisa and Mettler models	1988-2000	MM	15
Autoclave - Market Forge Sterilmatic	1988	LM	5
Calorimeter - Parr 1241 EA Adiabatic	1987	LM	5
Centrifuge - Damon/IEC Model K	1992	LM	15
Colony Counter - Quebec Darkfield	1988	LM	5
Conductivity Meters (2): Amber Science Model 604 YSI Model 34	 1987 1991	 LM LM	 8 8
Dissolved Oxygen Meter - YSI Model 58 (3)	1987, 1988, 1991	LM	8
Distillation apparatus (Midi) - Easy Still (2)	1996, 2000	LM	7
Drying Ovens (10): Shel-Lab and VWR models	1988 - 2000	LM	15
Flash Point Testers: ERDCO Setaflash Tester (2)	1988, 1991	LM	5
Flow-Injection Analyzer - Lachat Quick Chem AE	1990	LM	5
Ion Chromatographs (4) Dionex 2000i with Peaknet Data Systems (2) Dionex DX-120 with Peaknet Data System Dionex DX-100 with Peaknet Data System	 1988 1998 2000	 LM LM LM	 5 5 3

MAJOR ANALYTICAL EQUIPMENT

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY (continued)			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Ion Selective Electrode Meters (5)			
Fisher Scientific Accument Model 50	1997	LM	9
Fisher Scientific Accument Model 25	1993	LM	9
Fisher Scientific Accument Model 20	2000	LM	9
Orion Model 920A	1990	LM	9
Corning pH/ion Meter Model 135	1992	LM	9
Microscopes (3):			
Olympus BH-2	1987	LM	13
Bausch & Lomb	1988	LM	13
Swift	1988	LM	13
Muffle Furnaces- Sybron Thermolyne Model F-A1730	1991	LM	15
pH Meters (3):			
Beckman 34 (2)	1989	LM	9
Fisher Scientific Accument Model 20	1993	LM	9
Sieve Shakers (2):			
CE Tyler - Portable RX 24	1990	LM	5
WS Tyler - RX 86	1991	LM	5
Total Organic Carbon (TOC) Analyzers (3)			
O-I Corporation, Model 700 (2)	1986, 1993	LM	3
Coulemetrics Model 5012	1997	LM	5
Total Organic Halogen (TOX) Analyzers (4):			
Mitsubishi (MCI) TOX-10	1986	LM	5
Mitsubishi TOX-Sigma	1995	LM	5
Mitsubishi TOX-100 (2)	2001	LM	4
Turbidimeter - Hach Model 2100N	1996	LM	8
UV-Visible Spectrophotometers (2):			
Hitachi 100-40 Single Beam	1986	LM	10
Milton Roy 1001 Plus	1991	LM	10
Vacuum Pumps (2):			
Welch Duo-Seal Model 1376	1990	LM	13
Busch R-5 Series Single Stage	1991	LM	13
Water Baths/Incubators (6):			
Hach Model 15320 Incubator	1986	LM	6
Precision Model L-6 (2)	1989, 1990	LM	6
VWR 1540	1991	LM	6
Fisher 11-680-626M Incubator	1992	LM	6
Fisher Isotemp Incubator	2001	LM	6

MAJOR ANALYTICAL EQUIPMENT

METALS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (6)			
Various Mettler AE 200 analytical balance	1990	MM	12
Various Mettler models (5)	1988	MM	12
Atomic Absorption Spectrophotometers (6):			
Varian SpectrAA 300 Zeeman AA and IBM Data Stations (2)	1989	LM	3
Varian SpectrAA Zeeman/220 AA w/Data Systems (2)	2000	MM	3
Varian SpectrAA 20 with Flame, Cold Vapor, and Hydride Systems			
CETAC Mercury Analyzer	1988	LM	4
	2000	LM	3
Atomic Fluorescence Spectrophotometer - Brook-Rand Model III	1996	LM	4
Centrifuge - IEC Model Clinical Centrifuge	1990	LM	12
Drying Oven - VWR Model 1370F	1990	LM	12
TCLP Extractors (2)	1989	LM	6
Freeze Dryers (2) - Labconco	1988, 1992	LM	5
Muffle Furnace - Thermolyne Furnatrol Model 53600	1991	LM	5
Shaker - Burrell Wrist Action Model 75	1990	LM	12

ICP LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) - Thermo Jarrell Ash Model 61E	1988	LM	5
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES): Thermo Jarrell Ash, Model IRIS	2000	MM	3
Inductively Coupled Plasma Mass Spectrometer (ICP-MS): VG PQ-S	1997	MM	4
Inductively Coupled Plasma Mass Spectrometer (ICP-MS): VG Excell	2001	MM	4

MAJOR ANALYTICAL EQUIPMENT

GC SEMIVOLATILE ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
SAMPLE PREPARATION			
Accelerated Solvent Extractor - Dionex ASE 200	1996	LM	5
Analytical Balance (2) Mettler BB240 Satorious B610	 1987 1999	 MM MM	 7 7
Aspirator pumps - Labconco Cole Parmer (1)	1994	LM	7
Centrifuges (2): Adams Model DYNAC Sorvall Model GLC-1	 1986 1988	 LM LM	 7 7
Drying Oven - Fisher Model 655 G	1991	LM	7
Evaporators (6): Organomation N-Evap (3) Organomation S-Evap (3)	 1989,1998, 2001 1989-1991	 LM LM	 7 7
Extractors: Lab-Line Multi-Unit Extraction Heaters (60)	1987-1992	LM	7
ABC GPC - single column (2)	1998, 1999	LM	4
Muffle Furnace - Parflow MIC 6000	1994	LM	7
Sonic Water Bath - Branson Model 8200	1991	LM	7
Vacuum Pump - Edwards	1992	LM	7
INSTRUMENTATION			
Analytical Balance - Mettler AT 250	1989	MM	8
Chromatography Data Systems (12) HP Enviroquant (8) Thruput Target (4)	 1994-2002 1998-2000	 LM LM	 4 3
Gas Chromatographs (12): Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual NPD Detectors Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual ECD Detectors (7) Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FPD Detectors Varian 3400 with Autosampler &Dual ECD Detector Agilent 6890 GC with Agilent 7683 Autosampler and Dual ECD Detectors (2)	 1989 1990 – 1995 1991 1999 2001	 LM LM LM LM LM	 3 7 4 2 2

MAJOR ANALYTICAL EQUIPMENT

PETROLEUM HYDROCARBONS GC LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
SAMPLE PREPARATION			
Analytical Balance (3)			
Mettler BB300	1991	MM	7
Mettler BB240	1994	MM	7
Mettler AE166	1994	MM	7
Aspirator pumps - Labconco Cole Parmer (1)	1994	LM	7
Drying Oven (2) - Fisher Models 630F & 655 G	1991	LM	7
Evaporators (2):			
Organomation N-Evap	1990	LM	6
Organomation S-Evap	1991	LM	6
Extractors (21):			
Lab-Line Multi-Unit Soxhlet Extraction Heaters (18)	1987-1992	LM	6
Sonifier (Sonic Horn) (3):			
Branson	1994	LM	6
Ultrasonics	1994	LM	6
Fisher Model 550	1991	LM	6
INSTRUMENTATION			
HP Enviroquant Chromatography Data Systems (10)	1994-2002	LM	6
Gas Chromatographs (9):			
Varian 3300 with PID/FID detectors	1989	LM	3
O-I 4460A Purge and Trap Concentrator	1989	LM	3
Dynatech PTA-30 Autosampler	1996	LM	3
Hewlett-Packard 5890 Series II with PID/ PID/FID det.	1991	LM	3
Tekmar LSC-2000 Purge and Trap Concentrator	1991	LM	3
Dynatech Archon 5100 Autosampler	1991	LM	3
Hewlett-Packard 5890 Series II with PID/FID det.	1992	LM	3
Tekmar LSC-2000 Purge and Trap Concentrator	1988	LM	3
Dynatech PTA-30 Autosampler	1989	LM	3
Hewlett-Packard 5890 Series II with PID/PID/FID det.	1999	LM	3
O-I 4560A Purge and Trap Concentrator	1999	LM	3
Dynatech Archon 5100 Autosampler	1999	LM	3
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FID Detectors (3)	1990 - 1995	LM	5
Hewlett-Packard 5890 GC with HP 7673 Autosampler and FID/ECD Detectors	1994	LM	5
Agilent 6890 with Agilent 7873 Autosampler and Dual FID Detectors	2001	LM	5
High-Performance Liquid Chromatographs (2):			
HP 1090M Series II with HP 1046A Fluorescence Detector & Diode Array UV Detector (2)	1995, 1999	LM	3

MAJOR ANALYTICAL EQUIPMENT

GC/MS SEMIVOLATILE ORGANICS SAMPLE PREPARATION			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balances (2)			
Mettler BB300	1991	MM	6
Sartorius 610	2000	MM	6
Evaporators (4):			
Organomation N-Evap (2)	1989-1990, 2000	LM	5
Organomation S-Evap (2)	1990-1991	LM	5
Extractors (64):			
Continuous Liquid/Liquid Extractors (24)	1991	LM	5
Branson Model 450 Sonifier (2)	1991	LM	5
Tekmar Sonifier (2)	1994	LM	5
Soxhtherm (36)	2000	LM	3
GPC-ABC Model Autoprep 1000	1995	LM	4
Gas Chromatograph: Hewlett-Packard 5890 with HP 7673 autosampler and FID Detector	1994	LM	5

GC/MS SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
HP Enviroquant Chromatography Data Systems (9)	1994-2002	LM	8
Semivolatile GC/MS Systems (7):			
Hewlett-Packard 6890/5973 with HP 6890 Autosampler (2)	1997, 2001	MM	6
Hewlett-Packard 5890/5970 with HP 7673 Autosampler (2)	1990,1994	MM	6
Hewlett-Packard 5890/5972 with HP 7673 Autosampler (3)	1993, 1994, 1998	MM	6

MAJOR ANALYTICAL EQUIPMENT

VOLATILE ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balances (2)			
Mettler PE 160	1989	MM	8
Mettler BB300	1993	MM	8
Baxter Vortex Mixer	1989	LM	8
Extractors (10):			
Millipore TCLP Zero Headspace Extractors (10)	1987-1992	LM	2
TCLP Extractor - Tumbler (12 position)	1989	LM	2
HP Enviroquant Chromatography Data Systems (10)	1994-2002	LM	8
Drying Ovens (2):			
Narco 420	1989	LM	8
VWR 1305 U	1991	LM	8
Sonic Water Bath - Branson Model 2200	1989	LM	8
Volatile GC/MS Systems (5):			
Hewlett-Packard 5890/5970	1989	MM	6
Tekmar 3000 Purge and Trap Concentrator	1995	LM	6
Dynatech ARCHON 5100 Autosampler	1996	LM	6
Hewlett-Packard 5890/5970	1999	MM	6
Tekmar 3000 Purge and Trap Concentrator	2001	LM	6
Dynatech ARCHON 5100 Autosampler	1999	LM	6
Hewlett-Packard 5890/5971	1991	MM	6
Tekmar 3000 Purge and Trap Concentrator	2001	LM	6
Dynatech ARCHON 5100 Autosampler	1995	LM	6
Hewlett-Packard 5890/5972A	1993	MM	6
Tekmar 3000 Purge and Trap Concentrator	1995	LM	6
Dynatech ARCHON 5100 Autosampler	1996	LM	6
Hewlett-Packard 6890/5973	2001	MM	6
Tekmar 3100 Purge and Trap Concentrator	2001	LM	6
Varian Archon Autosampler	2001	LM	6

MAJOR ANALYTICAL EQUIPMENT

AUTOMATED DATA PROCESSING EQUIPMENT			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
1-WAN: LIMs Sample Manager using ORACLE DBMS running on SEQUENT UNIX/DYNIX platform connected/linked on a frame relay WAN environment.	1994-1995	LM	NA
3- Network Servers Pentium/Pentium II class. 1 for Administration, 1 for Data Acquisition, and 1 for Applications. Network operating system: Microsoft Windows2000®. Data acquisition capacity at 24GB with redundant tape and disk arrays.	1994-2000	LM	NA
Appoximately 40-HP class Laserjet printers (various types from IIIs to SI Ivs and 8150s) linked via LAN.	1991-2002	LM	NA
Approximately 115 HP/Gateway/Dell PC/Workstations on LAN hooked up with 10BT/100BT and TCP/IP for LIMs Terminal Emulation.	1993-2002	LM	NA
Microsoft Office Small Business Edition as the base application for all PC/Workstations.	1994-2000	LM	NA
E-Mail with link to SMTP for internal/external messaging. Web mail via Cobalt Qube interface. Microsoft Outlook® 98 and 2000.	1994-2001	LM	NA
Standard EXCEL® reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1994-1996	LM	NA
Facsimile Machines 9600-14400 Baud, 11 Pages/Minute (4)	1991-1998	LM	NA
Dot Matrix NLQ (3)	1991-1992	LM	NA
Thruput, MARRS, Stealth, Harold reporting software systems.	1998-2001	LM	NA

NA: Not applicable. This equipment administered by IT staff but may be used by all staff.

APPENDIX D

DATA QUALITY CAPABILITIES

GENERAL CHEMISTRY/WATER CHEMISTRY ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC**
110.2	Color	Water	NA	NA	20	NA
120.1 / *SM* 2510B	Conductivity	Water	2 umhos/cm	0.6 umhos/cm	20	NA
130.2 / *SM* 2340C	Hardness as $CaCO_3$	Water	10 mg/L	3 mg/L	20	NA
150.1 / *SM* 4500-H^+B	pH	Water	NA	NA	NA	NA
160.1 / *SM* 2540C	Solids, Total Dissolved (Filterable)	Water	5 mg/L	5 mg/L	20	NA
160.2 / *SM* 2540D	Solids, Total Suspended (Nonfilterable)	Water	5 mg/L	5 mg/L	20	NA
160.3 / *SM* 2540B	Solids, Total	Soil	NA (%)	NA (%)	20	NA
		Water	5 mg/L	5 mg/L	20	NA
160.4 / *SM* 2540E	Solids, Volatile	Soil	NA (%)	NA (%)	20	NA
		Water	5 mg/L	5 mg/L	20	NA
160.5 / *SM* 2540F	Solids, Settleable	Water	0.1 ml/L	NA	20	NA
180.1 / *SM* 2130B	Turbidity	Water	0.1 NTU	0.06 NTU	20	NA
300.0 / *SM* 4110B Ion Chromatography (After Extraction)	Bromide	Soil	2 mg/Kg		20	90-110[d]
	Chloride		2 mg/Kg		20	90-110[d]
	Fluoride		2 mg/Kg		20	90-110[d]
	Nitrate as Nitrogen		1 mg/Kg		20	90-110[d]
	Nitrite as Nitrogen		1 mg/Kg		20	90-110[d]
	Sulfate		2 mg/Kg		20	90-110[d]
300.0 / *SM* 4110B Ion Chromatography	Bromide	Water	0.2 mg/L	0.09 mg/L	20	90-110[d]
	Chloride		0.2 mg/L	0.06 mg/L	20	90-110[d]
	Fluoride		0.2 mg/L	0.009 mg/L	20	90-110[d]
	Nitrate as Nitrogen		0.1 mg/L	0.007 mg/L	20	90-110[d]
	Nitrite as Nitrogen		0.1 mg/L	0.007 mg/L	20	90-110[d]
	Sulfate		0.2 mg/L	0.02 mg/L	20	90-110[d]
305.2 / *SM* 2310B	Acidity as $CaCO_3$	Water	NA (mg/L)	NA (mg/L)	20	NA
310.1 / *SM* 2320B	Alkalinity as $CaCO_3$	Water	2 mg/L	0.6 mg/L	20	NA
325.3 / *SM* 4500-Cl^-B	Chloride, Titrimetric	Water	0.2 mg/L	0.2 mg/L	20	85-115
330.4 / *SM* 4500-Cl-F	Chlorine, Total Residual	Water	0.1 mg/L	0.06 mg/L	20	85-115
335.1 / *SM* 4500-CN^-G	Cyanides Amenable to Chlorination	Soil	0.2 mg/Kg		20	85-115[b]
		Water	0.01 mg/L	0.003 mg/L	20	85-115[b]
335.2 / *SM* 4500-CN^-E	Cyanide, Total	Soil	0.2 mg/Kg		20	85-115[b]
		Water	0.01 mg/L	0.003 mg/L	20	85-115[b]
SM 4500-CN^-I	Cyanide, Weak Acid Dissociable	Soil	0.2 mg/Kg		20	85-115[b]
		Water	0.01 mg/L	0.003 mg/L	20	85-115[b]
340.1 / *SM* 4500-F^-B	Fluoride, Bellack Distillation	Soil	1 mg/Kg		20	85-115[b]
		Water	1.0 mg/L	0.4 mg/L	20	85-115[b]
340.2 / *SM* 4500-F^-C	Fluoride	Soil	2 mg/Kg		20	85-115[b]
		Water	0.2 mg/L	0.005 mg/L	20	85-115[b]
350.3 / *SM* 4500-NH_3F	Ammonia as Nitrogen	Soil	0.2 mg/Kg		20	85-115[b]
		Water	0.05 mg/L	0.03 mg/L	20	85-115[b]
351.4	Nitrogen, Total Kjeldahl	Soil	20 mg/Kg	6 mg/kg	20	85-115[b]
		Water	0.1 mg/L	0.07 mg/L	20	85-115[b]

GENERAL CHEMISTRY/WATER CHEMISTRY ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
353.2 / SM 4500-NO_3^--F	Nitrogen, Nitrate + Nitrite as Nitrogen	Soil	1 mg/Kg		20	85-115[b]
		Water	0.2 mg/L	0.02 mg/L	20	85-115[b]
354.1 / *SM* 4500-NO_2^- - B	Nitrite as Nitrogen, Colorimetric	Water	0.01 mg/L	0.004 mg/L	20	85-115[b]
365.3 / *SM* 4500-PE	Orthophosphate as Phosphorus	Soil	(e) mg/Kg		20	85-115[b]
		Water	0.01 mg/L	0.003 mg/L	20	85-115[b]
365.3M 365.3 / *SM* 4500-PE	Phosphorus, Total	Soil	0.2 mg/Kg		20	85-115[b]
		Water	0.01 mg/L	0.002 mg/L	20	85-115[b]
376.1 / *SM* 4500-S^{2-}D	Sulfide	Water	2 mg/L	0.2 mg/L	20	85-115[b]
377.1 / *SM* 4500-SO_3^{2-}B	Sulfite	Water	2 mg/L	0.3 mg/L	20	NA
SM 5550B	Tannin and Lignin	Water	0.2 mg/L	0.02 mg/L	20	85-115[b]
405.1 / *SM* 5210B	Biological Oxygen Demand	Water	4 mg/L	0.6 mg/L	20	NA
410.1 and 410.2 / *SM* 5220C	Chemical Oxygen Demand	Soil	50 mg/Kg		20	85-115[b]
		Water	5 mg/L	3 mg/L	20	85-115[b]
ASTM D4129-82M 415.1	Total Organic Carbon	Soil	0.05%	0.02%	20	85-115[b]
		Water	0.5 mg/L	0.09 mg/L	20	85-115[b]
420.1 / *SM* 5530C	Phenolics, Total	Soil	0.5 mg/Kg		20	85-115[b]
		Water	0.01 mg/L	0.004 mg/L	20	85-115[b]
425.1 / *SM* 5540C	Surfactants (MBAS)	Water	0.05 mg/L	0.02 mg/L	20	75-125[b]
ASTM D1498	Oxidation-Reduction Potential	Water	NA	NA	20	NA
1010	Flashpoint, Pensky-Marten	Soil	NA	NA		
		Water	NA	NA	20	NA
1020	Flashpoint, Setaflash	Soil	NA	NA		
		Water	NA	NA	20	NA
1110	Corrosivity	Liquid	NA	NA	20	NA
1650A/B/C	Absorbable Organic Halides	Water	10 ug/L	5 ug/L	20	71-116[d]
3060A/7196A 7196A	Hexavalent Chromium	Soil	0.5 mg/Kg	0.2 mg/Kg	20	85-115[b]
		Water	0.05 mg/L	0.008 mg/L	20	85-115[b]
9010A	Cyanide, Total and Amenable	Soil	0.2 mg/Kg		20	85-115[b]
		Water	0.01 mg/L	0.003 mg/L	20	85-115[b]
9020	Total Organic Halides	Water	10 ug/L	5 ug/L	20	83-117
9030	Sulfides	Water	0.1 mg/L	0.05 mg/L	20	60-130[b]
9030M	Sulfides	Soil	0.1 mg/kg	0.05 mg/kg	20	60-130[b]
9040	Corrosivity (pH)	Water	NA	NA	20	NA
9045C	pH	Soil	NA	NA	20	NA
9050	Specific Conductance	Water	2 mg/L	0.5 mg/L	20	NA
9060A	Total Organic Carbon	Water	0.5 mg/L	0.09 mg/L	20	85-115[b]
9066	Phenolics, Total	Soil	0.5 mg/Kg		20	85-115[b]
		Water	0.01 mg/L	0.004 mg/L	20	85-115[b]
9095	Paint Filter Test	Soil	NA	NA	NA	NA
9252	Chloride, Titrimetric	Water	0.2 mg/L	0.2 mg/L	20	85-115[b]

METALS ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC
200.7 (ICP)	Aluminum	Water	50 ug/L	30 ug/L	20	85-115
	Antimony		50 ug/L	30 ug/L	20	85-115[d]
	Barium		5 ug/L	2 ug/L	20	85-115[d]
	Beryllium		5 ug/L	1 ug/L	20	85-115[d]
	Boron		50 ug/L	20 ug/L	20	85-115[d]
	Cadmium		5 ug/L	2 ug/L	20	85-115[d]
	Calcium		50 ug/L	20 ug/L	20	85-115[d]
	Chromium		5 ug/L	3 ug/L	20	85-115[d]
	Cobalt		10 ug/L	4 ug/L	20	85-115[d]
	Copper		10 ug/L	8 ug/L	20	85-115[d]
	Iron		20 ug/L	20 ug/L	20	85-115[d]
	Magnesium		20 ug/L	20 ug/L	20	85-115[d]
	Manganese		5 ug/L	1 ug/L	20	85-115[d]
	Molybdenum		10 ug/L	5 ug/L	20	85-115[d]
	Nickel		20 ug/L	20 ug/L	20	85-115[d]
	Potassium		2000 ug/L	400 ug/L	20	85-115[d]
	Silver		10 ug/L	7 ug/L	20	85-115[d]
	Sodium		100 ug/L	40 ug/L	20	85-115[d]
	Tin		50 ug/L	20 ug/L	20	85-115[d]
	Vanadium		10 ug/L	6 ug/L	20	85-115[d]
	Zinc		10 ug/L	2 ug/L	20	85-115[d]
206.2/200.9[f] (GFAA)	Arsenic		5 ug/L	1 ug/L	20	85-115
239.2/200.9[f] (GFAA)	Lead		2 ug/L	1 ug/L	20	85-115[d]
245.1 (CVAA)	Mercury		0.2 ug/L	0.1 ug/L	20	85-115[d]
1631	Mercury		1 ng/L	0.1 ng/L	20	71-125[d]
270.2/200.9[f] (GFAA)	Selenium		5 ug/L	1 ug/L	20	85-115[d]
279.2/200.9[f] (GFAA)	Thallium		5 ug/L	1 ug/L	20	85-115[d]
200.8 (ICP/MS)	Aluminum	Soil/Sed.	2.0 mg/kg	2 mg/kg	30	70-130[m]
	Antimony		0.05 mg/kg	0.05 mg/kg	30	70-130[m]
	Arsenic		0.5 mg/kg	0.1 mg/kg	30	70-130[m]
	Barium		0.02 mg/kg	0.01 mg/kg	30	70-130[m]
	Beryllium		0.02 mg/kg	0.01 mg/kg	30	70-130[m]
	Cadmium		0.05 mg/kg	0.02 mg/kg	30	70-130[m]
	Chromium		0.2 mg/kg	0.03 mg/kg	30	70-130[m]
	Cobalt		0.02 mg/kg	0.01 mg/kg	30	70-130[m]
	Copper		0.1 mg/kg	0.04 mg/kg	30	70-130[m]
	Lead		0.05 mg/kg	0.03 mg/kg	30	70-130[m]
	Manganese		0.02 mg/kg	0.01 mg/kg	30	70-130[m]
	Molybdenum		0.02 mg/kg	0.01 mg/kg	30	70-130[m]
	Nickel		0.2 mg/kg	0.02 mg/kg	30	70-130[m]
	Selenium		1.0 mg/kg	0.2 mg/kg	30	70-130[m]
	Silver		0.02 mg/kg	0.01 mg/kg	30	70-130[m]

METALS ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
200.8 (ICP/MS)	Thallium	Soil	0.02 mg/kg	0.004 mg/kg	30	70-130[m]
	Vanadium		0.2 mg/kg	0.02 mg/kg	30	70-130[m]
	Zinc		0.5 mg/kg	0.1 mg/kg	30	70-130[m]
200.8 (ICP/MS)	Aluminum	Water	1.0 ug/L	0.6 ug/L	20	85-115[d]
	Antimony		0.05 ug/L	0.02 ug/L	20	85-115[d]
	Arsenic		0.5 ug/L	0.2 ug/L	20	85-115[d]
	Barium		0.02 ug/L	0.02 ug/L	20	85-115[d]
	Beryllium		0.02 ug/L	0.006 ug/L	20	85-115[d]
	Cadmium		0.05 ug/L	0.05 ug/L	20	85-115[d]
	Chromium		0.2 ug/L	0.05 ug/L	20	85-115[d]
	Cobalt		0.02 ug/L	0.005 ug/L	20	85-115[d]
	Copper		0.1 ug/L	0.05 ug/L	20	85-115[d]
	Lead		0.02 ug/L	0.006 ug/L	20	85-115[d]
	Manganese		0.05 ug/L	0.05 ug/L	20	85-115[d]
	Molybdenum		0.02 ug/L	0.009 ug/L	20	85-115[d]
	Nickel		0.2 ug/L	0.03 ug/L	20	85-115[d]
	Selenium		1.0 ug/L	0.8 ug/L	20	85-115[d]
	Silver		0.02 ug/L	0.007 ug/L	20	85-115[d]
	Thallium		0.02 ug/L	0.004 ug/L	20	85-115[d]
	Vanadium		0.2 ug/L	0.05 ug/L	20	85-115[d]
	Zinc		0.5 ug/L	0.08 ug/L	20	85-115[d]
6010B (ICP)	Aluminum	Soil	10 mg/Kg	7 mg/Kg	30	75-125[m]
	Antimony		10 mg/Kg	5 mg/Kg	30	75-125[m]
	Barium		1 mg/Kg	0.2 mg/Kg	30	75-125[m]
	Beryllium		1 mg/Kg	0.2 mg/Kg	30	75-125[m]
	Boron		10 mg/Kg	2 mg/Kg	30	75-125[m]
	Cadmium		1 mg/Kg	0.4 mg/Kg	30	75-125[m]
	Calcium		10 mg/Kg	4 mg/Kg	30	75-125[m]
	Chromium		2 mg/Kg	0.9 mg/Kg	30	75-125[m]
	Cobalt		2 mg/Kg	0.6 mg/Kg	30	75-125[m]
	Copper		2 mg/Kg	0.9 mg/Kg	30	75-125[m]
	Iron		4 mg/Kg	1 mg/Kg	30	75-125[m]
	Lead		20 mg/Kg	5 mg/Kg	30	75-125[m]
	Magnesium		4 mg/Kg	2 mg/Kg	30	75-125[m]
	Manganese		1 mg/Kg	0.2 mg/Kg	30	75-125[m]
	Molybdenum		2 mg/Kg	0.6 mg/Kg	30	75-125[m]
	Nickel		4 mg/Kg	2 mg/Kg	30	75-125[m]
	Potassium		400 mg/Kg	200 mg/Kg	30	75-125[m]
	Silver		2 mg/Kg	0.8 mg/Kg	30	75-125[m]
	Sodium		20 mg/Kg	4 mg/Kg	30	75-125[m]
	Tin		10 mg/Kg	5 mg/Kg	30	75-125[m]
	Vanadium		2 mg/Kg	2 mg/Kg	30	75-125[m]
	Zinc		2 mg/Kg	0.6 mg/Kg	30	75-125[m]

METALS ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%RE
7060A (GFAA)	Arsenic	Soil	1 mg/Kg	0.2 mg/Kg	30	60-130[m]
7421 (GFAA)	Lead		1 mg/Kg	0.2 mg/Kg	30	60-130[m]
7471A (CVAA)	Mercury		0.02 mg/Kg	0.01 mg/Kg	30	60-130[m]
7740 (GFAA)	Selenium		1 mg/Kg	0.7 mg/Kg	30	60-130[m]
7841 (GFAA)	Thallium		1 mg/Kg	0.2 mg/Kg	30	60-130[m]
6010B (ICP)	Aluminum	Water	50 ug/L	30 ug/L	20	92-108
	Antimony		50 ug/L	30 ug/L	20	91-112
	Barium		5 ug/L	2 ug/L	20	77-127
	Beryllium		5 ug/L	1 ug/L	20	91-111
	Boron		50 ug/L	20 ug/L	20	85-115[d]
	Cadmium		5 ug/L	2 ug/L	20	90-112
	Calcium		50 ug/L	20 ug/L	20	90-110
	Chromium		5 ug/L	3 ug/L	20	93-111
	Cobalt		10 ug/L	4 ug/L	20	92-110
	Copper		10 ug/L	8 ug/L	20	92-109
	Iron		20 ug/L	20 ug/L	20	92-110
	Magnesium		20 ug/L	20 ug/L	20	89-108
	Manganese		5 ug/L	1 ug/L	20	91-109
	Molybdenum		10 ug/L	5 ug/L	20	85-115[d]
	Nickel		20 ug/L	20 ug/L	20	91-112
	Potassium		2000 ug/L	400 ug/L	20	88-11
	Silver		10 ug/L	7 ug/L	20	89-107
	Sodium		100 ug/L	40 ug/L	20	91-114
	Tin		50 ug/L	20 ug/L	20	85-115[d]
	Vanadium		10 ug/L	6 ug/L	20	93-111
	Zinc		10 ug/L	2 ug/L	20	91-111
7060A (GFAA)	Arsenic	Water	5 ug/L	1 ug/L	20	82-116
7421 (GFAA)	Lead		2 ug/L	1 ug/L	20	84-111
7470A (CVAA)	Mercury		0.2 ug/L	0.1 ug/L	20	83-108
7740 (GFAA)	Selenium		5 ug/L	1 ug/L	20	78-112
7841 (GFAA)	Thallium		5 ug/L	1 ug/L	20	86-114
6020 (ICP/MS)	Aluminum	Soil/Sed.	2.0 mg/kg	2 mg/kg	30	70-130[m]
	Antimony		0.05 mg/kg	0.05 mg/kg	30	70-130[m]
	Arsenic		0.5 mg/kg	0.1 mg/kg	30	70-130[m]
	Barium		0.02 mg/kg	0.01 mg/kg	30	70-130[m]
	Beryllium		0.02 mg/kg	0.01 mg/kg	30	70-130[m]
	Cadmium		0.05 mg/kg	0.02 mg/kg	30	70-130[m]
	Chromium		0.2 mg/kg	0.03 mg/kg	30	70-130[m]
	Cobalt		0.02 mg/kg	0.01 mg/kg	30	70-130[m]
	Copper		0.1 mg/kg	0.04 mg/kg	30	70-130[m]
	Lead		0.05 mg/kg	0.03 mg/kg	30	70-130[m]
	Manganese		0.02 mg/kg	0.01 mg/kg	30	70-130[m]
	Molybdenum		0.02 mg/kg	0.01 mg/kg	30	70-130

METALS ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC)**
6020 (ICP/MS)	Nickel	Soil/Sed.	0.2 mg/kg	0.02 mg/kg	30	70-130[m]
	Selenium		1.0 mg/kg	0.2 mg/kg	30	70-130[m]
	Silver		0.02 mg/kg	0.01 mg/kg	30	70-130[m]
	Thallium		0.02 mg/kg	0.004 mg/kg	30	70-130[m]
	Vanadium		0.2 mg/kg	0.02 mg/kg	30	70-130[m]
	Zinc		0.5 mg/kg	0.1 mg/kg	30	70-130[m]
6020 (ICP/MS)	Aluminum	Water	1.0 ug/L	0.6 ug/L	20	85-115[d]
	Antimony		0.05 ug/L	0.02 ug/L	20	85-115[d]
	Arsenic		0.5 ug/L	0.2 ug/L	20	85-115[d]
	Barium		0.02 ug/L	0.02 ug/L	20	85-115[d]
	Beryllium		0.02 ug/L	0.006 ug/L	20	85-115[d]
	Cadmium		0.05 ug/L	0.05 ug/L	20	85-115[d]
	Chromium		0.2 ug/L	0.05 ug/L	20	85-115[d]
	Cobalt		0.02 ug/L	0.005 ug/L	20	85-115[d]
	Copper		0.1 ug/L	0.05 ug/L	20	85-115[d]
	Lead		0.02 ug/L	0.006 ug/L	20	85-115[d]
	Manganese		0.05 ug/L	0.05 ug/L	20	85-115[d]
	Molybdenum		0.02 ug/L	0.009 ug/L	20	85-115[d]
	Nickel		0.2 ug/L	0.03 ug/L	20	85-115[d]
	Selenium		1.0 ug/L	0.8 ug/L	20	85-115[d]
	Silver		0.02 ug/L	0.007 ug/L	20	85-115[d]
	Thallium		0.02 ug/L	0.004 ug/L	20	85-115[d]
	Vanadium		0.2 ug/L	0.05 ug/L	20	85-115[d]
	Zinc		0.5 ug/L	0.08 ug/L	20	85-115[d]

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC
1664	Hexane Extractable Material	Water	5 mg/L	1.2 mg/L	18	78-114[d]
1664	Hexane Extractable Material - SGT	Water	5 mg/L	0.86 mg/L	34	64-132[d]
9071A/1664	Hexane Extractable Material	Soil	100 mg/Kg	50 mg/Kg	40	78-114[d]
9071A/1664	Hexane Extractable Material - SGT	Soil	100 mg/Kg	40 mg/Kg	40	64-132[d]
600/4-81-045 PCBs	Aroclor 1016	Oil	1 mg/Kg		40	26-106
	Aroclor 1221		2 mg/Kg		40	
	Aroclor 1232		1 mg/Kg		40	
	Aroclor 1242		1 mg/Kg		40	
	Aroclor 1248		1 mg/Kg		40	
	Aroclor 1254		1 mg/Kg		40	
	Aroclor 1260		1 mg/Kg		40	39-116
	Decachlorobiphenyl[g]		NA	NA	NA	34-125
608 Chlorinated Pesticides and PCBs	alpha-BHC	Water	0.02 ug/L	0.001 ug/L	30	
	gamma-BHC (Lindane)		0.02 ug/L	0.002 ug/L	30	65-118
	beta-BHC		0.02 ug/L	0.001 ug/L	30	
	Heptachlor		0.02 ug/L	0.004 ug/L	30	50-119
	delta-BHC		0.02 ug/L	0.005 ug/L	30	
	Aldrin		0.02 ug/L	0.003 ug/L	30	58-117
	Heptachlor Epoxide		0.02 ug/L	0.005 ug/L	30	
	Endosulfan I		0.02 ug/L	0.002 ug/L	30	
	4,4'-DDE		0.02 ug/L	0.003 ug/L	30	
	Dieldrin		0.02 ug/L	0.001 ug/L	30	65-129
	Endrin		0.02 ug/L	0.003 ug/L	30	66-125
	4,4'-DDD		0.02 ug/L	0.001 ug/L	30	
	Endosulfan II		0.02 ug/L	0.001 ug/L	30	
	4,4'-DDT		0.02 ug/L	0.001 ug/L	30	69-130
	Endosulfan Sulfate		0.02 ug/L	0.001 ug/L	30	
	Toxaphene		0.5 ug/L	0.05 ug/L	30	
	Chlordane		0.2 ug/L	0.01 ug/L	30	
	Aroclor 1016		0.2 ug/L	0.04 ug/L	30	
	Aroclor 1221		0.4 ug/L	0.04 ug/L	30	
	Aroclor 1232		0.2 ug/L	0.04 ug/L	30	
	Aroclor 1242		0.2 ug/L	0.04 ug/L	30	
	Aroclor 1248		0.2 ug/L	0.04 ug/L	30	
	Aroclor 1254		0.2 ug/L	0.04 ug/L	30	
	Aroclor 1260		0.2 ug/L	0.04 ug/L	30	
	Tetrachloro-*m*-xylene[g]		NA	NA	NA	13-116
	Decachlorobiphenyl[g]		NA	NA	NA	10-125
625 Base Neutral Acid Extractables	*N*-Nitrosodimethylamine	Water	25 ug/L	0.5 ug/L	30	
	Bis(2-chloroethyl) Ether		10 ug/L	0.5 ug/L	30	
	1,2-Dichlorobenzene		10 ug/L	0.3 ug/L	30	
	1,3-Dichlorobenzene		10 ug/L	0.3 ug/L	30	
	1,4-Dichlorobenzene		10 ug/L	0.3 ug/L	30	51-89
	Bis(2-chloroisopropyl) Ether		10 ug/L	0.7 ug/L	30	
	N-Nitrosodi-*n*-propylamine		10 ug/L	0.6 ug/L	30	55-102
	Hexachloroethane		10 ug/L	0.5 ug/L	30	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC)**
625 Base Neutral Acid Extractables	Nitrobenzene	Water	10 ug/L	0.9 ug/L	30	
	Isophorone		10 ug/L	0.4 ug/L	30	
	Bis(2-chloroethoxy)methane		10 ug/L	0.8 ug/L	30	
	1,2,4-Trichlorobenzene		10 ug/L	0.6 ug/L	30	55-92
	Naphthalene		10 ug/L	0.7 ug/L	30	
	Hexachlorobutadiene		10 ug/L	0.6 ug/L	30	
	2-Chloronaphthalene		10 ug/L	0.6 ug/L	30	
	Dimethyl Phthalate		10 ug/L	0.8 ug/L	30	
	Acenaphthylene		10 ug/L	0.6 ug/L	30	
	Acenaphthene		10 ug/L	0.7 ug/L	30	60-103
	2,4-Dinitrotoluene		10 ug/L	1.3 ug/L	30	75-120
	2,6-Dinitrotoluene		10 ug/L	1.4 ug/L	30	
	Diethyl Phthalate		10 ug/L	1.0 ug/L	30	
	4-Chlorophenyl Phenyl Ether		10 ug/L	0.8 ug/L	30	
	Fluorene		10 ug/L	0.9 ug/L	30	
	4-Bromophenyl Phenyl Ether		10 ug/L	0.8 ug/L	30	
	Hexachlorobenzene		10 ug/L	1.0 ug/L	30	
	Phenanthrene		10 ug/L	0.7 ug/L	30	
	Anthracene		10 ug/L	0.8 ug/L	30	
	Di-*n* -butyl Phthalate		10 ug/L	1.4 ug/L	30	
	Fluoranthene		10 ug/L	1.1 ug/L	30	
	Pyrene		10 ug/L	1.2 ug/L	30	59-115
	Butyl Benzyl Phthalate		10 ug/L	1.6 ug/L	30	
	3,3'-Dichlorobenzidine		25 ug/L	0.7 ug/L	30	
	Benz(a)anthracene		10 ug/L	0.7 ug/L	30	
	Bis(2-ethylhexyl) Phthalate		10 ug/L	5 ug/L	30	
	Chrysene		10 ug/L	0.5 ug/L	30	
	Di-*n* -octyl Phthalate		10 ug/L	0.6 ug/L	30	
	Benzo(b)fluoranthene		10 ug/L	0.8 ug/L	30	
	Benzo(k)fluoranthene		10 ug/L	0.6 ug/L	30	
	Benzo(a)pyrene		10 ug/L	0.7 ug/L	30	
	Indeno(1,2,3-c,d)pyrene		10 ug/L	1.1 ug/L	30	
	Dibenz(a,h)anthracene		10 ug/L	1.4 ug/L	30	
	Benzo(g,h,i)perylene		10 ug/L	1.2 ug/L	30	
	Phenol		10 ug/L	0.5 ug/L	30	33-112
	2-Chlorophenol		10 ug/L	0.7 ug/L	30	38-112
	2-Nitrophenol		10 ug/L	0.8 ug/L	30	
	2,4-Dimethylphenol		10 ug/L	1.3 ug/L	30	
	2,4-Dichlorophenol		10 ug/L	0.9 ug/L	30	
	4-Chloro-3-methylphenol		10 ug/L	0.8 ug/L	30	62-111
	2,4,6-Trichlorophenol		10 ug/L	0.7 ug/L	30	
	2,4-Dinitrophenol		25 ug/L	6 ug/L	30	
	4-Nitrophenol		25 ug/L	1.4 ug/L	30	45-132
	2-Methyl-4,6-dinitrophenol		25 ug/L	1.1 ug/L	30	
	Pentachlorophenol		25 ug/L	1.1 ug/L	30	59-121

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC
625 Base Neutral Acid Extractables	2-Fluorophenol[g]	Water	NA	NA	NA	37-101
	Phenol-D6[g]		NA	NA	NA	40-109
	2,4,6-Tribromophenol[g]		NA	NA	NA	44-129
	Nitrobenzene-D5[g]		NA	NA	NA	49-108
	2-Fluorobiphenyl[g]		NA	NA	NA	56-104
	Terphenyl-D14[g]		NA	NA	NA	22-140
1653 Chlorinated Phenolics	4-Chlorophenol	Water	1.25 ug/L	0.03 ug/L	NA	NA
	2,4-Dichlorophenol		2.5 ug/L	0.06 ug/L	NA	NA
	2,6-Dichlorophenol		2.5 ug/L	0.03 ug/L	NA	NA
	2,4,5-Trichlorophenol		2.5 ug/L	0.05 ug/L	NA	NA
	2,4,6-Trichlorophenol		2.5 ug/L	0.04 ug/L	NA	NA
	2,3,4,6-Tetrachlorophenol		2.5 ug/L	0.04 ug/L	NA	NA
	Pentachlorophenol		5 ug/L	0.2 ug/L	NA	NA
	4-Chloroguaiacol		1.25 ug/L	0.02 ug/L	NA	NA
	3,4-Dichloroguaiacol		2.5 ug/L	0.02 ug/L	NA	NA
	4,5-Dichloroguaiacol		2.5 ug/L	0.03 ug/L	NA	NA
	4,6-Dichloroguaiacol		2.5 ug/L	0.03 ug/L	NA	NA
	3,4,5-Trichloroguaiacol		2.5 ug/L	0.06 ug/L	NA	NA
	3,4,6-Trichloroguaiacol		2.5 ug/L	0.03 ug/L	NA	NA
	4,5,6-Trichloroguaiacol		2.5 ug/L	0.04 ug/L	NA	NA
	Tetrachloroguaiacol		5 ug/L	0.05 ug/L	NA	NA
	4-Chlorocatechol		1.25 ug/L	0.02 ug/L	NA	NA
	3,4-Dichlorocatechol		2.5 ug/L	0.03 ug/L	NA	NA
	3,6-Dichlorocatechol		2.5 ug/L	0.03 ug/L	NA	NA
	4,5-Dichlorocatechol		2.5 ug/L	0.02 ug/L	NA	NA
	3,4,5-Trichlorocatechol		5 ug/L	0.04 ug/L	NA	NA
	3,4,6-Trichlorocatechol		5 ug/L	0.07 ug/L	NA	NA
	Tetrachlorocatechol		5 ug/L	0.04 ug/L	NA	NA
	5-Chlorovanillin		2.5 ug/L	0.05 ug/L	NA	NA
	6-Chlorovanillin		2.5 ug/L	0.05 ug/L	NA	NA
	5,6-Dichlorovanillin		5 ug/L	0.2 ug/L	NA	NA
	2-Chlorosyringaldehyde		2.5 ug/L	0.04 ug/L	NA	NA
	2,6-Dichlorosyringaldehyde		5 ug/L	0.2 ug/L	NA	NA
	Trichlorosyringol		2.5 ug/L	0.03 ug/L	NA	NA
	2,4-Dichlorophenol-D3[g]		NA	NA	NA	27-143[d]
	4-Chloroguaiacol-$^{13}C_6$[g]		NA	NA	NA	43-168[d]
	3,4,5-Trichlorophenol[g]		NA	NA	NA	24-167[d]
	5-Chlorovanillin-$^{13}C_6$[g]		NA	NA	NA	32-254[d]
	4,5-Dichlorocatechol-$^{13}C_6$[g]		NA	NA	NA	D-190[d]
	4,5,6-Trichloroguaiacol-$^{13}C_6$[g]		NA	NA	NA	51-139[d]
	Pentachlorophenol-$^{13}C_6$[g]		NA	NA	NA	27-167[d]
	Tetrachloroguaiacol-$^{13}C_6$[g]		NA	NA	NA	27-161[d]
	Tetrachlorocatechol-$^{13}C_6$[g]		NA	NA	NA	D-184[d]

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC)**
8015B	Methanol	Water	1 mg/L	0.2 mg/L	30	70-130[d]
	2-Propanol		1 mg/L	0.2 mg/L	30	70-130[d]
	1-Propanol		1 mg/L	0.2 mg/L	30	70-130[d]
	Ethylene Glycol		5 mg/L	2 mg/L	30	70-130[d]
	Propylene Glycol		5 mg/L	3 mg/L	30	70-130[d]
	Cyclohexanol[g]		NA	NA	NA	70-130[d]
	2,3-Butylene Glycol[g]		NA	NA	NA	59-162
8081A Chlorinated Pesticides	alpha-BHC	Soil	5 ug/Kg	2 ug/Kg	40	
	gamma-BHC (Lindane)		5 ug/Kg	0.4 ug/Kg	40	58-141
	beta-BHC		5 ug/Kg	0.5 ug/Kg	40	
	Heptachlor		5 ug/Kg	0.5 ug/Kg	40	42-145
	delta-BHC		5 ug/Kg	2 ug/Kg	40	
	gamma-Chlordane		5 ug/Kg	0.5 ug/Kg	40	
	alpha-Chlordane		5 ug/Kg	0.5 ug/Kg	40	
	Aldrin		5 ug/Kg	0.5 ug/Kg	40	47-150
	Heptachlor Epoxide		5 ug/Kg	2 ug/Kg	40	
	Endosulfan I		5 ug/Kg	0.4 ug/Kg	40	
	4,4'-DDE		5 ug/Kg	0.7 ug/Kg	40	
	Dieldrin		5 ug/Kg	0.6 ug/Kg	40	56-149
	Endrin		5 ug/Kg	0.3 ug/Kg	40	58-150
	4,4'-DDD		5 ug/Kg	0.7 ug/Kg	40	
	Endosulfan II		5 ug/Kg	0.5 ug/Kg	40	
	4,4'-DDT		5 ug/Kg	0.6 ug/Kg	40	60-150
	Endrin Aldehyde		5 ug/Kg	0.9 ug/Kg	40	
	Endosulfan Sulfate		5 ug/Kg	0.5 ug/Kg	40	
	Methoxychlor		5 ug/Kg	2 ug/Kg	40	
	Toxaphene		250 ug/Kg	17 ug/Kg	40	
	Tetrachloro-*m*-xylene[g]		NA	NA	NA	33-118
	Decachlorobiphenyl[g]		NA	NA	NA	32-141
8081A Chlorinated Pesticides	alpha-BHC	Water	0.01 ug/L	0.001 ug/L	30	
	gamma-BHC (Lindane)		0.01 ug/L	0.002 ug/L	30	65-118
	beta-BHC		0.01 ug/L	0.001 ug/L	30	
	Heptachlor		0.01 ug/L	0.004 ug/L	30	50-119
	delta-BHC		0.01 ug/L	0.005 ug/L	30	
	gamma-Chlordane		0.01 ug/L	0.001 ug/L	30	
	alpha-Chlordane		0.01 ug/L	0.001 ug/L	30	
	Aldrin		0.01 ug/L	0.003 ug/L	30	58-117
	Heptachlor Epoxide		0.01 ug/L	0.005 ug/L	30	
	Endosulfan I		0.01 ug/L	0.002 ug/L	30	
	4,4'-DDE		0.01 ug/L	0.003 ug/L	30	
	Dieldrin		0.01 ug/L	0.001 ug/L	30	65-129
	Endrin		0.01 ug/L	0.003 ug/L	30	66-125
	4,4'-DDD		0.01 ug/L	0.001 ug/L	30	
	Endosulfan II		0.01 ug/L	0.001 ug/L	30	
	4,4'-DDT		0.01 ug/L	0.001 ug/L	30	69-130

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC**
8081A Chlorinated Pesticides	Endrin Aldehyde	Water	0.01 ug/L	0.002 ug/L	30	
	Endosulfan Sulfate		0.01 ug/L	0.001 ug/L	30	
	Methoxychlor		0.01 ug/L	0.003 ug/L	30	
	Toxaphene		0.5 ug/L	0.05 ug/L	30	
	Tetrachloro-*m*-xylene[g]		NA	NA	NA	28-105
	Decachlorobiphenyl[g]		NA	NA	NA	10-134
8082 PCBs	Aroclor 1016	Soil	0.1 mg/Kg	0.01 mg/Kg	40	35-144
	Aroclor 1221		0.2 mg/Kg	0.01 mg/Kg	40	
	Aroclor 1232		0.1 mg/Kg	0.01 mg/Kg	40	
	Aroclor 1242		0.1 mg/Kg	0.01 mg/Kg	40	
	Aroclor 1248		0.1 mg/Kg	0.01 mg/Kg	40	
	Aroclor 1254		0.1 mg/Kg	0.01 mg/Kg	40	
	Aroclor 1260		0.1 mg/Kg	0.01 mg/Kg	40	47-153
	Decachlorobiphenyl[g]		NA	NA	NA	57-149
8082 PCBs	Aroclor 1016	Water	0.2 ug/L	0.04 ug/L	30	50-138
	Aroclor 1221		0.4 ug/L	0.04 ug/L	30	
	Aroclor 1232		0.2 ug/L	0.04 ug/L	30	
	Aroclor 1242		0.2 ug/L	0.04 ug/L	30	
	Aroclor 1248		0.2 ug/L	0.04 ug/L	30	
	Aroclor 1254		0.2 ug/L	0.04 ug/L	30	
	Aroclor 1260		0.2 ug/L	0.04 ug/L	30	61-135
	Decachlorobiphenyl[g]		NA	NA	NA	10-157
8082 PCB Congeners	2,4'-Dichlorobiphenyl (PCB 8)	Soil	0.5 ug/Kg	0.1 ug/Kg	40	25-131
	2,2',5-Trichlorobiphenyl (PCB 18)		0.5 ug/Kg	0.05 ug/Kg	40	
	2,4,4'-Trichlorobiphenyl (PCB 28)		0.5 ug/Kg	0.07 ug/Kg	40	43-137
	2,2',3,5'-Tetrachlorobiphenyl (PCB 44)		0.5 ug/Kg	0.05 ug/Kg	40	
	2,2',5,5'-Tetrachlorobiphenyl (PCB 52)		0.5 ug/Kg	0.1 ug/Kg	40	57-117
	2,3',4,4'-Tetrachlorobiphenyl (PCB 66)		0.5 ug/Kg	0.1 ug/Kg	40	59-121
	3,3',4,4'-Tetrachlorobiphenyl (PCB 77)		0.5 ug/Kg	0.05 ug/Kg	40	55-126
	2,2',3,4,5'-Pentachlorobiphenyl (PCB 87)		0.5 ug/Kg	0.07 ug/Kg	40	
	2,2',3,4',5-Pentachlorobiphenyl (PCB 90)		0.5 ug/Kg	0.05 ug/Kg	40	63-114
	2,2',4,5,5'-Pentachlorobiphenyl (PCB 101)		0.5 ug/Kg	0.03 ug/Kg	40	
	2,3,3',4,4'-Pentachlorobiphenyl (PCB 105)		0.5 ug/Kg	0.09 ug/Kg	40	63-120
	2,3,4,4',5-Pentachlorobiphenyl (PCB 114)		0.7 ug/Kg	0.3 ug/Kg	40	
	2,3',4,4',5-Pentachlorobiphenyl (PCB 118)		0.5 ug/Kg	0.04 ug/Kg	40	
	2',3,4,4',5-Pentachlorobiphenyl (PCB 123)		0.5 ug/Kg	0.08 ug/Kg	40	
	3,3',4,4',5-Pentachlorobiphenyl (PCB 126)		0.5 ug/Kg	0.1 ug/Kg	40	
	2,2',3,3',4,4'-Hexachlorobiphenyl (PCB 128)		0.5 ug/Kg	0.3 ug/Kg	40	
	2,2',3,4,4',5'-Hexachlorobiphenyl (PCB 138)		0.5 ug/Kg	0.04 ug/Kg	40	
	2,2',4,4',5,5'-Hexachlorobiphenyl (PCB 153)		0.5 ug/Kg	0.1 ug/Kg	40	
	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 156)		0.5 ug/Kg	0.09 ug/Kg	40	68-120
	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 157)		0.5 ug/Kg	0.2 ug/Kg	40	
	2,3',4,4',5,5'-Hexachlorobiphenyl (PCB 167)		0.7 ug/Kg	0.3 ug/Kg	40	
	3,3',4,4',5,5'-Hexachlorobiphenyl (PCB 169)		0.5 ug/Kg	0.1 ug/Kg	40	
	2,2',3,3',4,4',5-Heptachlorobiphenyl (PCB 170)		0.5 ug/Kg	0.06 ug/Kg	40	
	2,2',3,4,4',5,5'-Heptachlorobiphenyl (PCB 180)		0.5 ug/Kg	0.03 ug/Kg	40	68-120

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
8082 PCB Congeners	2,2',3,4,4',5',6-Heptachlorobiphenyl (PCB 183)	Soil	0.5 ug/Kg	0.09 ug/Kg	40	67-116
	2,2',3,4,4',6,6'-Heptachlorobiphenyl(PCB184)		0.5 ug/Kg	0.04 ug/Kg	40	63-115
	2,2',3,4',5,5',6-Heptachlorobiphenyl (PCB187)		0.5 ug/Kg	0.1 ug/Kg	40	
	2,3,3',4,4',5,5'-Heptachlorobiphenyl (PCB189)		0.5 ug/Kg	0.09 ug/Kg	40	70-121
	2,2',3,3',4,4',5,6-Octachlorobiphenyl (PCB195)		0.5 ug/Kg	0.1 ug/Kg	40	
	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (PCB206)		0.5 ug/Kg	0.09 ug/Kg	40	
	Decachlorobiphenyl (PCB 209)		0.5 ug/Kg	0.05 ug/Kg	40	60-120
	2,2',4,4',6,6'-Hexabromobiphenyl[g]		NA	NA	NA	45-148
8082 PCB Congeners	2,4'-Dichlorobiphenyl (PCB 8)	Water	5 ng/L	0.8 ng/L	30	70-130[d]
	2,2',5-Trichlorobiphenyl (PCB 18)		5 ng/L	0.8 ng/L	30	
	2,4,4'-Trichlorobiphenyl (PCB 28)		5 ng/L	0.4 ng/L	30	70-130[d]
	2,2',3,5'-Tetrachlorobiphenyl (PCB 44)		5 ng/L	0.3 ng/L	30	
	2,2',5,5'-Tetrachlorobiphenyl (PCB 52)		5 ng/L	0.5 ng/L	30	70-130[d]
	2,3',4,4'-Tetrachlorobiphenyl (PCB 66)		5 ng/L	0.7 ng/L	30	70-130[d]
	3,3',4,4'-Tetrachlorobiphenyl (PCB 77)		5 ng/L	0.8 ng/L	30	70-130[d]
	2,2',3,4,5'-Pentachlorobiphenyl (PCB 87)		5 ng/L	0.6 ng/L	30	
	2,2',3,4',5-Pentachlorobiphenyl (PCB 90)		5 ng/L	0.5 ng/L	30	70-130[d]
	2,2',4,5,5'-Pentachlorobiphenyl (PCB 101)		5 ng/L	0.4 ng/L	30	
	2,3,3',4,4'-Pentachlorobiphenyl (PCB 105)		5 ng/L	2 ng/L	30	70-130[d]
	2,3,4,4',5-Pentachlorobiphenyl (PCB 114)		5 ng/L	0.3 ng/L	30	
	2,3',4,4',5-Pentachlorobiphenyl (PCB 118)		5 ng/L	0.3 ng/L	30	
	2',3,4,4',5-Pentachlorobiphenyl (PCB 123)		5 ng/L	0.3 ng/L	30	
	3,3',4,4',5-Pentachlorobiphenyl (PCB 126)		5 ng/L	1 ng/L	30	
	2,2',3,3',4,4'-Hexachlorobiphenyl (PCB 128)		5 ng/L	2 ng/L	30	
	2,2',3,4,4',5'-Hexachlorobiphenyl (PCB 138)		5 ng/L	0.5 ng/L	30	
	2,2',4,4',5,5'-Hexachlorobiphenyl (PCB 153)		5 ng/L	0.4 ng/L	30	
	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 156)		5 ng/L	0.3 ng/L	30	70-130[d]
	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 157)		5 ng/L	0.4 ng/L	30	
	2,3',4,4',5,5'-Hexachlorobiphenyl (PCB 167)		5 ng/L	0.9 ng/L	30	
	3,3',4,4',5,5'-Hexachlorobiphenyl (PCB 169)		5 ng/L	0.8 ng/L	30	
	2,2',3,3',4,4',5-Heptachlorobiphenyl (PCB 170)		5 ng/L	0.4 ng/L	30	
	2,2',3,4,4',5,5'-Heptachlorobiphenyl (PCB 180)		5 ng/L	0.4 ng/L	30	70-130[d]
	2,2',3,4,4',5',6-Heptachlorobiphenyl (PCB 183)		5 ng/L	0.5 ng/L	30	70-130[d]
	2,2',3,4,4',6,6'-Heptachlorobiphenyl (PCB 184)		5 ng/L	0.4 ng/L	30	70-130[d]
	2,2',3,4',5,5',6-Heptachlorobiphenyl (PCB 187)		5 ng/L	0.7 ng/L	30	
	2,3,3',4,4',5,5'-Heptachlorobiphenyl (PCB 189)		5 ng/L	0.6 ng/L	30	70-130[d]
	2,2',3,3',4,4',5,6-Octachlorobiphenyl (PCB 195)		5 ng/L	1 ng/L	30	
	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (PCB 206)		5 ng/L	0.7 ng/L	30	
	Decachlorobiphenyl (PCB 209)		5 ng/L	0.9 ng/L	30	70-130[d]
	2,2',4,4',6,6'-Hexabromobiphenyl[g]		NA	NA	NA	59-133
8141A Organo-phosphorus Pesticides	Azinphos Methyl	Soil	0.05 mg/Kg	0.01 mg/Kg	40	
	Bolstar		0.05 mg/Kg	0.01 mg/Kg	40	52-119
	Chlorpyrifos		0.05 mg/Kg	0.01 mg/Kg	40	53-119
	Coumaphos		0.1 mg/Kg	0.01 mg/Kg	40	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC
8141A Organo-phosphorus Pesticides	Demeton-O and Demeton-S[h]	Soil	0.1 mg/Kg	0.03 mg/Kg	40	
	Diazinon		0.05 mg/Kg	0.01 mg/Kg	40	
	Dichlorvos		0.05 mg/Kg	0.02 mg/Kg	40	
	Dimethoate		0.05 mg/Kg	0.02 mg/Kg	40	
	Disulfoton		0.05 mg/Kg	0.02 mg/Kg	40	10-133
	EPN		0.05 mg/Kg	0.01 mg/Kg	40	
	Ethoprop		0.05 mg/Kg	0.02 mg/Kg	40	
	Ethyl Parathion		0.05 mg/Kg	0.01 mg/Kg	40	
	Fensulfothion		0.05 mg/Kg	0.02 mg/Kg	40	46-139
	Fenthion		0.05 mg/Kg	0.02 mg/Kg	40	
	Merphos		0.05 mg/Kg	0.01 mg/Kg	40	
	Mevinphos		0.05 mg/Kg	0.03 mg/Kg	40	
	Parathion Methyl		0.05 mg/Kg	0.01 mg/Kg	40	60-121
	Phorate		0.05 mg/Kg	0.02 mg/Kg	40	
	Ronnel		0.05 mg/Kg	0.02 mg/Kg	40	54-131
	Stirophos		0.05 mg/Kg	0.01 mg/Kg	40	
	Sulfotepp		0.05 mg/Kg	0.02 mg/Kg	40	
	Tokuthion (Prothiofos)		0.05 mg/Kg	0.01 mg/Kg	40	
	Trichloronate		0.05 mg/Kg	0.01 mg/Kg	40	
	Malathion		0.05 mg/Kg	0.01 mg/Kg	40	
	Triphenyl Phosphate[g]		NA	NA	NA	49-131
8141A Organo-phosphorus Pesticides	Azinphos Methyl	Water	1 ug/L	0.05 ug/L	30	
	Bolstar		0.2 ug/L	0.3 ug/L	30	36-120
	Chlorpyrifos		0.2 ug/L	0.05 ug/L	30	65-113
	Coumaphos		1 ug/L	0.08 ug/L	30	
	Demeton-O and Demeton-S[h]		1 ug/L	0.1 ug/L	30	
	Diazinon		0.2 ug/L	0.05 ug/L	30	
	Dichlorvos		0.5 ug/L	0.1 ug/L	30	
	Dimethoate		0.5 ug/L	0.07 ug/L	40	
	Disulfoton		1 ug/L	0.3 ug/L	30	37-100
	EPN		0.2 ug/L	0.08 ug/L	40	
	Ethoprop		0.2 ug/L	0.06 ug/L	30	
	Ethyl Parathion		0.5 ug/L	0.05 ug/L	40	
	Fensulfothion		0.5 ug/L	0.2 ug/L	30	50-142
	Fenthion		0.5 ug/L	0.2 ug/L	30	
	Merphos		0.2 ug/L	0.08 ug/L	30	
	Mevinphos		0.5 ug/L	0.07 ug/L	30	
	Parathion Methyl		0.5 ug/L	0.06 ug/L	30	65-110
	Phorate		0.5 ug/L	0.2 ug/L	30	
	Ronnel		0.2 ug/L	0.08 ug/L	30	49-131
	Stirophos		0.2 ug/L	0.08 ug/L	30	
	Sulfotepp		0.2 ug/L	0.05 ug/L	40	
8141A Organo-phosphorus Pesticides	Tokuthion (Prothiofos)	Water	0.2 ug/L	0.05 ug/L	30	
	Trichloronate		0.2 ug/L	0.05 ug/L	30	
	Malathion		0.2 ug/L	0.08 ug/L	30	
	Triphenyl Phosphate[g]		NA	NA	NA	17-150

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
8151A Chlorinated Herbicides	Dalapon	Soil	50 ug/Kg	4 ug/Kg	40	
	Dicamba		50 ug/Kg	2 ug/Kg	40	
	MCPA		10 mg/Kg	2 mg/Kg	40	
	MCPP		10 mg/Kg	2 mg/Kg	40	
	Dichloroprop		50 ug/Kg	4 ug/Kg	40	
	2,4-D		50 ug/Kg	4 ug/Kg	40	32-147
	2,4,5-TP (Silvex)		50 ug/Kg	5 ug/Kg	40	24-126
	2,4,5-T		50 ug/Kg	5 ug/Kg	40	23-137
	2,4-DB		50 ug/Kg	5 ug/Kg	40	
	Dinoseb		50 ug/Kg	7 ug/Kg	40	
	2,4-Dichlorophenylacetic Acid[f]		NA	NA	NA	26-146
8151A Chlorinated Herbicides	Dalapon	Water	0.4 ug/L	0.03 ug/L	30	
	Dicamba		0.4 ug/L	0.1 ug/L	30	
	MCPA		100 ug/L	14 ug/L	30	
	MCPP		100 ug/L	3 ug/L	30	
	Dichloroprop		0.4 ug/L	0.08 ug/L	30	
	2,4-D		0.4 ug/L	0.07 ug/L	30	48-125
	2,4,5-TP (Silvex)		0.2 ug/L	0.05 ug/L	30	37-139
	2,4,5-T		0.2 ug/L	0.04 ug/L	30	51-135
	2,4-DB		0.4 ug/L	0.2 ug/L	30	
	Dinoseb		0.2 ug/L	0.04 ug/L	30	
	2,4-Dichlorophenylacetic Acid[f]		NA	NA	NA	56-131
8151M Chlorinated Phenols	2,4,6-Trichlorophenol	Soil	5 ug/Kg	0.7 ug/Kg	40	
	2,3,4,5 -Tetrachlorophenol		10 ug/Kg	3 ug/Kg	40	52-108
	2,3,5,6 -Tetrachlorophenol		10 ug/Kg	4 ug/Kg	40	52-108
	Pentachlorophenol		5 ug/Kg	2 ug/Kg	40	47-113
	4-Bromo-2,6-dichlorophenol[f]		NA	NA	NA	21-128
8151M Chlorinated Phenols	2,4,6-Trichlorophenol	Water	0.5 ug/L	0.03 ug/L	30	
	2,3,4,5 -Tetrachlorophenol		0.5 ug/L	0.2 ug/L	30	46-100
	2,3,5,6 -Tetrachlorophenol		0.5 ug/L	0.2 ug/L	30	46-100
	Pentachlorophenol		0.5 ug/L	0.06 ug/L	30	28-128
	4-Bromo-2,6-dichlorophenol[f]		NA	NA	NA	38-119
8270C * Base Neutral Acid Extractables	*N*-Nitrosodimethylamine	Soil	2 mg/Kg	0.07 mg/Kg	40	
	Aniline		1 mg/Kg	0.3 mg/Kg	40	
	Bis(2-chloroethyl) Ether		0.3 mg/Kg	0.02 mg/Kg	40	
	1,2-Dichlorobenzene		0.3 mg/Kg	0.04 mg/Kg	40	
	1,3-Dichlorobenzene		0.3 mg/Kg	0.03 mg/Kg	40	
	1,4-Dichlorobenzene		0.3 mg/Kg	0.04 mg/Kg	40	37-115
	Bis(2-chloroisopropyl) Ether		0.3 mg/Kg	0.02 mg/Kg	40	
	N-Nitrosodi-*n*-propylamine		0.3 mg/Kg	0.03 mg/Kg	40	46-117
	Hexachloroethane		0.3 mg/Kg	0.03 mg/Kg	40	
	Nitrobenzene		0.3 mg/Kg	0.03 mg/Kg	40	
	Isophorone		0.3 mg/Kg	0.03 mg/Kg	40	
	Bis(2-chloroethoxy)methane		0.3 mg/Kg	0.03 mg/Kg	40	
	1,2,4-Trichlorobenzene		0.3 mg/Kg	0.02 mg/Kg	40	39-118
	Naphthalene		0.3 mg/Kg	0.03 mg/Kg	40	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC**
8270C * Base Neutral Acid Extractables	4-Chloroaniline	Soil	0.3 mg/Kg	0.05 mg/Kg	40	
	Hexachlorobutadiene		0.3 mg/Kg	0.03 mg/Kg	40	
	2-Methylnaphthalene		0.3 mg/Kg	0.03 mg/Kg	40	
	Hexachlorocyclopentadiene		0.3 mg/Kg	0.05 mg/Kg	40	
	2-Chloronaphthalene		0.3 mg/Kg	0.03 mg/Kg	40	
	2-Nitroaniline		2 mg/Kg	0.03 mg/Kg	40	
	Dimethyl Phthalate		0.3 mg/Kg	0.03 mg/Kg	40	
	Acenaphthylene		0.3 mg/Kg	0.03 mg/Kg	40	
	3-Nitroaniline		2 mg/Kg	0.05 mg/Kg	40	
	Acenaphthene		0.3 mg/Kg	0.03 mg/Kg	40	58-98
	Dibenzofuran		0.3 mg/Kg	0.03 mg/Kg	40	
	2,4-Dinitrotoluene		0.3 mg/Kg	0.03 mg/Kg	40	53-127
	2,6-Dinitrotoluene		0.3 mg/Kg	0.03 mg/Kg	40	
	Diethyl Phthalate		0.3 mg/Kg	0.03 mg/Kg	40	
	4-Chlorophenyl Phenyl Ether		0.3 mg/Kg	0.02 mg/Kg	40	
	Fluorene		0.3 mg/Kg	0.03 mg/Kg	40	
	4-Nitroaniline		2 mg/Kg	0.04 mg/Kg	40	
	N-Nitrosodiphenylamine		0.3 mg/Kg	0.03 mg/Kg	40	
	4-Bromophenyl Phenyl Ether		0.3 mg/Kg	0.03 mg/Kg	40	
	Hexachlorobenzene		0.3 mg/Kg	0.03 mg/Kg	40	
	Phenanthrene		0.3 mg/Kg	0.02 mg/Kg	40	
	Anthracene		0.3 mg/Kg	0.02 mg/Kg	40	
	Di-*n*-butyl Phthalate		0.3 mg/Kg	0.03 mg/Kg	40	
	Fluoranthene		0.3 mg/Kg	0.03 mg/Kg	40	
	Pyrene		0.3 mg/Kg	0.05 mg/Kg	40	56-108
	Butyl Benzyl Phthalate		0.3 mg/Kg	0.05 mg/Kg	40	
	3,3'-Dichlorobenzidine		2 mg/Kg	0.3 mg/Kg	40	
	Benz(a)anthracene		0.3 mg/Kg	0.03 mg/Kg	40	
	Bis(2-ethylhexyl) Phthalate		0.3 mg/Kg	0.04 mg/Kg	40	
	Chrysene		0.3 mg/Kg	0.03 mg/Kg	40	
	Di-*n*-octyl Phthalate		0.3 mg/Kg	0.07 mg/Kg	40	
	Benzo(b)fluoranthene		0.3 mg/Kg	0.03 mg/Kg	40	
	Benzo(k)fluoranthene		0.3 mg/Kg	0.03 mg/Kg	40	
	Benzo(a)pyrene		0.3 mg/Kg	0.02 mg/Kg	40	
	Indeno(1,2,3-cd)pyrene		0.3 mg/Kg	0.03 mg/Kg	40	
	Dibenz(a,h)anthracene		0.3 mg/Kg	0.04 mg/Kg	40	
	Benzo(g,h,i)perylene		0.3 mg/Kg	0.03 mg/Kg	40	
	Phenol		0.3 mg/Kg	0.03 mg/Kg	40	38-114
	2-Chlorophenol		0.3 mg/Kg	0.03 mg/Kg	40	52-98
	Benzyl Alcohol		0.3 mg/Kg	0.05 mg/Kg	40	
	2-Methylphenol		0.3 mg/Kg	0.03 mg/Kg	40	
	3- and 4-Methylphenol[h]		0.3 mg/Kg	0.04 mg/Kg	40	
	2-Nitrophenol		0.3 mg/Kg	0.03 mg/Kg	40	
	2,4-Dimethylphenol		0.3 mg/Kg	1 mg/Kg	40	
	Benzoic Acid		2 mg/Kg	0.07 mg/Kg	40	
	2,4-Dichlorophenol		0.3 mg/Kg	0.03 mg/Kg	40	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC)**
8270C * Base Neutral Acid Extractables	4-Chloro-3-methylphenol	Soil	0.3 mg/Kg	0.04 mg/Kg	40	48-104
	2,4,6-Trichlorophenol		0.3 mg/Kg	0.03 mg/Kg	40	
	2,4,5-Trichlorophenol		0.3 mg/Kg	0.03 mg/Kg	40	
	2,4-Dinitrophenol		2 mg/Kg	0.2 mg/Kg	40	
	4-Nitrophenol		2 mg/Kg	0.06 mg/Kg	40	52-104
	2-Methyl-4,6-dinitrophenol		2 mg/Kg	0.05 mg/Kg	40	
	Pentachlorophenol		2 mg/Kg	0.04 mg/Kg	40	52-104
*** Other Method 8270C options available for achieving lower MRLs**						
Additional Non-routine Analytes (Appendix I, II, IX, Etc.)						
8270C Base Neutral Acid Extractables	2-Picoline	Soil	0.7 mg/Kg			
	N-Nitrosodiethylamine		0.3 mg/Kg			
	Methyl Methanesulfonate		0.3 mg/Kg			
	N-Nitrosomethylethylamine		0.3 mg/Kg			
	Pentachloroethane		1 mg/Kg			
	Acetophenone		0.3 mg/Kg	0.02 mg/Kg		
	N-Nitrosopyrrolidine		0.3 mg/Kg			
	N-Nitrosomorpholine		0.3 mg/Kg			
	N-Nitrosopiperidine		0.3 mg/Kg			
	O,O,O-Triethyl Phosphorothioate		0.3 mg/Kg			
	2,6-Dichlorophenol		0.3 mg/Kg			
	Hexachloropropene		0.3 mg/Kg			
	N-Nitrosodi-n-butylamine		0.3 mg/Kg			
	p-Phenylenediamine		0.3 mg/Kg	2 mg/Kg		
	Safrole		0.3 mg/Kg			
	1,2,4,5-Tetrachlorobenzene		0.3 mg/Kg			
	Isosafrole		0.7 mg/Kg			
	1,3-Dinitrobenzene		0.3 mg/Kg			
	Pentachlorobenzene		0.3 mg/Kg			
	1-Naphthylamine		0.3 mg/Kg			
	2-Naphthylamine		0.3 mg/Kg			
	2,3,4,6-Tetrachlorophenol		2 mg/Kg	0.7 mg/Kg		
	Diphenylamine		0.3 mg/Kg			
	1,3,5-Trinitrobenzene		0.7 mg/Kg	0.05 mg/Kg		
	Phenacetin		2 mg/Kg			
	4-Aminobiphenyl		0.3 mg/Kg			
	4-Nitroquinoline N-Oxide		3 mg/Kg	0.04 mg/Kg		
	Aramite, Total		2 mg/Kg	0.04 mg/Kg		
	3,3'-Dimethylbenzidine		2 mg/Kg	2 mg/Kg		
	7,12-Dimethylbenz(a)anthracene		0.3 mg/Kg			
	Hexachlorophene		5 mg/Kg			
	3-Methylcholanthrene		0.3 mg/Kg			
	a,a-Dimethylphenethylamine		1 mg/Kg			
	2-Acetylaminofluorene		4 mg/Kg			
	o-Toluidine		0.3 mg/Kg			
	Ethyl methanesulfonate		0.3 mg/Kg			
	1,4-Naphthoquinone		0.3 mg/Kg			

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC
8270C Base Neutral Acid Extractables	5-Nitro-o-toluidine	Soil	0.3 mg/Kg			
	p-Dimethylaminoazobenzene		0.3 mg/Kg			
	Pentachloronitrobenzene		2 mg/Kg			
	Methapyrilene		1 mg/Kg			
	Chlorobenzilate		0.3 mg/Kg			
	1,2-Diphenylhydrazine		0.3 mg/Kg			
	1,4-Dichlorobutane		0.3 mg/Kg			
	1-Chloronaphthalen		0.3 mg/Kg			
	1-Methylnaphthalene		0.3 mg/Kg			
	Biphenyl		0.3 mg/Kg	0.01 mg/Kg		
	Atrazine		0.3 mg/Kg	0.02 mg/Kg		
	Benzidine		2 mg/Kg	0.5 mg/Kg		
	Carbazole		0.3 mg/Kg	0.02 mg/Kg		
	Diallate		0.3 mg/Kg			
	Dicyclopentadiene		0.3 mg/Kg	0.2 mg/Kg		
	Dimethoate		0.3 mg/Kg			
	Dinoseb		0.3 mg/Kg	0.07 mg/Kg		
	Disulfoton		0.3 mg/Kg			
	Famphur		4 mg/Kg	0.05 mg/Kg		
	Isodrin		0.3 mg/Kg			
	Kepone		4 mg/Kg	0.4 mg/Kg		
	Malathion		1 mg/Kg			
	Methyl Parathion		0.3 mg/Kg			
	Parathion		0.3 mg/Kg			
	Phorate		0.3 mg/Kg			
	Pronamide		0.3 mg/Kg	0.01 mg/Kg		
	Pyridine		0.3 mg/Kg	0.04 mg/Kg		
	Sulfotepp		0.3 mg/Kg	0.02 mg/Kg		
	Thionazin		2 mg/Kg			
	2-Fluorophenol[g]		NA	NA	NA	38-89
	Phenol-D6[g]		NA	NA	NA	29-111
	2,4,6-Tribromophenol[g]		NA	NA	NA	48-111
	Nitrobenzene-D5[g]		NA	NA	NA	46-103
	2-Fluorobiphenyl[g]		NA	NA	NA	53-106
	Terphenyl-D14[g]		NA	NA	NA	33-139
	N-Nitrosodimethylamine		25 ug/L	0.6 ug/L	30	
	Pyridine		25 ug/L	25 ug/L	30	
	Aniline		25 ug/L	5 ug/L	30	
	Bis(2-chloroethyl) Ether		10 ug/L	0.5 ug/L	30	
	1,2-Dichlorobenzene		10 ug/L	0.3 ug/L	30	
	1,3-Dichlorobenzene		10 ug/L	0.3 ug/L	30	
	1,4-Dichlorobenzene		10 ug/L	0.4 ug/L	30	51-89
	Bis(2-chloroisopropyl) Ether		10 ug/L	0.8 ug/L	30	
	N-Nitrosodi-*n*-propylamine		10 ug/L	0.7 ug/L	30	55-102
	Hexachloroethane		10 ug/L	0.5 ug/L	30	
	Nitrobenzene		10 ug/L	0.8 ug/L	30	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC)**
8270C * Base Neutral Acid Extractables	Isophorone	Water	10 ug/L	0.5 ug/L	30	
	Bis(2-chloroethoxy)methane		10 ug/L	0.8 ug/L	30	
	1,2,4-Trichlorobenzene		10 ug/L	0.6 ug/L	30	55-92
	Naphthalene		10 ug/L	0.7 ug/L	30	
	4-Chloroaniline		10 ug/L	2 ug/L	30	
	Hexachlorobutadiene		10 ug/L	0.7 ug/L	30	
	2-Methylnaphthalene		10 ug/L	0.9 ug/L	30	
	Hexachlorocyclopentadiene		10 ug/L	0.5 ug/L	30	
	2-Chloronaphthalene		10 ug/L	0.6 ug/L	30	
	2-Nitroaniline		25 ug/L	2 ug/L	30	
	Dimethyl Phthalate		10 ug/L	0.9 ug/L	30	
	Acenaphthylene		10 ug/L	0.6 ug/L	30	
	3-Nitroaniline		25 ug/L	2 ug/L	30	
	Acenaphthene		10 ug/L	0.8 ug/L	30	60-103
	Dibenzofuran		10 ug/L	0.6 ug/L	30	
	2,4-Dinitrotoluene		10 ug/L	2 ug/L	30	75-120
	2,6-Dinitrotoluene		10 ug/L	2 ug/L	30	
	Diethyl Phthalate		10 ug/L	1 ug/L	30	
	4-Chlorophenyl Phenyl Ether		10 ug/L	0.8 ug/L	30	
	Fluorene		10 ug/L	0.9 ug/L	30	
	4-Nitroaniline		25 ug/L	2 ug/L	30	
	N-Nitrosodiphenylamine		10 ug/L	1 ug/L	30	
	4-Bromophenyl Phenyl Ether		10 ug/L	0.8 ug/L	30	
	Hexachlorobenzene		10 ug/L	1 ug/L	30	
	Phenanthrene		10 ug/L	0.7 ug/L	30	
	Anthracene		10 ug/L	0.8 ug/L	30	
	Carbazole		10 ug/L	0.9 ug/L	30	
	Di-*n*-butyl Phthalate		10 ug/L	2 ug/L	30	
	Fluoranthene		10 ug/L	2 ug/L	30	
	Pyrene		10 ug/L	2 ug/L	30	59-115
	Butyl Benzyl Phthalate		10 ug/L	2 ug/L	30	
	3,3'-Dichlorobenzidine		25 ug/L	0.7 ug/L	30	
	Benz(a)anthracene		10 ug/L	0.7 ug/L	30	
	Bis(2-ethylhexyl) Phthalate		10 ug/L	5 ug/L	30	
	Chrysene		10 ug/L	0.5 ug/L	30	
	Di-*n*-octyl Phthalate		10 ug/L	0.6 ug/L	30	
	Benzo(b)fluoranthene		10 ug/L	0.8 ug/L	30	
	Benzo(k)fluoranthene		10 ug/L	0.6 ug/L	30	
	Benzo(a)pyrene		10 ug/L	0.7 ug/L	30	
	Indeno(1,2,3-cd)pyrene		10 ug/L	2 ug/L	30	
	Dibenz(a,h)anthracene		10 ug/L	2 ug/L	30	
	Benzo(g,h,i)perylene		10 ug/L	2 ug/L	30	
	Phenol		10 ug/L	0.5 ug/L	30	33-112
	2-Chlorophenol		10 ug/L	0.7 ug/L	30	38-112
	Benzyl Alcohol		10 ug/L	0.7 ug/L	30	
	2-Methylphenol		10 ug/L	0.9 ug/L	30	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC**
8270C * Base Neutral Acid Extractables	3- and 4-Methylphenol[h]	Water	10 ug/L	0.5 ug/L	30	
	2-Nitrophenol		10 ug/L	0.8 ug/L	30	
	2,4-Dimethylphenol		10 ug/L	2 ug/L	30	
	Benzoic Acid		25 ug/L	2 ug/L	30	
	2,4-Dichlorophenol		10 ug/L	0.9 ug/L	30	
	4-Chloro-3-methylphenol		10 ug/L	0.8 ug/L	30	62-111
	2,4,6-Trichlorophenol		10 ug/L	0.7 ug/L	30	
	2,4,5-Trichlorophenol		10 ug/L	0.9 ug/L	30	
	2,4-Dinitrophenol		25 ug/L	6 ug/L	30	
	4-Nitrophenol		25 ug/L	2 ug/L	30	45-132
	2-Methyl-4,6-dinitrophenol		25 ug/L	2 ug/L	30	
	Pentachlorophenol		25 ug/L	2 ug/L	30	59-121
*** Other Method 8270C options available for achieving lower MRLs**						
Additional Non-routine Analytes (Appendix I, II, IX, Etc.)						
8270C Base Neutral Acid Extractables	2-Picoline	Water	20 ug/L	5 ug/L		
	N-Nitrosodiethylamine		10 ug/L			
	Methyl Methanesulfonate		10 ug/L			
	N-Nitrosomethylethylamine		10 ug/L			
	Pentachloroethane		25 ug/L			
	Acetophenone		10 ug/L	0.6 ug/L		
	N-Nitrosopyrrolidine		10 ug/L			
	N-Nitrosomorpholine		10 ug/L			
	N-Nitrosopiperidine		10 ug/L			
	O,O,O-Triethyl Phosphorothioate		10 ug/L			
	2,6-Dichlorophenol		10 ug/L			
	Hexachloropropene		10 ug/L			
	N-Nitrosodi-n-butylamine		10 ug/L			
	p-Phenylenediamine		100 ug/L			
	Safrole		10 ug/L			
	1,2,4,5-Tetrachlorobenzene		10 ug/L			
	Isosafrole		10 ug/L			
	1,3-Dinitrobenzene		10 ug/L			
	Pentachlorobenzene		10 ug/L			
	1-Naphthylamine		10 ug/L			
	2-Naphthylamine		10 ug/L			
	2,3,4,6-Tetrachlorophenol		10 ug/L			
	Diphenylamine		10 ug/L			
	1,3,5-Trinitrobenzene		25 ug/L	2 ug/L		
	Phenacetin		50 ug/L			
	4-Aminobiphenyl		10 ug/L			
	4-Nitroquinoline N-Oxide		100 ug/L	2 ug/L		
	Aramite, Total		50 ug/L	2 ug/L		
	3,3'-Dimethylbenzidine		10 ug/L	3 ug/L		
	7,12-Dimethylbenz(a)anthracene		10 ug/L			
	Hexachlorophene		150 ug/L			
	3-Methylcholanthrene		10 ug/L			

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC)**
8270C Base Neutral Acid Extractables	a,a-Dimethylphenethylamine	Water	25 ug/L			
	2-Acetylaminofluorene		100 ug/L			
	o-Toluidine		10 ug/L			
	Ethyl methanesulfonate		10 ug/L			
	1,4-Naphthoquinone		10 ug/L			
	5-Nitro-o-toluidine		10 ug/L			
	p-Dimethylaminoazobenzene		10 ug/L			
	Pentachloronitrobenzene		50 ug/L			
	Methapyrilene		100 ug/L	20 ug/L		
	Chlorobenzilate		10 ug/L			
	1,2-Diphenylhydrazine		10 ug/L			
	1,4-Dichlorobutane		10 ug/L			
	1-Chloronaphthalen		10 ug/L			
	1-Methylnaphthalene		10 ug/L			
	Biphenyl		10 ug/L	0.4 ug/L		
	Atrazine		10 ug/L	0.5 ug/L		
	Benzidine		50 ug/L	30 ug/L		
	Carbazole		10 ug/L			
	Diallate		10 ug/L			
	Dicyclopentadiene		10 ug/L			
	Dimethoate		10 ug/L			
	Dinoseb		10 ug/L	2 ug/L		
	Disulfoton		10 ug/L			
	Famphur		10 ug/L	2 ug/L		
	Isodrin		10 ug/L			
	Kepone		10 ug/L	4 ug/L		
	Malathion		25 ug/L			
	Methyl Parathion		10 ug/L			
	Parathion		10 ug/L			
	Phorate		10 ug/L			
	Pronamide		10 ug/L	0.5 ug/L		
	Pyridine		25 ug/L			
	Sulfotepp		10 ug/L	0.6 ug/L		
	Thionazin		25 ug/L			
	2-Fluorophenol[g]		NA	NA	NA	37-101
	Phenol-D6[g]		NA	NA	NA	40-109
	2,4,6-Tribromophenol[g]		NA	NA	NA	44-129
	Nitrobenzene-D5[g]		NA	NA	NA	49-108
	2-Fluorobiphenyl[g]		NA	NA	NA	56-104
	Terphenyl-D14[g]		NA	NA	NA	22-140
8310 Polynuclear Aromatic Hydrocarbons	Naphthalene	Soil	0.1 mg/Kg	0.02 mg/Kg	40	
	Acenaphthene		0.1 mg/Kg	0.02 mg/Kg	40	36-116
	Acenaphthylene		0.1 mg/Kg	0.02 mg/Kg	40	
	Fluorene		0.02 mg/Kg	0.002 mg/Kg	40	
	Phenanthrene		0.01 mg/Kg	0.002 mg/Kg	40	
	Anthracene		0.01 mg/Kg	0.001 mg/Kg	40	

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC
8310 Polynuclear Aromatic Hydrocarbons	Fluoranthene	Soil	0.02 mg/Kg	0.004 mg/Kg	40	56-111
	Pyrene		0.02 mg/Kg	0.003 mg/Kg	40	
	Benz(a)anthracene		0.01 mg/Kg	0.001 mg/Kg	40	
	Chrysene		0.01 mg/Kg	0.002 mg/Kg	40	
	Benzo(b)fluoranthene		0.02 mg/Kg	0.002 mg/Kg	40	
	Benzo(k)fluoranthene		0.01 mg/Kg	0.001 mg/Kg	40	
	Benzo(a)pyrene		0.01 mg/Kg	0.001 mg/Kg	40	42-126
	Dibenz(a,h)anthracene		0.02 mg/Kg	0.003 mg/Kg	40	
	Benzo(g,h,i)perylene		0.02 mg/Kg	0.002 mg/Kg	40	
	Indeno(1,2,3-cd)pyrene		0.01 mg/Kg	0.001 mg/Kg	40	
	p -Terphenyl[g]		NA	NA	NA	44-133
8310 Polynuclear Aromatic Hydrocarbons	Naphthalene	Water	1 ug/L	0.6 ug/L	30	
	Acenaphthene		1 ug/L	0.5 ug/L	30	43-105
	Acenaphthylene		1 ug/L	0.7 ug/L	30	
	Fluorene		0.2 ug/L	0.05 ug/L	30	
	Phenanthrene		0.1 ug/L	0.03 ug/L	30	
	Anthracene		0.1 ug/L	0.02 ug/L	30	
	Fluoranthene		0.2 ug/L	0.05 ug/L	30	61-110
	Pyrene		0.2 ug/L	0.06 ug/L	30	
	Benz(a)anthracene		0.1 ug/L	0.04 ug/L	30	
	Chrysene		0.1 ug/L	0.03 ug/L	30	
	Benzo(b)fluoranthene		0.2 ug/L	0.03 ug/L	30	
	Benzo(k)fluoranthene		0.1 ug/L	0.03 ug/L	30	
	Benzo(a)pyrene		0.1 ug/L	0.04 ug/L	30	68-109
	Dibenz(a,h)anthracene		0.2 ug/L	0.08 ug/L	30	
	Benzo(g,h,i)perylene		0.2 ug/L	0.08 ug/L	30	
	Indeno(1,2,3-cd)pyrene		0.1 ug/L	0.06 ug/L	30	
	p -Terphenyl[g]		NA	NA	NA	44-109
8315	Formaldehyde	Soil	4 mg/Kg	1 mg/Kg	40	39-153[d]
8315	Formaldehyde	Water	100 ug/L	30 ug/L	30	39-153[d]
8330 Explosives	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	Soil	2 mg/Kg	0.2 mg/Kg	40	
	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)		2 mg/Kg	0.2 mg/Kg	40	
	1,3,5-Trinitrobenzene		2 mg/Kg	0.2 mg/Kg	40	
	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)		2 mg/Kg	0.4 mg/Kg	40	
	1,3-Dinitrobenzene		2 mg/Kg	0.2 mg/Kg	40	
	2,4,6-Trinitrotoluene		2 mg/Kg	0.2 mg/Kg	40	57-118
	Nitrobenzene		2 mg/Kg	0.2 mg/Kg	40	65-113
	4-Amino-2,6-dinitrotoluene		2 mg/Kg	0.4 mg/Kg	40	
	2-Amino-4,6-dinitrotoluene		2 mg/Kg	0.2 mg/Kg	40	
	2,6-Dinitrotoluene		2 mg/Kg	0.2 mg/Kg	40	
	2,4-Dinitrotoluene		2 mg/Kg	0.2 mg/Kg	40	65-117
	2-Nitrotoluene		2 mg/Kg	0.3 mg/Kg	40	
	4-Nitrotoluene		2 mg/Kg	0.4 mg/Kg	40	
	3-Nitrotoluene		2 mg/Kg	0.3 mg/Kg	40	
	1-Chloro-3-nitrobenzene[g]		NA	NA	NA	63-102

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC)**
8330 Explosives	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	Water	2 ug/L	0.2 ug/L	30	
	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)		2 ug/L	0.2 ug/L	30	
	1,3,5-Trinitrobenzene		2 ug/L	0.2 ug/L	30	
	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)		2 ug/L	0.2 ug/L	30	
	1,3-Dinitrobenzene		2 ug/L	0.2 ug/L	30	
	2,4,6-Trinitrotoluene		2 ug/L	0.2 ug/L	30	74-122
	Nitrobenzene		2 ug/L	0.3 ug/L	30	24-114
	4-Amino-2,6-dinitrotoluene		2 ug/L	0.2 ug/L	30	
	2-Amino-4,6-dinitrotoluene		2 ug/L	0.3 ug/L	30	
	2,6-Dinitrotoluene		2 ug/L	0.3 ug/L	30	
	2,4-Dinitrotoluene		2 ug/L	0.2 ug/L	30	35-141
	2-Nitrotoluene		2 ug/L	0.3 ug/L	30	
	4-Nitrotoluene		2 ug/L	0.4 ug/L	30	
	3-Nitrotoluene		2 ug/L	0.4 ug/L	30	
	1-Chloro-3-nitrobenzene[g]		NA	NA	NA	78-100
GC/MS-SIM Polynuclear Aromatic Hydrocarbons	Naphthalene	Soil	5 ug/Kg	0.2 ug/Kg	40	
	1-Methylnaphthalene		5 ug/Kg	0.09 ug/Kg	40	
	2-Methylnaphthalene		5 ug/Kg	0.2 ug/Kg	40	
	Biphenyl		5 ug/Kg	0.1 ug/Kg	40	
	2,6-Dimethylnaphthalene		5 ug/Kg	0.1 ug/Kg	40	
	Acenaphthylene		5 ug/Kg	0.08 ug/Kg	40	
	Dibenzofuran		5 ug/Kg	0.3 ug/Kg	40	
	Acenaphthene		5 ug/Kg	0.1 ug/Kg	40	45-123
	2,3,5-Trimethylnaphthalene		5 ug/Kg	0.09 ug/Kg	40	
	Fluorene		5 ug/Kg	0.2 ug/Kg	40	
	Phenanthrene		5 ug/Kg	0.3 ug/Kg	40	
	Anthracene		5 ug/Kg	0.2 ug/Kg	40	
	1-Methylphenanthrene		5 ug/Kg	0.08 ug/Kg	40	
	Fluoranthene		5 ug/Kg	0.2 ug/Kg	40	
	Pyrene		5 ug/Kg	0.2 ug/Kg	40	62-128
	Benz(a)anthracene		5 ug/Kg	0.2 ug/Kg	40	
	Chrysene		5 ug/Kg	0.2 ug/Kg	40	
GC/MS-SIM Polynuclear Aromatic Hydrocarbons	Benzo(b)fluoranthene	Soil	5 ug/Kg	0.2 ug/Kg	40	
	Benzo(k)fluoranthene		5 ug/Kg	0.2 ug/Kg	40	
	Benzo(a)pyrene		5 ug/Kg	0.1 ug/Kg	40	50-138
	Benzo(e)pyrene		5 ug/Kg	0.2 ug/Kg	40	
	Indeno(1,2,3-cd)pyrene		5 ug/Kg	0.2 ug/Kg	40	
	Dibenz(a,h)anthracene		5 ug/Kg	0.2 ug/Kg	40	
	Benzo(g,h,i)perylene		5 ug/Kg	0.2 ug/Kg	40	
	Fluorene-d10[g]		NA	NA	NA	42-130
	Fluoranthene-d10[g]		NA	NA	NA	55-130
	Terphenyl-d14[g]		NA	NA	NA	60-119

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC
GC/MS-SIM Polynuclear Aromatic Hydrocarbons	Naphthalene	Water	0.02 ug/L	0.004 ug/L	30	
	1-Methylnaphthalene		0.02 ug/L	0.003 ug/L	30	
	2-Methylnaphthalene		0.02 ug/L	0.003 ug/L	30	
	Biphenyl		0.02 ug/L	0.004 ug/L	30	
	2,6-Dimethylnaphthalene		0.02 ug/L	0.003 ug/L	30	
	Acenaphthylene		0.02 ug/L	0.002 ug/L	30	
	Dibenzofuran		0.02 ug/L	0.008 ug/L	30	
	Acenaphthene		0.02 ug/L	0.002 ug/L	30	44-109
	2,3,5-Trimethylnaphthalene		0.02 ug/L	0.002 ug/L	30	
	Fluorene		0.02 ug/L	0.003 ug/L	30	
	Phenanthrene		0.02 ug/L	0.004 ug/L	30	
	Anthracene		0.02 ug/L	0.002 ug/L	30	
	1-Methylphenanthrene		0.02 ug/L	0.002 ug/L	30	
	Fluoranthene		0.02 ug/L	0.003 ug/L	30	
	Pyrene		0.02 ug/L	0.003 ug/L	30	55-128
	Benz(a)anthracene		0.02 ug/L	0.003 ug/L	30	
	Chrysene		0.02 ug/L	0.002 ug/L	30	
	Benzo(b)fluoranthene		0.02 ug/L	0.002 ug/L	30	
	Benzo(k)fluoranthene		0.02 ug/L	0.002 ug/L	30	
	Benzo(a)pyrene		0.02 ug/L	0.002 ug/L	30	45-137
	Benzo(e)pyrene		0.02 ug/L	0.002 ug/L	30	
	Indeno(1,2,3-cd)pyrene		0.02 ug/L	0.003 ug/L	30	
	Dibenz(a,h)anthracene		0.02 ug/L	0.002 ug/L	30	
	Benzo(g,h,i)perylene		0.02 ug/L	0.004 ug/L	30	
	Fluorene-d10[g]		NA	NA	NA	31-97
	Fluoranthene-d10[g]		NA	NA	NA	31-113
	Terphenyl-d14[g]		NA	NA	NA	30-115
AK 102	Diesel Range Petroleum Hydrocarbons	Soil	10 mg/Kg	4 mg/Kg	40	75-125[d]
8015B			10 mg/Kg	4 mg/Kg	40	63-133
NWTPH-Dx			25 mg/Kg	4 mg/Kg	40	63-133
AK 102	o-Terphenyl[g]		NA	NA	NA	50-150[d]
8015B	o-Terphenyl[g]		NA	NA	NA	41-151
NWTPH-Dx	o-Terphenyl[g]		NA	NA	NA	50-150[d]
AK 102	Diesel Range Petroleum Hydrocarbons	Water	100 ug/L	50 ug/L	30	75-125[d]
8015B			250 ug/L	50 ug/L	30	51-143
NWTPH-Dx			250 ug/L	50 ug/L	30	51-143
AK 102	o-Terphenyl[g]		NA	NA	NA	50-150[d]
8015B	o-Terphenyl[g]		NA	NA	NA	35-123
NWTPH-Dx	o-Terphenyl[g]		NA	NA	NA	50-150[d]
AK 103	Residual Range Petroleum Hydrocarbons	Soil	100 mg/Kg	3 mg/Kg	40	60-120[d]
	n-Triacontane[g]		NA	NA	NA	50-150[d]

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC)**
524.2	Dichlorodifluoromethane (CFC 12)	Drinking Water	0.5 ug/L	0.2 ug/L	NA	70-130[d]
	Chloromethane		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	Vinyl Chloride		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	Bromomethane		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	Chloroethane		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	Trichlorofluoromethane (CFC 11)		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	1,1-Dichloroethene		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	Methylene Chloride		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	trans-1,2-Dichloroethene		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	2,2-Dichloropropane		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	cis-1,2-Dichloroethene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	1,1-Dichloroethane		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	Chloroform		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	Bromochloromethane		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	1,1,1-Trichloroethane (TCA)		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	1,1-Dichloropropene		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	Carbon Tetrachloride		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	Benzene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	1,2-Dichloroethane		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	Trichloroethene (TCE)		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	1,2-Dichloropropane		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	Bromodichloromethane		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	Dibromomethane		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	cis-1,3-Dichloropropene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	Toluene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	trans-1,3-Dichloropropene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	1,1,2-Trichloroethane		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	Tetrachloroethene (PCE)		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	1,3-Dichloropropane		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	1,2,3-Trichlorobenzene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	Dibromochloromethane		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	1,2-Dibromoethane (EDB)		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	Chlorobenzene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	Ethylbenzene		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	1,1,1,2-Tetrachloroethane		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	Styrene		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	m&p Xylenes		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	o-Xylene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	Bromoform		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	Isopropylbenzene		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	1,1,2,2-Tetrachloroethane		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	1,2,3-Trichloropropane		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	Bromobenzene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	n -Propylbenzene		0.5 ug/L	0.1 ug/L	NA	70-130[d]

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%RE
524.2	1,3,5-Trimethylbenzene	Water	0.5 ug/L	0.1 ug/L	NA	70-130[d]
	2-Chlorotoluene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	4-Chlorotoluene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	tert-Butylbenzene		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	1,2,4-Trimethylbenzene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	sec-Butylbenzene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	4-Isopropyltoluene		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	1,3-Dichlorobenzene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	1,4-Dichlorobenzene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	n-Butylbenzene		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	1,2-Dichlorobenzene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	1,2-Dibromo-3-chloropropane (DBCP)		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	1,2,4-Trichlorobenzene		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	Hexachlorobutadiene		0.5 ug/L	0.2 ug/L	NA	70-130[d]
	Naphthalene		0.5 ug/L	0.1 ug/L	NA	70-130[d]
	1,2-Dichlorobenzene-D4[g]		0.5 ug/L	NA	NA	73-120
	4-Bromofluorobenzene[g]		0.5 ug/L	NA	NA	82-106
602	Benzene	Water	0.5 ug/L	0.2 ug/L	30	39-150[d]
	Toluene		1 ug/L	0.09 ug/L	30	46-148[d]
	Ethylbenzene		1 ug/L	0.06 ug/L	30	32-160[d]
	m&p-Xylenes		1 ug/L	0.3 ug/L	30	32-160[d]
	o-Xylene		1 ug/L	0.08 ug/L	30	32-160[d]
	1,4-Difluorobenzene[g]		NA	NA	NA	77-117
624	Chloromethane	Water	5 ug/L	0.2 ug/L	30	D-273[d]
	Vinyl Chloride		5 ug/L	0.3 ug/L	30	D-251[d]
	Bromomethane		5 ug/L	0.3 ug/L	30	D-242[d]
	Chloroethane		5 ug/L	0.2 ug/L	30	14-230[d]
	Trichlorofluoromethane (CFC 11)		5 ug/L	0.2 ug/L	30	17-181[d]
	Trichlorotrifluoroethane (CFC 113)		5 ug/L	0.2 ug/L	30	
	1,1-Dichloroethene		5 ug/L	0.2 ug/L	30	D-234[d]
	Carbon Disulfide		5 ug/L	0.2 ug/L	30	
	Methylene Chloride		5 ug/L	0.2 ug/L	30	D-221[d]
	trans-1,2-Dichloroethene		5 ug/L	0.2 ug/L	30	54-156[d]
	2-Butanone (MEK)		20 ug/L	4 ug/L	30	
	1,1-Dichloroethane		5 ug/L	0.1 ug/L	30	59-155[d]
	Chloroform		5 ug/L	0.1 ug/L	30	51-138[d]
	1,1,1-Trichloroethane (TCA)		5 ug/L	0.2 ug/L	30	52-162[d]
	Carbon Tetrachloride		5 ug/L	0.2 ug/L	30	70-140[d]
	Benzene		5 ug/L	0.2 ug/L	30	37-151[d]
	1,2-Dichloroethane		5 ug/L	0.2 ug/L	30	49-155[d]
	Vinyl Acetate		10 ug/L	0.6 ug/L	30	
	Trichloroethene (TCE)		5 ug/L	0.2 ug/L	30	71-157[d]
	1,2-Dichloropropane		5 ug/L	0.2 ug/L	30	D-210[d]
	Bromodichloromethane		5 ug/L	0.2 ug/L	30	35-155[d]
	2-Chloroethyl Vinyl Ether		10 ug/L	0.2 ug/L	30	D-305[c]
	trans-1,3-Dichloropropene		5 ug/L	0.09 ug/L	30	17-183[d]

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
624	2-Hexanone	Water	20 ug/L	4 ug/L	30	
	4-Methyl-2-pentanone (MIBK)		20 ug/L	3 ug/L	30	
	Toluene		5 ug/L	0.1 ug/L	30	47-150[d]
	cis-1,3-Dichloropropene		5 ug/L	0.09 ug/L	30	D-227[d]
	1,1,2-Trichloroethane		5 ug/L	0.1 ug/L	30	52-150[d]
	Tetrachloroethene (PCE)		5 ug/L	0.2 ug/L	30	64-148[d]
	Dibromochloromethane		5 ug/L	0.2 ug/L	30	53-149[d]
	Chlorobenzene		5 ug/L	0.1 ug/L	30	37-160[d]
	Ethylbenzene		5 ug/L	0.1 ug/L	30	37-162[d]
	Styrene		5 ug/L	0.1 ug/L	30	
	m&p-Xylenes		5 ug/L	0.2 ug/L	30	
	o-Xylene		5 ug/L	0.08 ug/L	30	
	Bromoform		5 ug/L	0.3 ug/L	30	45-169[d]
	1,1,2,2-Tetrachloroethane		5 ug/L	0.2 ug/L	30	46-157[d]
	1,3-Dichlorobenzene		5 ug/L	0.2 ug/L	30	59-156[d]
	1,4-Dichlorobenzene		5 ug/L	0.09 ug/L	30	18-190[d]
	1,2-Dichlorobenzene		5 ug/L	0.09 ug/L	30	18-190[d]
	Dibromofluoromethane[g]		NA	NA	NA	69-123
	Toluene-D8[g]		NA	NA	NA	84-116
	4-Bromofluorobenzene[g]		NA	NA	NA	75-117
8021B BTEX (Low Level)	Benzene	Soil	5 ug/Kg	0.5 ug/Kg	40	61-119
	Toluene		5 ug/Kg	0.6 ug/Kg	40	59-118
	Ethylbenzene		5 ug/Kg	0.7 ug/Kg	40	51-132
	m&p-Xylenes		5 ug/Kg	2 ug/Kg	40	
	o-Xylene		5 ug/Kg	2 ug/Kg	40	
	1,4-Difluorobenzene[g]		NA	NA	NA	65-117
8021B BTEX (Mid Level)	Benzene	Soil	0.05 mg/Kg	0.02 mg/Kg	40	63-124
	Toluene		0.1 mg/Kg	0.03 mg/Kg	40	57-124
	Ethylbenzene		0.1 mg/Kg	0.03 mg/Kg	40	57-128
	m&p-Xylenes		0.1 mg/Kg	0.05 mg/Kg	40	50-135
	o-Xylene		0.1 mg/Kg	0.03 mg/Kg	40	48-127
	4-Bromofluorobenzene[g]		NA	NA	NA	37-130
8021B BTEX	Benzene	Water	0.5 ug/L	0.02 ug/L	30	79-117
	Toluene		1 ug/L	0.09 ug/L	30	79-117
	Ethylbenzene		1 ug/L	0.06 ug/L	30	76-124
	m&p-Xylenes		1 ug/L	0.3 ug/L	30	76-129
	o-Xylene		1 ug/L	0.08 ug/L	30	74-120
	1,4-Difluorobenzene[g]		NA	NA	NA	84-114
8260B Volatile Organics	Dichlorodifluoromethane (CFC 12)	Soil	5 ug/Kg	0.3 ug/Kg	40	
	Chloromethane		5 ug/Kg	0.6 ug/Kg	40	
	Vinyl Chloride		5 ug/Kg	0.8 ug/Kg	40	
	Bromomethane		5 ug/Kg	0.9 ug/Kg	40	
	Chloroethane		5 ug/Kg	0.6 ug/Kg	40	
	Trichlorofluoromethane (CFC 11)		5 ug/Kg	0.7 ug/Kg	40	
	Trichlorotrifluoromethane (CFC 113)		5 ug/Kg	2 ug/Kg	40	
	Acetone		50 ug/Kg	4 ug/Kg	40	

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC**
8260B Volatile Organics	1,1-Dichloroethene	Soil	5 ug/Kg	0.7 ug/Kg	40	76-135
	Carbon Disulfide		5 ug/Kg	0.9 ug/Kg	40	
	Methylene Chloride		10 ug/Kg	0.5 ug/Kg	40	
	trans -1,2-Dichloroethene		5 ug/Kg	0.6 ug/Kg	40	
	1,1-Dichloroethane		5 ug/Kg	0.5 ug/Kg	40	
	Methyl tert-butyl ether		5 ug/Kg	1 ug/Kg	40	
	2-Butanone (MEK)		20 ug/Kg	2 ug/Kg	40	
	2,2-Dichloropropane		5 ug/Kg	0.6 ug/Kg	40	
	cis-1,2-Dichloroethene		5 ug/Kg	0.5 ug/Kg	40	
	Chloroform		5 ug/Kg	0.5 ug/Kg	40	
	Bromochloromethane		5 ug/Kg	0.5 ug/Kg	40	
	1,1,1-Trichloroethane (TCA)		5 ug/Kg	0.5 ug/Kg	40	
	1,1-Dichloropropene		5 ug/Kg	0.6 ug/Kg	40	
	Carbon Tetrachloride		5 ug/Kg	0.7 ug/Kg	40	
	1,2-Dichloroethane		5 ug/Kg	0.5 ug/Kg	40	
	Benzene		5 ug/Kg	0.5 ug/Kg	40	84-115
	Trichloroethene (TCE)		5 ug/Kg	0.5 ug/Kg	40	78-128
	1,2-Dichloropropane		5 ug/Kg	0.4 ug/Kg	40	
	Bromodichloromethane		5 ug/Kg	0.4 ug/Kg	40	
	Dibromomethane		5 ug/Kg	0.4 ug/Kg	40	
	2-Hexanone		20 ug/Kg	2 ug/Kg	40	
	cis -1,3-Dichloropropene		5 ug/Kg	0.4 ug/Kg	40	
	Toluene		5 ug/Kg	0.5 ug/Kg	40	80-119
	trans -1,3-Dichloropropene		5 ug/Kg	0.4 ug/Kg	40	
	1,1,2-Trichloroethane		5 ug/Kg	0.5 ug/Kg	40	
	4-Methyl-2-pentanone (MIBK)		20 ug/Kg	2 ug/Kg	40	
	1,3-Dichloropropane		5 ug/Kg	0.4 ug/Kg	40	
	Tetrachloroethene (PCE)		5 ug/Kg	0.5 ug/Kg	40	
	Dibromochloromethane		5 ug/Kg	0.5 ug/Kg	40	
	1,2-Dibromoethane (EDB)		20 ug/Kg	0.5 ug/Kg	40	
	Chlorobenzene		5 ug/Kg	0.6 ug/Kg	40	77-117
	1,1,1,2-Tetrachloroethane		5 ug/Kg	0.5 ug/Kg	40	
	Ethylbenzene		5 ug/Kg	0.6 ug/Kg	40	
	m&p-Xylenes		5 ug/Kg	2 ug/Kg	40	
	o-Xylene		5 ug/Kg	0.7 ug/Kg	40	
	Styrene		5 ug/Kg	0.7 ug/Kg	40	
	Bromoform		5 ug/Kg	0.5 ug/Kg	40	
	Isopropylbenzene		20 ug/Kg	0.6 ug/Kg	40	
	1,1,2,2-Tetrachloroethane		5 ug/Kg	0.6 ug/Kg	40	
	1,2,3-Trichloropropane		5 ug/Kg	0.5 ug/Kg	40	
	Bromobenzene		5 ug/Kg	0.6 ug/Kg	40	
	n -Propylbenzene		20 ug/Kg	0.5 ug/Kg	40	
	2-Chlorotoluene		20 ug/Kg	0.6 ug/Kg	40	
	4-Chlorotoluene		20 ug/Kg	0.7 ug/Kg	40	
	1,3,5-Trimethylbenzene		20 ug/Kg	0.6 ug/Kg	40	
	tert -Butylbenzene		20 ug/Kg	0.6 ug/Kg	40	

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC)**
8260B Volatile Organics	1,2,4-Trimethylbenzene	Soil	20 ug/Kg	0.6 ug/Kg	40	
	sec -Butylbenzene		20 ug/Kg	0.6 ug/Kg	40	
	1,3-Dichlorobenzene		5 ug/Kg	0.7 ug/Kg	40	
	4-Isopropyltoluene		20 ug/Kg	0.7 ug/Kg	40	
	1,4-Dichlorobenzene		5 ug/Kg	0.8 ug/Kg	40	
	n-Butylbenzene		20 ug/Kg	0.8 ug/Kg	40	
	1,2-Dichlorobenzene		5 ug/Kg	0.7 ug/Kg	40	72-121
	1,2-Dibromo-3-chloropropane (DBCP)		20 ug/Kg	0.6 ug/Kg	40	
	1,2,4-Trichlorobenzene		20 ug/Kg	0.7 ug/Kg	40	
	1,2,3-Trichlorobenzene		20 ug/Kg	0.8 ug/Kg	40	
	Naphthalene		20 ug/Kg	0.8 ug/Kg	40	74-127
	Hexachlorobutadiene		20 ug/Kg	0.7 ug/Kg	40	
Additional Non-routine Analytes (Gasoline Additives, Appendix I, II, IX, Etc.)						
8260B Volatile Organics	Tert-Butyl Alcohol (TBA)	Soil	50 ug/Kg	10 ug/Kg		
	Diisopropyl ether (DIPE)		10 ug/Kg	5 ug/Kg		
	Tert-amyl Methyl Ether (TAME)		10 ug/Kg	5 ug/Kg		
	Allyl Chloride		20 ug/Kg			
	Acrolein		100 ug/Kg			
	Iodomethane		20 ug/Kg			
	Acetonitrile		20 ug/Kg			
	3-Chloro-1-propene		5 ug/Kg			
	n-Hexane		5 ug/Kg			
	Chloroprene		20 ug/Kg			
	Acrylonitrile		20 ug/Kg			
	Vinyl Acetate		20 ug/Kg	2 ug/Kg		
	Propionitrile		20 ug/Kg			
	Ethyl Acetate		5 ug/Kg			
	Methacrylonitrile		20 ug/Kg			
	Isobutyl Alcohol		100 ug/Kg			
	1-Chlorohexane		5 ug/Kg	0.8 ug/Kg		
	Methylmethacrylate		20 ug/Kg			
	1,4-Dioxane		250 ug/Kg	20 ug/Kg		
	2-Nitropropane		5 ug/Kg			
	Ethylmethacrylate		20 ug/Kg			
	cis-1,4-dichloro-2-butene		20 ug/Kg			
	trans-1,4-dichloro-2-butene		20 ug/Kg			
	Ethyl Ether		5 ug/Kg			
	1,3,5-Trichlorobenzene		5 ug/Kg	0.5 ug/Kg		
	2-Chloroethyl Vinyl Ether		10 ug/Kg	0.6 ug/Kg		
	Toluene-D8[g]		NA	NA	NA	80-125
	4-Bromofluorobenzene[g]		NA	NA	NA	80-123
	Dibromofluoromethane[g]		NA	NA	NA	84-121
	Toluene-D8[g]	Soil-mid	NA	NA	NA	32-166
	4-Bromofluorobenzene[g]		NA	NA	NA	40-153
	Dibromofluoromethane[g]		NA	NA	NA	41-152

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC
8260B Volatile Organics	Dichlorodifluoromethane (CFC 12)	Water	0.5 ug/L	0.2 ug/L	30	
	Chloromethane		0.5 ug/L	0.2 ug/L	30	
	Vinyl Chloride		0.5 ug/L	0.3 ug/L	30	
	Bromomethane		0.5 ug/L	0.3 ug/L	30	
	Chloroethane		0.5 ug/L	0.3 ug/L	30	
	Trichlorofluoromethane (CFC 11)		0.5 ug/L	0.2 ug/L	30	
	Trichlorotrifluoromethane (CFC 113)		0.5 ug/L	0.2 ug/L	30	
	Acetone		20 ug/L	5 ug/L	30	
	1,1-Dichloroethene		0.5 ug/L	0.2 ug/L	30	79-137
	Methyl tert-butyl ether		0.5 ug/L	0.08 ug/L	30	
	Carbon Disulfide		0.5 ug/L	0.2 ug/L	30	
	Methylene Chloride		2 ug/L	0.2 ug/L	30	
	trans -1,2-Dichloroethene		0.5 ug/L	0.2 ug/L	30	
	1,1-Dichloroethane		0.5 ug/L	0.1 ug/L	30	
	2-Butanone (MEK)		20 ug/L	5 ug/L	30	
	2,2-Dichloropropane		0.5 ug/L	0.2 ug/L	30	
	cis -1,2-Dichloroethene		0.5 ug/L	0.2 ug/L	30	
	Chloroform		0.5 ug/L	0.1 ug/L	30	
	Bromochloromethane		0.5 ug/L	0.2 ug/L	30	
	1,1,1-Trichloroethane (TCA)		0.5 ug/L	0.2 ug/L	30	
	1,1-Dichloropropene		0.5 ug/L	0.2 ug/L	30	
	Carbon Tetrachloride		0.5 ug/L	0.2 ug/L	30	
	1,2-Dichloroethane		0.5 ug/L	0.2 ug/L	30	
	Benzene		0.5 ug/L	0.2 ug/L	30	90-120
	Trichloroethene (TCE)		0.5 ug/L	0.2 ug/L	30	83-131
	1,2-Dichloropropane		0.5 ug/L	0.2 ug/L	30	
	Bromodichloromethane		0.5 ug/L	0.09 ug/L	30	
	Dibromomethane		0.5 ug/L	0.1 ug/L	30	
	2-Hexanone		20 ug/L	4 ug/L	30	
	cis -1,3-Dichloropropene		0.5 ug/L	0.09 ug/L	30	
	Toluene		0.5 ug/L	0.1 ug/L	30	89-119
	trans -1,3-Dichloropropene		0.5 ug/L	0.09 ug/L	30	
	1,1,2-Trichloroethane		0.5 ug/L	0.1 ug/L	30	
	4-Methyl-2-pentanone (MIBK)		20 ug/L	6 ug/L	30	
	1,3-Dichloropropane		0.5 ug/L	0.08 ug/L	30	
	Tetrachloroethene (PCE)		0.5 ug/L	0.2 ug/L	30	
	Dibromochloromethane		0.5 ug/L	0.09 ug/L	30	
	1,2-Dibromoethane (EDB)		2 ug/L	0.08 ug/L	30	
	Chlorobenzene		0.5 ug/L	0.1 ug/L	30	86-115
	1,1,1,2-Tetrachloroethane		0.5 ug/L	0.2 ug/L	30	
	Ethylbenzene		0.5 ug/L	0.2 ug/L	30	
	m&p-Xylenes		0.5 ug/L	0.3 ug/L	30	
	o-Xylene		0.5 ug/L	0.08 ug/L	30	
	Styrene		0.5 ug/L	0.1 ug/L	30	
	Bromoform		0.5 ug/L	0.3 ug/L	30	
	Isopropylbenzene		2 ug/L	0.07 ug/L	30	

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC)**
8260B Volatile Organics	1,1,2,2-Tetrachloroethane	Water	0.5 ug/L	0.2 ug/L	30	
	1,2,3-Trichloropropane		0.5 ug/L	0.3 ug/L	30	
	Bromobenzene		2 ug/L	0.2 ug/L	30	
	n -Propylbenzene		2 ug/L	0.1 ug/L	30	
	2-Chlorotoluene		2 ug/L	0.2 ug/L	30	
	4-Chlorotoluene		2 ug/L	0.2 ug/L	30	
	1,3,5-Trimethylbenzene		2 ug/L	0.2 ug/L	30	
	tert -Butylbenzene		2 ug/L	0.2 ug/L	30	
	1,2,4-Trimethylbenzene		2 ug/L	0.2 ug/L	30	
	sec -Butylbenzene		2 ug/L	0.2 ug/L	30	
	1,3-Dichlorobenzene		0.5 ug/L	0.2 ug/L	30	
	4-Isopropyltoluene		2 ug/L	0.2 ug/L	30	
	1,4-Dichlorobenzene		0.5 ug/L	0.1 ug/L	30	
	n -Butylbenzene		2 ug/L	0.3 ug/L	30	
	1,2-Dichlorobenzene		0.5 ug/L	0.09 ug/L	30	85-113
	1,2-Dibromo-3-chloropropane (DBCP)		2 ug/L	1 ug/L	30	
	1,2,4-Trichlorobenzene		2 ug/L	0.3 ug/L	30	
	1,2,3-Trichlorobenzene		2 ug/L	0.4 ug/L	30	
	Naphthalene		2 ug/L	0.3 ug/L	30	71-137
	Hexachlorobutadiene		2 ug/L	0.4 ug/L	30	
Additional Non-routine Analytes (Gasoline Additives, Appendix I, II, IX, Etc.)						
8260B Volatile Organics	Tert-Butyl Alcohol (TBA)	Water	20 ug/L	2 ug/L		
	Diisopropyl ether (DIPE)		2 ug/L	0.4 ug/L		
	Tert-amyl Methyl Ether (TAME)		2 ug/L	0.2 ug/L		
	Allyl Chloride		0.5 ug/L			
	Acrolein		20 ug/L	2 ug/L		
	Iodomethane		5 ug/L	0.3 ug/L		
	Acetonitrile		50 ug/L	4 ug/L		
	3-Chloro-1-propene		5 ug/L	0.3 ug/L		
	n-Hexane		0.5 ug/L	0.3 ug/L		
	Chloroprene		10 ug/L	0.3 ug/L		
	Acrylonitrile		5 ug/L	0.6 ug/L		
	Vinyl Acetate		5 ug/L	0.4 ug/L		
	Propionitrile		5 ug/L	2 ug/L		
	Ethyl Acetate		5 ug/L	2 ug/L		
	Methacrylonitrile		5 ug/L	0.2 ug/L		
	Isobutyl Alcohol		100 ug/L	5 ug/L		
	1-Chlorohexane		0.5 ug/L	0.2 ug/L		
	Methylmethacrylate		0.5 ug/L	0.2 ug/L		
	1,4-Dioxane		2500 ug/L	900 ug/L		
	2-Nitropropane		5 ug/L	0.3 ug/L		
	Ethylmethacrylate		5 ug/L	0.2 ug/L		
	cis-1,4-dichloro-2-butene		10 ug/L	0.8 ug/L		
	trans-1,4-dichloro-2-butene		10 ug/L	0.6 ug/L		
	Ethyl Ether		1 ug/L	0.5 ug/L		
	1,3,5-Trichlorobenzene		5 ug/L	0.6 ug/L		

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC**
8260B Volatile Organics	2-Chloroethyl Vinyl Ether	Water	5 ug/L	0.3 ug/L		
	Toluene-D8[g]		NA	NA	NA	87-118
	4-Bromofluorobenzene[g]		NA	NA	NA	81-113
	Dibromofluoromethane[g]		NA	NA	NA	78-124
AK 101	Gasoline Range Petroleum Hydrocarbons	Soil	5 mg/Kg	2 mg/Kg	40	60-120[d]
CA-TPH-G			5 mg/Kg	3 mg/Kg	40	78-128
NWTPH-Gx			5 mg/Kg	3 mg/Kg	40	78-128
AK 101	4-Bromofluorobenzene[g]		NA	NA	NA	60-120[d]
CA-TPH-G	4-Bromofluorobenzene[g]		NA	NA	NA	28-148
NWTPH-Gx	4-Bromofluorobenzene[g]		NA	NA	NA	50-150[d]
AK 101	Gasoline Range Petroleum Hydrocarbons	Water	50 ug/L	20 ug/L	30	60-120[d]
CA-TPH-G			50 ug/L	30 ug/L	30	73-124
NWTPH-Gx			50 ug/L	30 ug/L	30	73-124
AK 101	1,4-Difluorobenzene[g]		NA	NA	NA	60-120[d]
CA-TPH-G	1,4-Difluorobenzene[g]		NA	NA	NA	72-119
NWTPH-Gx	1,4-Difluorobenzene[g]		NA	NA	NA	50-150[d]

MICROBIOLOGICAL ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC)**
SM 9215B	Heterotrophic Plate Count (Pour)	Water	1 CFU/mL	NA	NA	NA
SM 9215C	Heterotrophic Plate Count (Spread)		1 CFU/mL	NA	NA	NA
SM 9221B	Coliform, Total (MPN)		2 MPN/100mL[i]	NA	NA	NA
SM 9221E	Coliform, Fecal (MPN)		2 MPN/100mL[i]	NA	NA	NA
SM 9223B	Coliform (Colilert)		Presence/100mL	NA	NA	NA
SM 9230B	Enterococcus		2 MPN/100mL	NA	NA	NA
SM 9230B	Fecal Streptococcus		2 MPN/100mL	NA	NA	NA
SM 10200H	Chlorophyll A		0.8 mg/L	0.3 mg/L	20	NA

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%RE(
ASTM D4129-82M	Total Organic Carbon	Sediment	0.05%	0.02%	20	85-115[b]
AVS	Sulfides	Sediment	0.5 mg/kg	-	20	60-130[b]
PSEP	Particle Size	Sediment	to 8 phi	NA	NA	NA
6010B - ICP	Cadmium	Sediment	0.2	0.1	30	75-125[m]
AVS-SEM	Copper		0.4	0.2	30	75-125[m]
	Lead		3	1	30	75-125[m]
	Nickel		0.5	0.5	30	75-125[m]
	Zinc		0.4	0.2	30	75-125[m]
7470A	Mercury		0.01	0.005	30	75-125[m]
200.8-ICP/MS	Aluminum	Tissue	1	0.08	30	70-130[m]
200.8-ICP/MS	Antimony		0.05	0.006	30	70-130[m]
200.8-ICP/MS	Arsenic		0.5	0.2	30	70-130[m]
200.8-ICP/MS	Barium		0.05	0.008	30	70-130[m]
200.8-ICP/MS	Beryllium		0.02	0.004	30	70-130[m]
200.8-ICP/MS	Cadmium		0.05	0.008	30	70-130[m]
6010B - ICP	Chromium		0.5	0.4	30	75-125[m]
200.8-ICP/MS	Cobalt		0.02	0.007	30	70-130[m]
200.8-ICP/MS	Copper		0.1	0.02	30	70-130[m]
200.8-ICP/MS	Lead		0.02	0.004	30	70-130[m]
200.8-ICP/MS	Manganese		0.05	0.02	30	70-130[m]
200.8-ICP/MS	Molybdenum		0.02	0.01	30	70-130
200.8-ICP/MS	Nickel		0.2	0.04	30	70-130[m]
7740 - GFAA	Selenium		1	1	30	60-130[m]
200.8-ICP/MS	Silver		0.02	0.003	30	70-130[m]
200.8-ICP/MS	Thallium		0.02	0.004	30	70-130[m]
6010B - ICP	Vanadium		1	0.5	30	75-125[m]
200.8-ICP/MS	Zinc		0.5	0.05	30	70-130[m]
200.8-ICP/MS	Arsenic	Seawater	0.5	0.04	20	56-99
Reductive	Beryllium		0.02	0.002	20	70-130
Precipitation	Cadmium		0.02	0.003	20	80-106
	Chromium		0.2	0.02	20	62-115
	Cobalt		0.02	0.006	20	70-130
	Copper		0.1	0.05	20	77-113
	Lead		0.02	0.009	20	82-116
	Nickel		0.2	0.05	20	77-114
	Silver		0.02	0.006	20	74-109
	Thallium		0.02	0.002	20	70-130
	Zinc		0.5	0.2	20	75-129
7742 - BRAA	Selenium	Seawater	1	0.3	20	75-125
8081A	alpha-BHC	Low-level	1 ug/Kg	0.2 ug/Kg	40	
Chlorinated	gamma-BHC (Lindane)	Sediment	1 ug/Kg	0.3 ug/Kg	40	58-141
Pesticides	beta-BHC		1 ug/Kg	0.2 ug/Kg	40	
	Heptachlor		1 ug/Kg	0.2 ug/Kg	40	42-145
	delta-BHC		1 ug/Kg	0.4 ug/Kg	40	

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
8081A Chlorinated Pesticides	gamma-Chlordane	Low-level Sediment	1 ug/Kg	0.2 ug/Kg	40	
	alpha-Chlordane		1 ug/Kg	0.2 ug/Kg	40	
	Aldrin		1 ug/Kg	0.3 ug/Kg	40	47-150
	Heptachlor Epoxide		1 ug/Kg	0.2 ug/Kg	40	
	Endosulfan I		1 ug/Kg	0.2 ug/Kg	40	
	4,4'-DDE		1 ug/Kg	0.3 ug/Kg	40	
	Dieldrin		1 ug/Kg	0.4 ug/Kg	40	56-149
	Endrin		1 ug/Kg	0.2 ug/Kg	40	58-150
	4,4'-DDD		1 ug/Kg	0.2 ug/Kg	40	
	Endosulfan II		1 ug/Kg	0.3 ug/Kg	40	
	4,4'-DDT		1 ug/Kg	0.2 ug/Kg	40	60-150
	Endrin Aldehyde		1 ug/Kg	0.4 ug/Kg	40	
	Endosulfan Sulfate		1 ug/Kg	0.2 ug/Kg	40	
	Endrin Ketone		1 ug/Kg	0.2 ug/Kg	40	
	Methoxychlor		1 ug/Kg	0.2 ug/Kg	40	
	Toxaphene		50 ug/Kg	6 ug/Kg	40	
	Tetrachloro-*m*-xylene[g]		NA	NA	NA	33-118
	Decachlorobiphenyl[g]		NA	NA	NA	32-141
8081A Chlorinated Pesticides	alpha-BHC	Tissue	1 ug/Kg	0.3 ug/Kg	40	
	gamma-BHC (Lindane)		1 ug/Kg	0.2 ug/Kg	40	70-130[d]
	beta-BHC		1 ug/Kg	0.4 ug/Kg	40	
	Heptachlor		1 ug/Kg	0.2 ug/Kg	40	70-130[d]
	delta-BHC		1 ug/Kg	0.4 ug/Kg	40	
	gamma-Chlordane		1 ug/Kg	0.2 ug/Kg	40	
	alpha-Chlordane		1 ug/Kg	0.1 ug/Kg	40	
	Aldrin		1 ug/Kg	0.2 ug/Kg	40	70-130[d]
	Heptachlor Epoxide		1 ug/Kg	0.1 ug/Kg	40	
	Endosulfan I		1 ug/Kg	0.2 ug/Kg	40	
	4,4'-DDE		1 ug/Kg	0.4 ug/Kg	40	
	Dieldrin		1 ug/Kg	0.3 ug/Kg	40	70-130[d]
	Endrin		1 ug/Kg	0.2 ug/Kg	40	70-130[d]
	4,4'-DDD		1 ug/Kg	0.4 ug/Kg	40	
	Endosulfan II		1 ug/Kg	0.1 ug/Kg	40	
	4,4'-DDT		1 ug/Kg	0.2 ug/Kg	40	70-130[d]
	Endrin Aldehyde		1 ug/Kg	0.2 ug/Kg	40	
	Endosulfan Sulfate		1 ug/Kg	0.2 ug/Kg	40	
	Endrin Ketone		1 ug/Kg	0.2 ug/Kg	40	
	Methoxychlor		1 ug/Kg	0.3 ug/Kg	40	
	Toxaphene		250 ug/Kg	20 ug/Kg	40	
	Tetrachloro-*m*-xylene[g]		NA	NA	NA	43-106
	Decachlorobiphenyl[g]		NA	NA	NA	46-126
8082 PCBs	Aroclor 1016	Low-level Sediment	10 ug/Kg	3 ug/Kg	40	35-144
	Aroclor 1221		20 ug/Kg	3 ug/Kg	40	
	Aroclor 1232		10 ug/Kg	3 ug/Kg	40	
	Aroclor 1242		10 ug/Kg	3 ug/Kg	40	

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC
8082 PCBs	Aroclor 1248	Low-level Sediment	10 ug/Kg	3 ug/Kg	40	
	Aroclor 1254		10 ug/Kg	3 ug/Kg	40	
	Aroclor 1260		10 ug/Kg	3 ug/Kg	40	47-153
	Decachlorobiphenyl[g]		NA	NA	NA	57-149
8082 PCBs	Aroclor 1016	Tissue	10 ug/Kg	5 ug/Kg	40	57-137
	Aroclor 1221		20 ug/Kg	5 ug/Kg	40	
	Aroclor 1232		10 ug/Kg	5 ug/Kg	40	
	Aroclor 1242		10 ug/Kg	5 ug/Kg	40	
	Aroclor 1248		10 ug/Kg	5 ug/Kg	40	
	Aroclor 1254		10 ug/Kg	5 ug/Kg	40	
	Aroclor 1260		10 ug/Kg	5 ug/Kg	40	64-136
	Decachlorobiphenyl[g]		NA	NA	NA	40-151
8082 PCBs	Aroclor 1016	Tissue Low-level	2 ug/Kg	0.7 ug/Kg	40	57-137
	Aroclor 1221		4 ug/Kg	0.7 ug/Kg	40	
	Aroclor 1232		2 ug/Kg	0.7 ug/Kg	40	
	Aroclor 1242		2 ug/Kg	0.7 ug/Kg	40	
	Aroclor 1248		2 ug/Kg	0.7 ug/Kg	40	
	Aroclor 1254		2 ug/Kg	0.7 ug/Kg	40	
	Aroclor 1260		2 ug/Kg	0.7 ug/Kg	40	64-136
	Decachlorobiphenyl[g]		NA	NA	NA	40-151
8082 PCBs	Aroclor 1016	Water Ultra-low Level	0.005 ug/Kg	0.002 ug/Kg	40	57-137
	Aroclor 1221		0.010 ug/Kg	0.002 ug/Kg	40	
	Aroclor 1232		0.005 ug/Kg	0.002 ug/Kg	40	
	Aroclor 1242		0.005 ug/Kg	0.002 ug/Kg	40	
	Aroclor 1248		0.005 ug/Kg	0.002 ug/Kg	40	
	Aroclor 1254		0.005 ug/Kg	0.002 ug/Kg	40	
	Aroclor 1260		0.005 ug/Kg	0.002 ug/Kg	40	64-136
	Decachlorobiphenyl[g]		NA	NA	NA	40-151
8082 PCB Congeners	2,4'-Dichlorobiphenyl (PCB 8)	Sediment	0.5 ug/Kg	0.1 ug/Kg	40	25-131
	2,2',5-Trichlorobiphenyl (PCB 18)		0.5 ug/Kg	0.05 ug/Kg	40	
	2,4,4'-Trichlorobiphenyl (PCB 28)		0.5 ug/Kg	0.07 ug/Kg	40	43-137
	2,2',3,5'-Tetrachlorobiphenyl (PCB 44)		0.5 ug/Kg	0.05 ug/Kg	40	
	2,2',5,5'-Tetrachlorobiphenyl (PCB 52)		0.5 ug/Kg	0.1 ug/Kg	40	57-117
	2,3',4,4'-Tetrachlorobiphenyl (PCB 66)		0.5 ug/Kg	0.1 ug/Kg	40	59-121
	3,3',4,4'-Tetrachlorobiphenyl (PCB 77)		0.5 ug/Kg	0.05 ug/Kg	40	55-126
	2,2',3,4,5'-Pentachlorobiphenyl (PCB 87)		0.5 ug/Kg	0.07 ug/Kg	40	
	2,2',3,4',5-Pentachlorobiphenyl (PCB 90)		0.5 ug/Kg	0.05 ug/Kg	40	63-114
	2,2',4,5,5'-Pentachlorobiphenyl (PCB 101)		0.5 ug/Kg	0.03 ug/Kg	40	
	2,3,3',4,4'-Pentachlorobiphenyl (PCB 105)		0.5 ug/Kg	0.09 ug/Kg	40	63-120
	2,3,4,4',5-Pentachlorobiphenyl (PCB 114)		0.7 ug/Kg	0.3 ug/Kg	40	
	2,3',4,4',5-Pentachlorobiphenyl (PCB 118)		0.5 ug/Kg	0.04 ug/Kg	40	
	2',3,4,4',5-Pentachlorobiphenyl (PCB 123)		0.5 ug/Kg	0.08 ug/Kg	40	
	3,3',4,4',5-Pentachlorobiphenyl (PCB 126)		0.5 ug/Kg	0.1 ug/Kg	40	
	2,2',3,3',4,4'-Hexachlorobiphenyl (PCB 128)		0.5 ug/Kg	0.3 ug/Kg	40	
	2,2',3,4,4',5'-Hexachlorobiphenyl (PCB 138)		0.5 ug/Kg	0.04 ug/Kg	40	
	2,2',4,4',5,5'-Hexachlorobiphenyl (PCB 153)		0.5 ug/Kg	0.1 ug/Kg	40	

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
8082 PCB Congeners	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 156)	Sediment	0.5 ug/Kg	0.09 ug/Kg	40	68-120
	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 157)		0.5 ug/Kg	0.2 ug/Kg	40	
	2,3',4,4',5,5'-Hexachlorobiphenyl (PCB 167)		0.7 ug/Kg	0.3 ug/Kg	40	
	3,3',4,4',5,5'-Hexachlorobiphenyl (PCB 169)		0.5 ug/Kg	0.1 ug/Kg	40	
	2,2',3,3',4,4',5-Heptachlorobiphenyl (PCB 170)		0.5 ug/Kg	0.06 ug/Kg	40	
	2,2',3,4,4',5,5'-Heptachlorobiphenyl (PCB 180)		0.5 ug/Kg	0.03 ug/Kg	40	68-120
	2,2',3,4,4',5',6-Heptachlorobiphenyl (PCB 183)		0.5 ug/Kg	0.09 ug/Kg	40	67-116
	2,2',3,4,4',6,6'-Heptachlorobiphenyl(PCB184)		0.5 ug/Kg	0.04 ug/Kg	40	63-115
	2,2',3,4',5,5',6-Heptachlorobiphenyl (PCB187)		0.5 ug/Kg	0.1 ug/Kg	40	
	2,3,3',4,4',5,5'-Heptachlorobiphenyl (PCB189)		0.5 ug/Kg	0.09 ug/Kg	40	70-121
	2,2',3,3',4,4',5,6-Octachlorobiphenyl (PCB195)		0.5 ug/Kg	0.1 ug/Kg	40	
	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (PCB206)		0.5 ug/Kg	0.09 ug/Kg	40	
	Decachlorobiphenyl (PCB 209)		0.5 ug/Kg	0.05 ug/Kg	40	60-120
	2,2',4,4',6,6'-Hexabromobiphenyl[g]		NA	NA	NA	45-148
8082 PCB Congeners	2,4'-Dichlorobiphenyl (PCB 8)	Tissue	2 ug/Kg	0.1 ug/Kg	40	25-131
	2,2',5-Trichlorobiphenyl (PCB 18)		0.5 ug/Kg	0.3 ug/Kg	40	
	2,4,4'-Trichlorobiphenyl (PCB 28)		0.5 ug/Kg	0.3 ug/Kg	40	43-137
	2,2',3,5'-Tetrachlorobiphenyl (PCB 44)		0.5 ug/Kg	0.2 ug/Kg	40	
	2,2',5,5'-Tetrachlorobiphenyl (PCB 52)		1 ug/Kg	0.5 ug/Kg	40	57-117
	2,3',4,4'-Tetrachlorobiphenyl (PCB 66)		0.5 ug/Kg	0.1 ug/Kg	40	59-121
	3,3',4,4'-Tetrachlorobiphenyl (PCB 77)		0.5 ug/Kg	0.3 ug/Kg	40	55-126
	2,2',3,4,5'-Pentachlorobiphenyl (PCB 87)		0.5 ug/Kg	0.2 ug/Kg	40	
	2,2',3,4',5-Pentachlorobiphenyl (PCB 90)		0.5 ug/Kg	0.1 ug/Kg	40	63-114
	2,2',4,5,5'-Pentachlorobiphenyl (PCB 101)		0.5 ug/Kg	0.2 ug/Kg	40	
	2,3,3',4,4'-Pentachlorobiphenyl (PCB 105)		0.5 ug/Kg	0.2 ug/Kg	40	63-120
	2,3,4,4',5-Pentachlorobiphenyl (PCB 114)		0.7 ug/Kg	0.2 ug/Kg	40	
	2,3',4,4',5-Pentachlorobiphenyl (PCB 118)		0.5 ug/Kg	0.2 ug/Kg	40	
	2',3,4,4',5-Pentachlorobiphenyl (PCB 123)		0.5 ug/Kg	0.2 ug/Kg	40	
	3,3',4,4',5-Pentachlorobiphenyl (PCB 126)		0.5 ug/Kg	0.2 ug/Kg	40	
	2,2',3,3',4,4'-Hexachlorobiphenyl (PCB 128)		0.5 ug/Kg	0.1 ug/Kg	40	
	2,2',3,4,4',5'-Hexachlorobiphenyl (PCB 138)		0.5 ug/Kg	0.1 ug/Kg	40	
	2,2',4,4',5,5'-Hexachlorobiphenyl (PCB 153)		1 ug/Kg	0.4 ug/Kg	40	
	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 156)		0.5 ug/Kg	0.2 ug/Kg	40	68-120
	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 157)		0.5 ug/Kg	0.2 ug/Kg	40	
	2,3',4,4',5,5'-Hexachlorobiphenyl (PCB 167)		0.5 ug/Kg	0.2 ug/Kg	40	
	3,3',4,4',5,5'-Hexachlorobiphenyl (PCB 169)		0.5 ug/Kg	0.2 ug/Kg	40	
	2,2',3,3',4,4',5-Heptachlorobiphenyl (PCB 170)		0.5 ug/Kg	0.1 ug/Kg	40	
	2,2',3,4,4',5,5'-Heptachlorobiphenyl (PCB 180)		0.5 ug/Kg	0.2 ug/Kg	40	68-120
	2,2',3,4,4',5',6-Heptachlorobiphenyl (PCB 183)		0.5 ug/Kg	0.3 ug/Kg	40	67-116
	2,2',3,4,4',6,6'-Heptachlorobiphenyl(PCB184)		0.5 ug/Kg	0.1 ug/Kg	40	63-115
	2,2',3,4',5,5',6-Heptachlorobiphenyl (PCB187)		0.5 ug/Kg	0.1 ug/Kg	40	
	2,3,3',4,4',5,5'-Heptachlorobiphenyl (PCB189)		0.5 ug/Kg	0.1 ug/Kg	40	70-121
	2,2',3,3',4,4',5,6-Octachlorobiphenyl (PCB195)		0.5 ug/Kg	0.2 ug/Kg	40	
	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (PCB206)		0.5 ug/Kg	0.1 ug/Kg	40	
	Decachlorobiphenyl (PCB 209)		0.5 ug/Kg	0.2 ug/Kg	40	60-120
	2,2',4,4',6,6'-Hexabromobiphenyl[g]		NA	NA	NA	45-148

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC
GC/MS-SIM Polynuclear Aromatic Hydrocarbons	Naphthalene	Sediment	5 ug/Kg	0.2 ug/Kg	40	
	1-Methylnaphthalene		5 ug/Kg	0.09 ug/Kg	40	
	2-Methylnaphthalene		5 ug/Kg	0.2 ug/Kg	40	
	Biphenyl		5 ug/Kg	0.1 ug/Kg	40	
	2,6-Dimethylnaphthalene		5 ug/Kg	0.1 ug/Kg	40	
	Acenaphthylene		5 ug/Kg	0.08 ug/Kg	40	
	Dibenzofuran		5 ug/Kg	0.3 ug/Kg	40	
	Acenaphthene		5 ug/Kg	0.1 ug/Kg	40	45-123
	2,3,5-Trimethylnaphthalene		5 ug/Kg	0.09 ug/Kg	40	
	Fluorene		5 ug/Kg	0.2 ug/Kg	40	
	Phenanthrene		5 ug/Kg	0.3 ug/Kg	40	
	Anthracene		5 ug/Kg	0.2 ug/Kg	40	
	1-Methylphenanthrene		5 ug/Kg	0.08 ug/Kg	40	
	Fluoranthene		5 ug/Kg	0.2 ug/Kg	40	
	Pyrene		5 ug/Kg	0.2 ug/Kg	40	62-128
	Benz(a)anthracene		5 ug/Kg	0.2 ug/Kg	40	
	Chrysene		5 ug/Kg	0.2 ug/Kg	40	
	Benzo(b)fluoranthene		5 ug/Kg	0.2 ug/Kg	40	
	Benzo(k)fluoranthene		5 ug/Kg	0.2 ug/Kg	40	
	Benzo(a)pyrene		5 ug/Kg	0.1 ug/Kg	40	50-138
	Benzo(e)pyrene		5 ug/Kg	0.2 ug/Kg	40	
	Indeno(1,2,3-cd)pyrene		5 ug/Kg	0.2 ug/Kg	40	
	Dibenz(a,h)anthracene		5 ug/Kg	0.2 ug/Kg	40	
	Benzo(g,h,i)perylene		5 ug/Kg	0.2 ug/Kg	40	
	Fluorene-d10[g]		NA	NA	NA	42-130
	Fluoranthene-d10[g]		NA	NA	NA	55-130
	Terphenyl-d14[g]		NA	NA	NA	60-119
GC/MS-SIM Polynuclear Aromatic Hydrocarbons	Naphthalene	Tissue	5 ug/Kg	5 ug/Kg	40	
	1-Methylnaphthalene		5 ug/Kg	5 ug/Kg	40	
	2-Methylnaphthalene		5 ug/Kg	5 ug/Kg	40	
	Biphenyl		5 ug/Kg	5 ug/Kg	40	
	2,6-Dimethylnaphthalene		5 ug/Kg	5 ug/Kg	40	
	Acenaphthylene		5 ug/Kg	4 ug/kg	40	
	Dibenzofuran		5 ug/Kg	4 ug/kg	40	
	Acenaphthene		5 ug/Kg	4 ug/kg	40	45-123
	2,3,5-Trimethylnaphthalene		5 ug/Kg	4 ug/kg	40	
	Fluorene		5 ug/Kg	3 ug/kg	40	
	Phenanthrene		5 ug/Kg	2 ug/kg	40	
	Anthracene		5 ug/Kg	2 ug/kg	40	
	1-Methylphenanthrene		5 ug/Kg	2 ug/kg	40	
	Fluoranthene		5 ug/Kg	2 ug/kg	40	
	Pyrene		5 ug/Kg	2 ug/kg	40	62-128
	Benz(a)anthracene		5 ug/Kg	2 ug/kg	40	
	Chrysene		5 ug/Kg	3 ug/kg	40	
	Benzo(b)fluoranthene		5 ug/Kg	3 ug/kg	40	
	Benzo(k)fluoranthene		5 ug/Kg	3 ug/kg	40	

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method [a]	Analyte	Matrix	Method Reporting Limit	Method Detection Limit	Precision[b] (RPD)	Accuracy[c] (%REC)
GC/MS-SIM Polynuclear Aromatic Hydrocarbons	Benzo(a)pyrene	Tissue	5 ug/Kg	3 ug/kg	40	50-138
	Benzo(e)pyrene		5 ug/Kg	2 ug/kg	40	
	Indeno(1,2,3-cd)pyrene		5 ug/Kg	2 ug/kg	40	
	Dibenz(a,h)anthracene		5 ug/Kg	3 ug/kg	40	
	Benzo(g,h,i)perylene		5 ug/Kg	2 ug/kg	40	
	Fluorene-d10[g]		NA	NA	NA	39-102
	Fluoranthene-d10[g]		NA	NA	NA	42-121
	Terphenyl-d14[g]		NA	NA	NA	50-123
GC/MS-SIM Polynuclear Aromatic Hydrocarbons	Naphthalene	Water	0.02 ug/L	0.004 ug/L	30	
	1-Methylnaphthalene		0.02 ug/L	0.003 ug/L	30	
	2-Methylnaphthalene		0.02 ug/L	0.003 ug/L	30	
	Biphenyl		0.02 ug/L	0.004 ug/L	30	
	2,6-Dimethylnaphthalene		0.02 ug/L	0.003 ug/L	30	
	Acenaphthylene		0.02 ug/L	0.002 ug/L	30	
	Dibenzofuran		0.02 ug/L	0.008 ug/L	30	
	Acenaphthene		0.02 ug/L	0.002 ug/L	30	44-109
	2,3,5-Trimethylnaphthalene		0.02 ug/L	0.002 ug/L	30	
	Fluorene		0.02 ug/L	0.003 ug/L	30	
	Phenanthrene		0.02 ug/L	0.004 ug/L	30	
	Anthracene		0.02 ug/L	0.002 ug/L	30	
	1-Methylphenanthrene		0.02 ug/L	0.002 ug/L	30	
	Fluoranthene		0.02 ug/L	0.003 ug/L	30	
	Pyrene		0.02 ug/L	0.003 ug/L	30	55-128
	Benz(a)anthracene		0.02 ug/L	0.003 ug/L	30	
	Chrysene		0.02 ug/L	0.002 ug/L	30	
	Benzo(b)fluoranthene		0.02 ug/L	0.002 ug/L	30	
	Benzo(k)fluoranthene		0.02 ug/L	0.002 ug/L	30	
	Benzo(a)pyrene		0.02 ug/L	0.002 ug/L	30	45-137
	Benzo(e)pyrene		0.02 ug/L	0.002 ug/L	30	
	Indeno(1,2,3-cd)pyrene		0.02 ug/L	0.003 ug/L	30	
	Dibenz(a,h)anthracene		0.02 ug/L	0.002 ug/L	30	
	Benzo(g,h,i)perylene		0.02 ug/L	0.004 ug/L	30	
	Fluorene-d10[g]		NA	NA	NA	31-97
	Fluoranthene-d10[g]		NA	NA	NA	31-113
	Terphenyl-d14[g]		NA	NA	NA	30-115
GC/FPD Organotin Compounds	Tributyltin	Sediment	1 ug/Kg	0.3 ug/Kg	40	30-131
	Dibutyltin		1 ug/Kg	0.3 ug/Kg	40	23-141
	Monobutyltin		1 ug/Kg	0.3 ug/Kg	40	10-127
	Tetrabutyltin		1 ug/Kg	0.4 ug/Kg	40	10-126
	Tripropyltin[g]		NA	NA	NA	22-113
GC/FPD Organotin Compounds	Tributyltin	Tissue	1 ug/Kg	0.3 ug/Kg	40	30-131
	Dibutyltin		1 ug/Kg	0.3 ug/Kg	40	23-141
	Monobutyltin		1 ug/Kg	0.3 ug/Kg	40	10-127
	Tetrabutyltin		1 ug/Kg	0.4 ug/Kg	40	10-126
	Tripropyltin[g]		NA	NA	NA	26-169

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method [a]	**Analyte**	**Matrix**	**Method Reporting Limit**	**Method Detection Limit**	**Precision[b] (RPD)**	**Accuracy[c] (%REC**
GC/FPD Organotin Compounds	Tributyltin	Water	0.02 ug/L	0.006 ug/L	30	22-157
	Dibutyltin		0.05 ug/L	0.007 ug/L	30	19-149
	Monobutyltin		0.05 ug/L	0.02 ug/L	30	23-150
	Tetrabutyltin		0.05 ug/L	0.007 ug/L	30	21-115
	Tripropyltin[g]		NA	NA	NA	41-128

Legend: NA = Not Applicable PCBs = Polychlorinated Biphenyls

a See Section 18 for list of references. Unless noted, the listed methods are EPA methods: *SM= Standard Methods for the Examination of Water and Wastewater* ; ASTM = American Society for Testing and Materials. Microbiology methods are *Standard Methods for the Examination of Water and Wastewater* , 18th edition.

b In-house limits, unless footnoted.

c Statistically derived Laboratory Control Sample (LCS) limits, unless footnoted.

d Method specified control limits.

e Method MRLs and matrix spike percent recovery data vary according to the method of extraction.

f Either method may be used, depending on project requirements.

g Surrogate compound

h These compounds coelute; therefore, results are reported as the combined concentration.

i MRL is 1.1 MPN/100mL for drinking water samples (10 tube method).

m Matrix spike control limits.

APPENDIX E

PREVENTIVE MAINTENANCE PROCEDURES

Preventive Maintenance Procedures

Instrument	Activity	Frequency
Refrigerators and Coolers	Record temperatures	Daily
	Clean coils	Annually
	Check coolant	Annually or if temperature outside limits
Vacuum Pumps	Clean and change pump oil	Every month or as needed
Fume Hoods	Face velocity measured	Quarterly
	Sash operation	As needed
	Change filters	Annually
	Inspect fan belts	Annually
Ovens	Clean	As needed or if temperature outside lim.
	Record temperatures	Daily, when in use
Incubators	Record temperatures	Daily, morning and evening
Water Baths	Record temperatures	Daily, morning and evening
	Wash with disinfectant solution	When water is murky, dirty, or growth appears
Autoclave	Check sterility	Every month
	Check temperature	Every month
	Clean	When mold or growth appears
Analytical Balances	Check alignment	Before every use
	Check calibration	Daily
	Clean pans and compartment	After every use
Dissolved Oxygen Meter	Change membrane	When fluctuations occur
pH probes	Condition probe	When fluctuations occur
Fluoride ISE	Store in storage solution	Between uses
Ammonia ISE	Store in storage solution	Between uses
UV-visible Spectrophotometer	Wavelength check	Annually
Total Organic Carbon Analyzers	Check IR zero	Weekly
	Check digestion/condensation vessels	Each use
	Clean digestion chamber	Every 2000 hours, or as needed
	Clean permeation tube	Every 2000 hours, or as needed
	Clean six-port valves	Every 200 - 2000 hours, or as needed
	Clean sample pump	Every 200 - 2000 hours, or as needed
	Clean carbon scrubber	Every 200 - 2000 hours, or as needed
	Clean IR cell	Every 2000 - 4000 hours, or as needed

Instrument	Activity	Frequency
Total Organic Halogen Analyzers	Change cell electrolyte	Daily
	Change electrode fluids	Daily
	Change pyrolysis tube	As needed
	Change inlet and outlet tubes	As needed
	Change electrodes	As needed
Flow Injection Analyzer	Check valve flares	Each use
	Check valve ports	Each use
	Check pump tubing	Each use
	Check light counts	Each use
	Check flow cell flares	Quarterly
	Change bulb	As needed
	Check manifold tubing	Each use
	Check T's and connectors	Each use
Ion Chromatographs	Change column	Every six months or as needed
	Change valve port face & hex nut	Every six months or as needed
	Clean valve slider	Every six months or as needed
	Change tubing	Annually or as needed
	Eluent pump	Annually
Atomic Absorption Spectro-photometers - FAA and CVAA	Check gases	Daily
	Clean burner head	Daily
	Check aspiration tubing	Daily
	Clean optics	Every three months
	Empty waste container	Weekly
Atomic Absorption Spectro-photometers - GFAA	Check gases	Daily
	Check argon dewar	Daily
	Change graphite tube	Daily, as needed
	Clean furnace windows	Monthly
ICP - AES	Check argon dewar	Daily
	Replace peristaltic pump tubing	Daily
	Empty waste container	Weekly
	Clean nebulizer, spray chamber, and torch	Every two weeks
	Replace water filter	Quarterly
	Replace vacuum air filters	Monthly

Instrument	Activity	Frequency
ICP - MS	Check argon dewar	Daily
	Check water level in chiller	Daily
	Complete instrument log	Daily
	Replace peristaltic pump tubing	Daily
	Clean sample and skimmer cones	As needed
	Clean RF contact strip	As needed
	Inspect nebulizer, spray chamber, and torch	Clean as needed
	Clean lens stack/extraction lens	As needed
	Check rotary pump oil	Monthly
	Change rotary pump oil	Every six months
Gel-Permeation Chromatographs	Clean and repack column	As needed
	Backflush valves	As needed
High Pressure Liquid Chromatographs	Backflush guard column	As needed
	Backflush column	As needed
	Change guard column	As needed when back pressure to high
	Change column	Annually or as needed
	Change in-line filters	As needed
	Leak check	After column maintenance
	Change pump seals	As needed
	Change pump diaphragm	Annually
	Clean flow cell	As needed
	Fluorescence detector check	Daily
	Diode array absorbance check	Daily
Gas Chromatographs, Semivolatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Change injection port liner	Weekly or as needed
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Clean FID	Weekly or as needed
	Clean NPD	Quarterly or as needed
	Clean ECD	Quarterly or as needed
	Leak test ECD	Annually

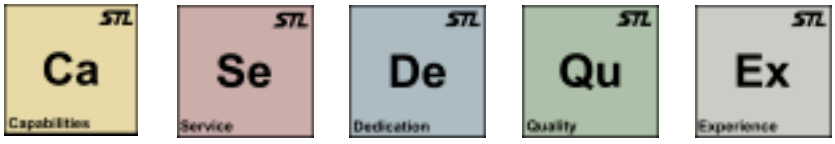
Instrument	Activity	Frequency
Gas Chromatograph/Mass Spectrometers, Semivolatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Annually or as needed
	Change septum	Daily, when in use
	Change injection port liner	Weekly or as needed
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean source	As needed when tuning problems
	Change pump oil	As specified by service specifications
Purge and Trap Concentrators	Change trap	Every four months or as needed
	Change transfer lines	Every six months or as needed
	Clean purge vessel	Daily
Gas Chromatographs, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Clean PID lamp	As needed
	Clean FID	As needed
	Change ion exchange resin	Every 60 days
	Replace nickel tubing	Quarterly or as needed
Gas Chromatograph/Mass Spectrometers, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Annually or as needed
	Change septum	Daily
	Clip first foot of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean jet separator	As needed
	Clean source	As needed when tuning problems
	Change pump oil	As specified by service specifications

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

CORPORATE
STATEMENT OF QUALIFICATIONS

SEPTEMBER 2000



Providing the Elements for your Success

SEVERN TRENT LABORATORIES

www.stl-inc.com

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

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TABLE OF CONTENTS

SECTION 1 COMPANY OVERVIEW

1.1 Company Overview 4
1.2 U.S. Laboratories 4

SECTION 2 SERVICES

2.1 Standard Services 7
2.1.1 Organic Analyses 7
2.1.2 Inorganic Analyses 8
2.2 Specialty Services 8
2.2.1 Analytical 9
2.2.2 Value-Added 11
2.3 Data Management 13
2.3.1 Data Reporting 13
2.3.2 Electronic Deliverables 14
2.4 Client Services 14
2.4.1 Project Management 14

SECTION 3 RESOURCES

3.1 Facilities 17
3.2 Instrumentation 18
3.2.1 Capacity 18
3.3 Personnel 19

SECTION 4 QUALITY ASSURANCE

4.1 Quality Assurance Policy 28
4.1.1 Objectives of the STL Quality System 28
4.1.2 Management Commitment to Quality Assurance 28
4.1.3 Proficiency Testing 28
4.1.4 Double Blind Performance Evaluation 29
4.1.5 Client Confidentiality and Proprietary Rights 29
4.1.6 Record Retention and Archival 29
4.2 Certifications 29

TABLE OF CONTENTS (continued)

SECTION 5 PROJECT EXPERIENCE

5.1 Project Descriptions 31
5.1.1 Federal/State/Local Agencies 31
5.1.2 Department of Defense 32
5.1.3 Industrial 34
5.1.4 Mixed Waste 35
5.1.5 Air 36
5.1.6 Geotechnical 37
5.1.7 On-Site Technologies Division 37
5.1.8 Aquatic Toxicity 38
5.1.9 Dredged Material Evaluation 38
5.1.10 Tissue Analyses 39
5.1.11 Dioxin 39
5.1.12 Radiochemistry 39

TABLES

Table 1A – U.S. & U.K. Facilities Overview
Table 2A – Analytical Protocols and Programs
Table 2B – Summary of Specialty Services
Table 3A – Laboratory Square Footage
Table 3B – Equipment List Summary
Table 3C – Corporate Management
Table 3D – General Managers
Table 3E – Key Laboratory Personnel

FIGURES

Figure 1A – U.S. Locations
Figure 3A – Corporate Management

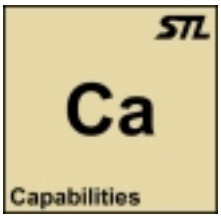
APPENDIX

Appendix A - Table of Analytical Services
Appendix B - Certifications

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

SECTION 1



COMPANY OVERVIEW

1.1 COMPANY OVERVIEW

Severn Trent Laboratories (STL) consists of 35 laboratory facilities; 29 in the United States and six in the United Kingdom. STL has grown in the U.S through the acquisitions of established, well managed, high quality and respected environmental analytical testing facilities. These laboratories have extensive experience with all matrices, methods, protocols and programs working on behalf of industry, commerce and government.

STL is a part of Severn Trent Services Inc. (STS), a major group of U.S. based companies with 5,000 employees throughout the U.S., Europe and Asia Pacific and annual revenues of $ 600 million. Both companies are owned by Severn Trent Plc., a $2 billion British water, waste and utility services company, one of the top 100 publicly traded companies in the United Kingdom, employing some 13,500 people.

The following table outlines the current number of sites and employees for STL's operations worldwide.

Table 1A - US & UK Facilities Overview

Location	# of Sites	# of Employees
US	**29**	**2000**
UK	**6**	**435**
Total	**35**	**2435**

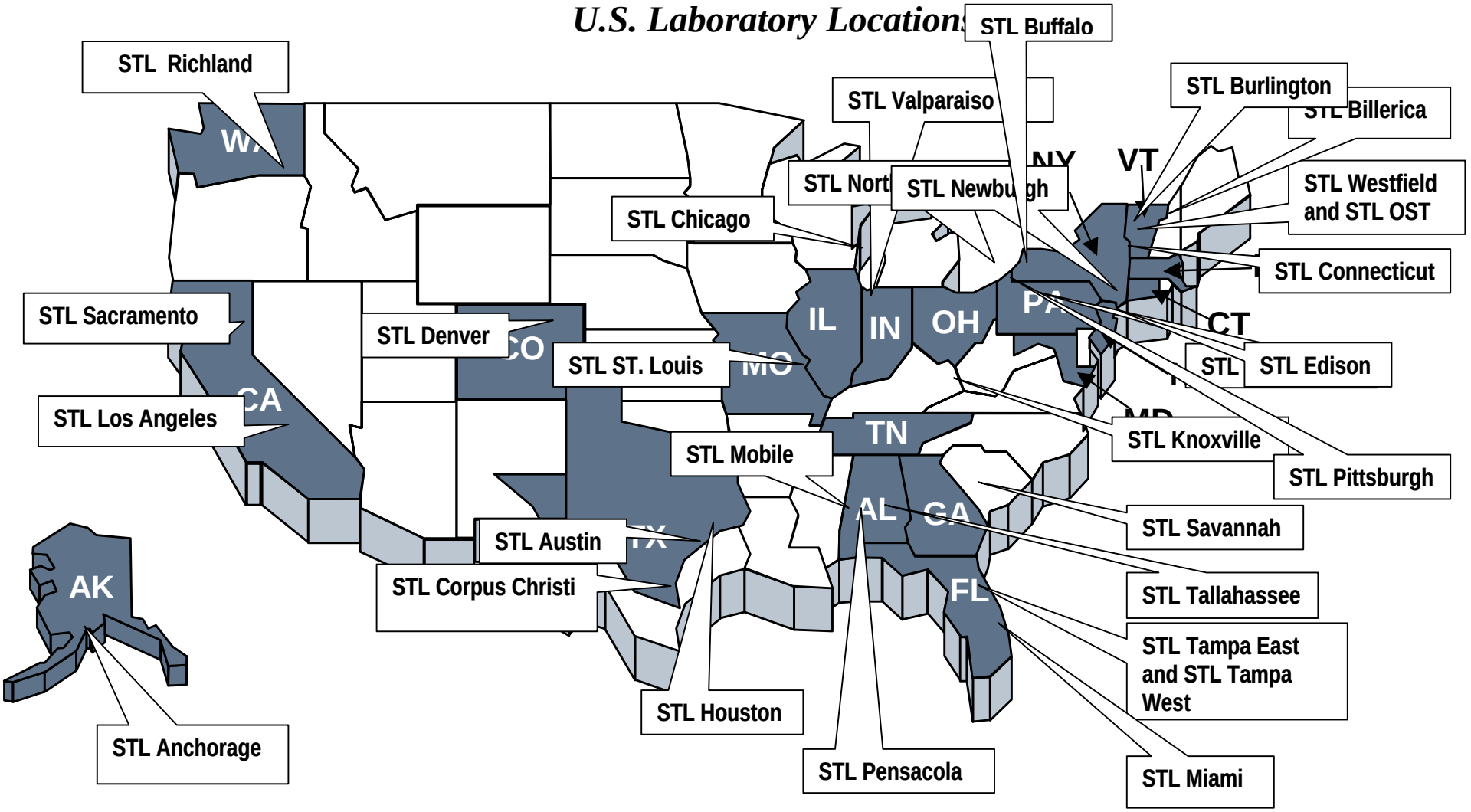
1.2 U.S. LABORATORIES

With 29 laboratory locations and over 1,500 chemists, microbiologists and environmental scientists, STL is well positioned as the leading group of laboratories for environmental testing in the U.S. Through continued investment in facilities, equipment, methods and people, STL can provide a broad range of services to meet clients' varying requirements.

This Statement of Qualifications outlines the services provided by STL's U.S. laboratories. STL's testing capabilities include chemical, physical and biological analyses of a variety of matrices, including aqueous, solid, drinking water, waste, tissue, air and saline/estuarine samples. Specialty capabilities include air toxics testing, mixed waste testing, tissue preparation and analysis, aquatic toxicology and microscopy. STL also operates one of the largest on-site analytical services in the United States via our On-Site Technologies (OST) Division.

The following page illustrates STL's U.S. locations.

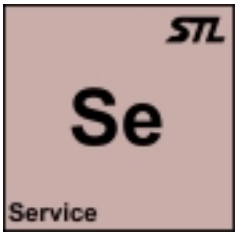
Figure 1A – U.S. Locations



SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

SECTION 2



SERVICES

2.1 STANDARD SERVICES

STL understands that scientifically sound, legally defensible analytical data is one of the most critical elements for the success of an environmental project. To ensure a project's data quality objectives are met, STL provides a superior standard of service using the latest technological advances and a commitment to customer service.

STL provides complete cradle-to-grave services, from initial preplanning and consultation, to project management and implementation, to final results and sample disposal.

STL performs analyses under various regulatory programs using both published and laboratory developed and validated test methods. In support of these activities, STL is certified/qualified in 50 states including Washington D.C. and Puerto Rico and participates in several federal programs.

Table 2A – Analytical Protocols and Programs

PROTOCOLS	PROGRAMS
• **EPA SW846** → Appendix IX → TCLP/SPLP/MEP • **Drinking Water** → 40 CFR 141, 143 • **Wastewater** → 40 CFR 136 → 600, Standard Methods → MCAWW	• **Department of Defense (DOD)** → U. S. Army → Air Force → Navy → Coast Guard → National Guard and others • **Department of Energy (DOE)** • **Resource Conservation and Recovery Act (RCRA)** • **Safe Drinking Water Act (SDWA)** • **Clean Water Act (CWA)** • **National Pollution Discharge Elimination System (NPDES)** • **Comprehensive Environmental Response, Compensation and Liability Act (CERCLA)/Superfund** • **Clean Air Act (CAA)** • **Marine Protection, Research, and Sanctuaries Act – (MPRSA)**
• **CLP Statement of Work** → TCL/TAL	
• **ASTM/NIOSH/EPA** → Toxic Organic Compounds in Ambient Air	
• **State Specific Protocols** → VPH/EPH • **USACE Dredged Materials**	

2.1.1 Organic Analyses

STL routinely tests for organic compounds using a variety of GC, GC/MS and HPLC test methods as described in Federal Register, EPA SW846 and the CLP Statement of Work. These analyses include volatiles, semivolatiles, pesticides, herbicides, polychlorinated biphenyls (PCBs), dioxins/furans, explosives, total petroleum hydrocarbons (TPH), Appendix IX, TCLP and other regulatory lists.

2.1.2 Inorganic Analyses

For high-speed, accurate metals analyses, STL uses a full array of instrumentation, including both sequential and simultaneous ICP. Low-level detection limits are achieved utilizing Trace-ICP, ICP/MS, Cold Vapor AA and Graphite Furnace AA. STL also provides a full range of wet chemistry analyses for nutrients, BOD, COD and virtually all other general chemistry parameters.

2.2 SPECIALTY SERVICES

In addition to routine analyses, STL has the capability to provide custom testing services for special projects requiring more sophisticated analyses. STL also provides a variety of value-added services that augment the laboratories' analytical capabilities.

Table 2B - Summary of Specialty Services

SPECIALTY SERVICES	
Analytical	**Value-Added**
• Radiochemistry and Mixed Waste Analyses • Microscopy Consulting Services • Geotechnical Analyses • Ambient Air Analyses • Source Emissions Air Analysis • Aquatic Toxicology • Tissue Analyses • Dredged Material Evaluation • CWA Chemical Degradation Analysis • Explosives Analyses (Method 8330) • Pulp & Paper Environmental Analytical Services • Natural Attenuation Byproducts • PCB Congener Analysis • Selective Ion Monitoring • Isotopic Analyses (Methods 1624 and 1625) • AVS/SEM • Alkyl Tins • Incidental PCBs (Method 680) • Low & High Resolution Dioxin / Furans • Specialty Pesticides & Herbicides by GC and HPLC • High Resolution GC / High Resolution MS • Liquid Chromatography/Mass Spectrometry (LC/MS) Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) • Boiler & Industrial Furnace (BIF)	• Mobile Lab Services • Field Sampling • Courier Service • EDD/GIS Capabilities • Consultant Training/Education • National Project Coordination • QAPP Preparation • QAPP Development • Expert Witness Testimony • Hazardous Materials/Health and Safety Training • Technical Support for Pulp & Paper Process Improvements • SF SampleScan • Method Development

2.2.1 Analytical

➢ **Radiochemistry**

STL St. Louis and STL Richland are fully licensed to receive, handle and analyze low-level environmental radioactive samples. Both laboratories provide a broad range of chemical and radionuclide determinations. STL St. Louis, licensed by the Nuclear Regulatory Commission (NRC), analyzes environmental samples, such as soil, water, waste, sediment and vegetation using a wide variety of radiochemical procedures from known regulatory methods (EPA, SW-846) to cutting edge technologies (DOE-EML, Eichrom®). STL Richland is licensed by the State of Washington to handle samples containing radioactive materials in environmental samples including vegetation, milk, wine and air particulates.

➢ **Mixed Waste Analyses**

In addition to STL St. Louis, STL Tampa West and STL Connecticut are also licensed by the NRC to analyze mixed waste samples. All laboratories offer a full array of organic and inorganic analysis.

➢ **Bioassay Monitoring**

Bioassay analysis identifies and assesses the degree, if any, of internal radiation exposure. STL Richland offers a full range of bioassay analyses for radioactive and stable toxic elements and compounds in urine, feces, hair, tissue, blood, bone and other biological materials.

➢ **Microscopy Consulting Services**

STL Billerica offers a variety of microscopy consulting services including Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray (EDX), Robinson Back Scatter Electron Detector, Transmission Electron Microscopy (TEM) and Optical Microscopy. These services can support a variety of industries including manufacturing, R&D, environmental, biomedical materials, engineering, electronics, forensics and graphic arts. Applications for this service include coating and thickness measurement, particle counting, printed wiring board manufacturing, electroplating, metal fabrication and non-destructive chemical analysis.

➢ **Geotechnical Testing**

To supplement geological studies, STL Burlington maintains a soil characterization laboratory. Laboratory technicians follow ASTM methods to define soil permeability, particle size, Atterberg limits, moisture, in place density, soils classification, specific gravity and hydraulic conductivity.

➢ **Air Analyses**

STL Austin, STL Burlington, STL Pensacola, STL Houston, STL Knoxville, STL Los Angeles, STL Miami, STL Sacramento and STL Savannah provide a variety of air services including the analysis of ambient, landfill and indoor air. Analyses include volatile and semivolatile compounds, pesticides/PCBs, polynuclear aromatic hydrocarbons and fixed gases. STL can also support air monitoring programs through sampling, continuous monitoring and permit consulting services.

➢ **Source Testing**

STL Austin, STL Sacramento and STL Knoxville laboratories offer analytical services for stack and process samples for various parameters including dioxin/furans, PAH, PCBs, metals, SVOC and

particulate in support of municipal waste incinerators, pulp and paper mills, sludge incinerators and chemical disposal incinerators.

- **Aquatic Toxicology**
  STL Westfield offers both freshwater and saltwater toxicity testing. This work is often completed pursuant to the requirements of the NPDES DMR program and stormwater monitoring assays. The protocols currently available from STL Westfield include 48-hour LC50 determinations, 7-day Modified Acute/ Chronic assays, NPDES Stormwater assays, Pass/Fail Acute screenings and Toxicity Identification (TIE) and Toxicity Reduction (TRE) Evaluations.

- **Tissue Analyses**
  STL Sacramento, STL Knoxville, STL Baltimore, STL Burlington, STL Savannah, STL Mobile, STL Tallahassee and STL Tampa West and STL Tampa East are recognized as leaders in the industry with respect to analysis of biological and vegetative tissues for a host of environmental parameters. At STL Baltimore, biological and vegetative tissues are prepared using American Society for Agronomy (ASA) and Association of Official Analytical Chemists (AOAC) methodologies, followed by routine analysis or by non-routine reference methods. At all laboratories, tissue sample analyses are generally tailored to project specific requirements, and require some development to ensure that target detection limits can be achieved with reduced sample sizes. Biological tissue analyses have been performed on aquatic, marine, terrestrial, and benthic species in support of site investigations, risk assessment, and permitting requirements on both full-body and specific organs for bioaccummulation.

- **Dredged Material Evaluation**
  STL Baltimore, STL Sacramento, STL Knoxville, STL Savannah, STL Mobile, STL Tallahassee and STL Tampa can support dredged material evaluation activities with analysis of sediment, eluate, elutriate, and tissue matrices for varied constituents of concern. Methodologies are performed in strict accordance with the regulatory requirements of the *Green Book*, *Inland Testing Manual*, *Management and Regulation of Dredging Activities in New Jersey's Tidal Waters*, and EPA's *Regional Implementation Manuals*. Specialty analyses have been developed to support such activities including STL's proprietary 'Co-planar PCBs by High Resolution Gas Chromatograph, High Resolution Mass Spectroscopy methodology conducted by STL Sacramento and STL Knoxville, Dioxin/Furans, PCB Congeners, AVS/SEM, and a chelation extraction procedure for metals determination. STL Burlington also has the capabilities to support dredged material evaluation activities for select analyses.

- **CWM Chemical Degradation Analysis**
  STL Baltimore, STL Sacramento, STL Denver and STL Knoxville offer analysis of Chemical Warfare Related Compounds. Compounds include environmental degradates of surety agents as well as other related chemicals. STL offer analytical services for the chemical degradation products water, soil/sediments, and tissue matrices. STL is fully capable of analysis for these compounds using the traditional USATHAMA methods. STL Sacramento pioneered the adaptation of Liquid Chromatography / Mass Spectrometry (LC/MS) and Liquid Chromatography/Mass Spectrometry (LC/MS) Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) methods that overcome some of the limitations of the traditional methods. STL can also provide analyses for the surety agents through our established partnership with an ERCD-certified laboratory.

➢ **Explosive Analysis**
STL Baltimore, STL Burlington, STL Chicago, STL Denver, STL Knoxville, STL Sacramento, STL St. Louis and STL Tallahassee can provide explosive analysis by Method 8330. This analysis can be performed on a variety of matrices including soils, waters and plant and animal tissues. STL Sacramento and STL Denver also uses Liquid Chromatography/Mass Spectrometry (LC/MS) Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS).

➢ **Pulp & Paper Environmental Analytical Services**
The Cluster Rule and Hazardous Air Pollutants (HAPs) monitoring requirements for the Pulp and Paper Industry can be provided through the STL Savannah and STL Mobile facilities. These facilities offer a comprehensive array of TAPPI, NCASI, EPA and other related tests to meet pulp and paper mill compliance testing needs which include the analysis of AOX, Chloroform, Chlorinated Phenolics, Methanol, Acetaldehyde, Methyl Ethyl Ketone and Propionaldehyde. STL Knoxville and STL Sacramento also provides dioxin/furan analyses on various matrices for routine and process control samples.

➢ **Natural Attenuation Byproducts (Intrinsic Bioremediation Evaluation)**
STL Savannah offers a wide array of analyses used to model intrinsic bioremediation. Specialty tests include chlorocatechols, dissolved gases, nutrients, demand and other traditional wet chemistry parameters.

➢ **Dioxin / Furan Analysis**
STL Knoxville and STL Sacramento offer dioxin/furan analysis on a wide variety of sample matrices including fish tissue, sludge, effluents, ambient air, stack emissions, pulp and paper products, soil and water. Analysis is conducted using state-of-the-art equipment; Methods used include EPA 8280, EPA 1613A, EPA SW-846 8290 and EPA-CLP DFLM01.0 for PCDD/PCDF. STL Savannah provides dioxin analysis using EPA Method 8280.

➢ **High Resolution Gas Chromatography High Resolution Mass Spectroscopy**
STL Knoxville and STL Sacramento have a long history of successful analysis of persistent organic pollutants in a wide variety of matrices for volatiles, semivolatiles, dioxins/furans, PAH and the use of STL's proprietary method for Co-planar PCBs

➢ **Boiler & Industrial Furnace Testing**
STL Austin can provide analyses in support of Boiler and Industrial Furnace Testing (40 CFR Part 266) (BIF). Tests include measurements of volatiles, semivolatiles, metals, anions and hexavalent chromium.

2.2.2 Value-Added

➢ **On-Site Technologies (OST) Division**
As one of the largest on-site analytical services divisions in the United States, STL's OST Division can provide a variety of on-site services.

Mobile Laboratory Services

STL's OST Division operates six mobile laboratories including 28' fifth wheel trailers and 45' box trailers. These laboratories are equipped to provide for on-site analysis of water and soil samples for volatile organics, PCBs, chlorinated pesticides and herbicides, total petroleum hydrocarbons, polyaromatic hydrocarbons and metals. Soil gas surveys for volatile organic compounds are also available. All of STL's mobile laboratories are designed to provide rapid, high quality and cost-effective data on location.

Contract/Operation Laboratories

The OST Division of STL provides for the staffing and operation of laboratories located at various contract/operation type projects including long-term remediation projects or site investigations. STL utilizes its experience with environmental analyses to provide for the rapid analysis of a variety of organic and inorganic constituents.

Sample Acquisition/Field Services

STL has several field sampling groups, including the OST Division, that provide a full range of sampling services for soil, air, groundwater, surface water, wastewater and other waste streams. These groups have successfully managed UST, NPDES, RCRA and state ISRA programs requiring field sampling and reporting in accordance with state and federal agencies. They have compiled field and analytical data and reports consisting of contour maps, photo-documentation, conclusions and recommendations.

Innovative In Situ Analyses

STL is committed to providing our clients with the most up-to-date technologies available for obtaining analytical data in the field. The OST Division is constantly reviewing innovative technologies which will allow STL to provide high quality and timely data at the point of sample collection.

- **Courier Service**

  STL offers sample pick-up for local clients. This service is designed for customer convenience and may have a small fee dependent upon the location of sample pick-up. STL's couriers are trained in sample handling and transportation protocols.

- **EDD/GIS Capabilities**

  In addition to producing standard electronic deliverables, some STL facilities have formed an alliance with GIS/Key and EQUIS, two companies that have developed GIS software packages for managing environmental data. Using GIS/Key and EQUIS, these STL facilities can produce the necessary files for electronic import of chemistry data and transmit data directly into a client database.

- **Consultant Training/Education**

  STL offers training programs on topics such as sample custody, laboratory quality assurance/quality control, method selection, state specific method variations and regulatory overview. STL also provides customized presentations based on client requests.

- **National Project Coordination**
  STL provides a national point of contact to oversee the administration of multiple-site projects that utilize several of STL's facilities. This contact works with each laboratory communicating customer expectations, resolving issues and implementing proactive logistical solutions.

- **Quality Assurance Project Plan (QAPP) Preparation**
  STL personnel can provide assistance in writing QAPPs for a variety of projects, ranging from one time sampling events to long term remedial investigations.

- **Method Development**
  STL can develop new, or modify existing, analytical methodologies for projects where existing methods do not meet project objectives. All method development goes through rigorous method validation to ensure accurate and complete data.

- **Hazardous Materials/Health and Safety Training**
  STL Miami offers four different Hazardous Materials/Health and Safety Training courses include 40 Hour HAZMAT/Health and Safety, 24 Hour Limited Exposure/Site Support, 8 Hour Hazardous Materials Supervisor/Management and 8 Hour Annual Refresher. STL Precision's environmental health and safety experts are also available to discuss individual requirements and design customized programs that address client's needs.

- **Technical Support for Pulp & Paper Process Improvements**
  In addition to environmental analytical services, STL Savannah and STL Mobile facilities offer the pulp & paper industry technical support such as feasibility analysis for engineering plant modifications, retention time and waste loading studies, capacity for wastewater treatment plant loading and chlorine dioxide generator optimization.

- **STL SampleScan**
  STL Denver offers an automated barcode sampling system for the accurate and unique identification of each sample container. This automation increases sampling speed and improves accuracy. This system allows for sample information to be downloaded into the fixed laboratory's laboratory information system for complete and accurate representation of samples delivered.

2.3 DATA MANAGEMENT

Through advanced information technology and electronic data exchange, STL is constantly striving to develop faster and more efficient methods of information gathering and distribution. STL knows there are no short cuts to producing consistently reliable results, however investments in information technology enable STL to quickly and efficiently gather, process and deliver sample results, saving valuable time and money for the client.

2.3.1 Data Reporting

STL can provide various types of data reporting based upon a project's needs. STL facilities can produce data packages as simple as an analytical report with results only, to reports as complex as CLP-like data

packages that include a narrative, analytical results, and supportive documentation including all raw data and chain of custody documentation.

STL's facilities also have experience producing reports for the Navy (NFESC), Air Force (AFCEE), Army Corps of Engineers (USACE), New Jersey Department of Environmental Protection (NJDEP) and New York State Department of Environmental Conservation (NYSDEC).

2.3.2 Electronic Deliverables

In addition to hard copy reports, STL can provide data electronically on diskette, CD-ROM, via e-mail or by modem. STL can produce a wide variety of file formats, compatible with all major software programs such as Lotus, Quattro Pro, Excel, Paradox, dBase, Oracle, Access, Word and Word Perfect. Utilizing the Laboratory Information Management System and subsystems, STL is able to produce deliverables with a broad range of data elements in many customized formats. STL has successfully delivered EPA CLP Agency Standard formats and also provides data to AFCEE in ERPIMS and USAEC in IRDMIS.

2.4 CLIENT SERVICES

STL's client services are built on a foundation consistent with the principles of Total Quality Management. Customer focus, support, continuous improvement, and measurement are the defining characteristics of STL's commitment to customer service.

At STL, every customer service representative is committed to understanding and meeting customer expectations. This proactive approach allows STL's personnel to provide solutions to current requirements while anticipating future needs and expectations. From the smallest detail to understanding the 'Big Picture', STL's customer service representatives diligently strive, meet and exceed customer expectations.

2.4.1 Project Management

STL uses a project management system to plan, coordinate, integrate and monitor project activities. Project management personnel work with the operations staff and clients to schedule projects, track progress, and review and produce final reports.

At STL each client is assigned a project manager to oversee the successful completion of their project from the definition stage to the final submittal of data. STL's project managers have extensive experience in the environmental field and are well equipped to handle both business and technical matters.

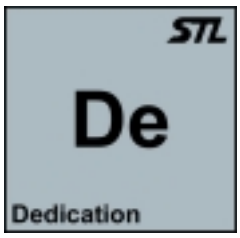
Prior to the initiation of all large projects, STL establishes a project team of qualified laboratory professionals. Once the project team has been established, a kick-off meeting is held to ensure that all team members have an understanding of the technical and administrative requirements of the project. Sample schedules, turnaround time and reporting requirements are communicated and delegation of responsibilities is established. If necessary, laboratory resources are shifted to ensure that all project requirements are met.

STL strongly encourages our clients to visit the laboratory and hold formal or informal sessions with employees in order to effectively communicate their needs on an ongoing basis.

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

SECTION 3



RESOURCES

3.1 FACILITIES

STL's facilities are designed for efficient, automated high-quality operations. Super minicomputers and PC networks are used for sample tracking, work scheduling, status reporting and electronic data transfer to clients.

All STL facilities are 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. STL also provides and requires the use of certain items of protective equipment including safety glasses, protective clothing, gloves, respirators, etc.

Because of the sensitivity of the work STL performs, access to all STL facilities is controlled through various security systems including locks, passwords, electronic access cards and manned reception areas.

Table 3A - Laboratory Square Footage

Facility	Square Footage
STL Sacramento	66,000
STL Savannah	55,000
STL Denver	54,000
STL North Canton	53,000
STL Chicago	51,000
STL Austin	43,000
STL Burlington	36,000
STL Richland	33,000
STL Buffalo	32,000
STL St. Louis	30,800
STL Edison	30,000
STL Pittsburgh	30,000
STL Knoxville	29,400
STL Houston	28,000
STL Los Angeles	27,000
STL Tallahassee	22,000
STL Anchorage	20,000
STL Pensacola	18,000
STL Connecticut	17,000
STL Baltimore	17,000
STL Mobile	14,000
STL Tampa West	12,000
STL Corpus Christi	12,000
STL Tampa East	11,200
STL Billerica	10,000
STL Westfield	10,000
STL Miami	9,000
STL Newburgh	8,000
STL Valparaiso	7,000
TOTAL	**757,400**

3.2 INSTRUMENTATION

STL has made a corporate commitment to routinely update and automate instrumentation. STL's facilities contain instrumentation and equipment for analyzing water, wastewater, solid waste, soil, sludge, tissue and air samples.

STL employs a system of preventative maintenance in order to ensure system up time, minimize corrective maintenance costs and ensure data validity. All routine maintenance is performed as recommended by the manufacturer and may be performed by an analyst, instrument specialist or outside technician.

Table 3B - Equipment List Summary

Number of Units	Type of Instrumentation
401	Gas Chromatographs (GC)
240	Gas Chromatographs/Mass Spectrometers (GC/MS)
197	Alpha Spectrometers
81	Atomic Absorption Spectrophotometer (AA)
79	Gas Proportional Counter
70	Inductively Coupled Argon Plasma Emission Spectrophotometers (ICP)
40	High Performance Liquid Chromatographs (HPLC)
34	Infrared Spectrophotometers (IR)
34	Cold Vapor Atomic Absorption Spectrophotometer (CVAA)
33	Ion Chromatograph (IC)
30	Wet Chemistry Autoanalyzer
29	UV-Visible Spectrophotometer
27	Total Organic Carbon Analyzer (TOC)
24	Total Organic Halogen Analyzer (TOX)
8	Gamma Spectrometer
7	Liquid Scintillation Detector
6	High Resolution Gas Chromatographs / Low Resolution Mass Spectrophotometers (HRGC/LRMS)
5	High Resolution Gas Chromatographs / High Resolution Mass Spectrophotometers (HRGC/HRMS)
4	Inductively Coupled Argon Plasma Emission Spectrophotometers – Mass Spectrometers (ICP-MS)
4	Kinetic Phosphorescence Analyzer
2	Transmission Electron Microscope (TEM)
2	Liquid Chromatography Mass Spectrophotometers (LCMS)
1	Fourier Transform Infrared Spectrophotometer (FTIR)
1	Scanning Electron Microscope (SEM)

3.2.1 Capacity

STL monitors capacity throughout its group of laboratories on a weekly basis. This monitoring allows STL to best utilize the resources available and schedule projects accordingly to ensure that client deadlines are met.

3.3 PERSONNEL

STL's staff of over 2000 professionals includes analytical chemists, microbiologists, quality assurance specialists, computer systems analysts, environmental technicians, client services staff, project managers and field personnel. STL is proud of its highly qualified and professional staff. Their development is valued as the key to success.

STL's Corporate structure is outlined in STL's Quality Management Plan (QMP), referenced in Section 4. The QMP describes Corporate management's role and responsibilities. Members of the corporate staff set the quality standards by which STL's business operations and personnel strive to achieve. Description of management's capabilities and experience is provided.

Each STL facility is under the supervision of a Laboratory Director who is responsible for the day to day operations of the laboratory facility. In addition, each facility has a Quality Assurance Manager who is responsible for overseeing the QA program of the laboratory. STL's Laboratory Directors and Quality Assurance Managers have an average of over 16 years of experience in the environmental field.

STL Savannah Key Personnel					
Name	**Education Level (Degree/Major) Institution**	**Date Hired**	**Dept. Position Held**	**STL SAVANNAH Experience Total/Position (Yrs)**	**Total Related Experience (Yrs)**
General Wet Chemistry Microbiology					
Blackshear, Kim	BS/BIO Savannah State College	9/20/93	Lead Chemist	7/7	7
Davis, Jr , Robert W	BA/ENV HEALTH University of Georgia	1/31/96	Lead Chemist	4/3	6
Hornsby, Theresa	BS/BIO Georgia Southern University	8/31/98	Department Manager	7/1	9
Sample Custody					
Sicay, Elizabeth R	AA/CHEM TECH Savannah Technical Institute	10/13/97	Sample Custody Supervisor	9/2	9
Inorganics – Metals					
Bryant, Daphine	BS/BIO Livingstone College	6/15/87	Lead Chemist	13/9	18
Madden, Dagmar	BA/CHEM Heinrich-Lauz Schule II	6/8/87	Analyst	13/9	13
Norwood, Susan H	BS/CHEM Armstrong Atlantic State University	7/29/88	Production Supervisor	12/8	12
Redmond, Robert	BS/CHEM Georgia Institute of Technology	3/11/92	Lead Chemist	8/5	17
Semivolatile Gas Chromatography and Liquid Chromatography					
Bearden, Laura C	BA/CHEM Southern Illinois University	8/19/91	Lead Chemist	9/9	9
McFall, Sametria	BS/BIO Savannah State College	8/1/94	Analyst	6/4	6
Mosely, Carol	BS/Medical Technology Georgia Southern University	10/30/95	Analyst	4/2	4
Semivolatile Mass Spectrometry					
Bearden, Robert	ST/ENG Motlow State Community College	8/27/90	Lead Chemist	10/10	10
Carter, Elizabeth	BS/BIO Appalachian State University	3/9/92	Lead Chemist	8/8	8
Humphreys, Kimberly	BS/MICROBIO University of Georgia	5/10/93	Production Supervisor	7/3	7
Volatile Gas Chromatography					
Jakubsen, Melanie	BS/CHEM Armstrong Atlantic State University	6/19/89	Production Supervisor	11/11	13
Jones, Abbie	BS/SCI North Carolina State University	5/11/92	Lead Chemist	8/5	11
Volatile Mass Spectrometry					
Grimm, Paul	BS/ANMSC University of Georgia	2/12/93	Production Supervisor	7/3	7

Figure 3A – Corporate Management

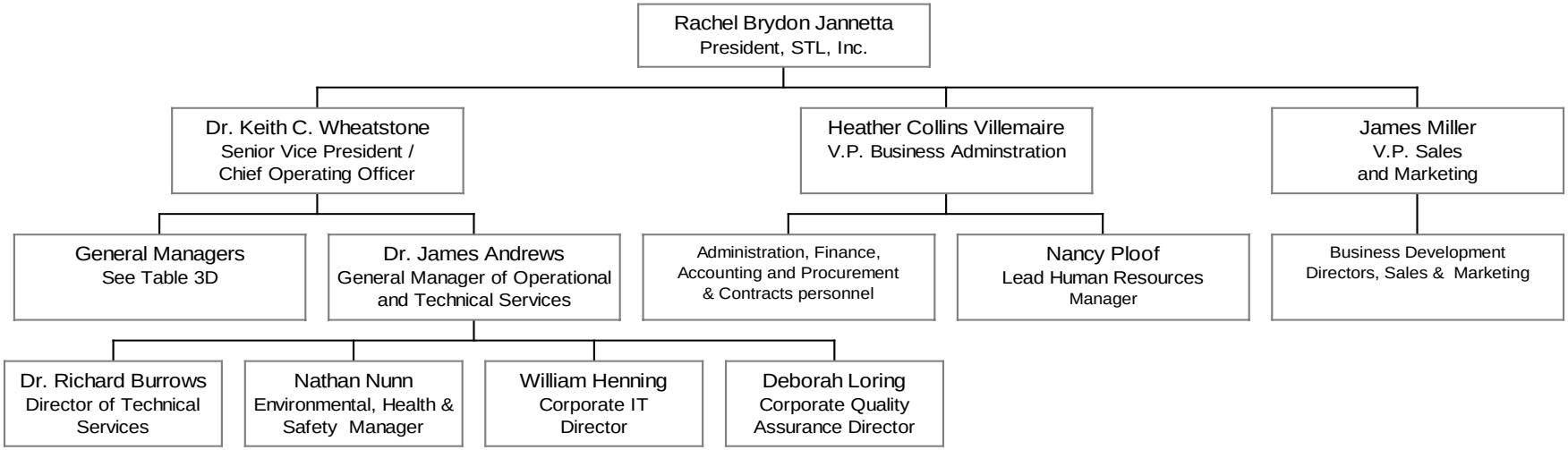


Table 3C – Corporate Management

Name	Title	Degree	Experience
Rachel Brydon Jannetta	President	Fellow of the Chartered Association of Certified Accountants (FCCA)	Ms. Jannetta has been President of STL for 7 years, responsible for the development and implementation of STL's US acquisition and growth strategy. Ms. Jannetta's business previous experience dates back to 1976 - 10 years in general management, 6 years in the financial sector.
Dr. Keith C. Wheatstone	Senior Vice President / Chief Operating Officer	Ph.D. Analytical Chemistry	Dr. Wheatstone has 38 years of environmental laboratory experience, 26 years of which has been with STL. Dr. Wheatstone has held various management positions including Director of Operations for STL-UK. Dr. Wheatstone has worked for STL in UK, India, Puerto Rico, Slovakia, Italy and Germany.
Heather Collins Villemaire	V.P. Business Administration	B. S. Business Administration	Ms. Villemaire has 13 years experience in Business Administration with nine years in the environmental testing industry. Her previous experience includes various management and officer positions including Director of Business Administration, Controller and Director.

Name	Title	Degree	Experience
James Miller	V.P. Sales & Marketing	B. S. Chemical Engineering / Journalism	Mr. Miller has over 14 years experience in the environmental laboratory industry. He was an officer of Recra Environmental where he worked for eight years. In addition, Mr. Miller worked at a major chemical company for six years.
Dr. James Andrews	General Manager of Operational and Technical Services	Ph.D. Nutritional Biochemistry	Dr. Andrews has over 30 years of experience; beginning Savannah Laboratories in 1975. He participated in research and method development with EPA and has worked with NELAC. Dr. Andrews is a member of a National Academy of Sciences subcommittee and affiliated with the ACIL.
Deborah Loring	Corporate QA Director	B. S. Chemistry	Ms. Loring has a total of 16 years experience in the environmental laboratory field that includes bench chemist, laboratory manager, regional QA manager and laboratory director.
Nathan Nunn	Environmental, Health & Safety Manager	B. S. Chemistry	A member of the American Society of Safety Engineers, Mr. Nunn has over 15 years of experience designing, implementing and managing programs to comply with Federal, State and Local regulations and employee training.
William Henning	Corporate IT Director	MBA; B. A. Sociology	Mr. Henning has worked in Information Technologies for over 22 years, 19 in the pharmaceutical industry.
Dr. Richard Burrows	Director of Technical Services	Ph.D. Analytical Chemistry	Dr. Burrows has over 18 years of experience in analytical chemistry in academic and commercial settings. He specializes in chromatography and mass spectrometry. Dr. Burrows has held management positions at a number of labs specifically Senior Scientist and Directory of Technology.
Nancy Ploof	Lead Human Resources Manager	Degree in progress	Ms. Ploof has over 3 years experience as Human Resource Manager working directly with local HRM and Coordinators. Past experience has included all aspects of HR, payroll processing, staff recruitment and Affirmative Action Officer. Ms. Ploof has a total of 11 years of Accounting experience.

Table 3D – General Managers

Name	Location Responsibility	Degree	Experience
Thomas Stephens	Mobile, Pensacola, Tallahassee, Tampa West	B. S. Chemistry	Mr. Stephens has over 31 years experience in environmental laboratory business, beginning with 15 years as chemist for Florida agencies, and 15 years as Lab Director and VP of Savannah Laboratories.

Name	Location Responsibility	Degree	Experience
Dr. Charlie Carter	Austin, Corpus Christi, Houston, Richland, St. Louis	Ph.D. Environmental Chemistry	Dr. Carter has over 20 years experience in the environmental testing industry. He is currently a director of the ACIL environmental section. Academic background includes studies in environmental chemistry and analytical chemistry and philosophy.
Jeff Graham	Knoxville, Tampa East, Miami	B. S. Chemistry	Mr. Graham has 16 years of experience in the environmental laboratory field.
Roger Freize	Anchorage, Los Angeles, Sacramento	MBA; B. S. Petroleum Engineering	Mr. Freize has over 15 years experience in the environmental laboratory field. Experience includes Lab Supervisor, Sales, Systems Manager, Laboratory Manager and General Manager.
Mark Nebiolo	Baltimore, North Canton, Pittsburgh	M. S. Biology	Mr. Nebiolo has more than 20 years experience in the environmental testing industry. He is experienced in all aspects of lab management operations and business development.
Ron Bayer	Billerica, Burlington, Connecticut, Newburgh, On-Site Laboratories, Westfield	M. S. Environmental Science	Mr. Bayer was the former owner and founder (in 1974) of Envirotest Labs in Newburgh, NY. He has 26 years of experience in the environmental testing field.
Robert Wyeth	Buffalo, Denver	M. S. Analytical Chemistry	Mr. Wyeth has over 30 years experience in the commercial environmental testing industry. He is an active participant in ACIL, WEF, NELAC and other related industry activities.
Daniel Santaniello	Edison	B. S. Electrical Engineering	Mr. Santaniello has over seven years of experience in the laboratory services business specializing in Information Systems and Operations. He was Corporate MIS Director for one and one half years.
Mike Healy	Chicago, Valpariaso	B. S. Environmental Biology	Mr. Healy has over 17 years experience in the environmental laboratory business that includes bench chemistry, field sampling, project management and laboratory management.
Dr. Jack Tuschall	Savannah	Ph.D. Environmental Engineering Science	Dr. Tuschall has extensive experience in managing full-service analytical laboratories, analytical method development; trace organic, inorganic and mixed-waste analyses; design and implementation of quality assurance programs.

Table 3 E - Key Laboratory Personnel

Name	Title	Laboratory	Degree	Years of Exp.
Jesse Smith	Laboratory Director	STL Mobile	B.S. Science	28
Charles Newton	QA Manager	STL Mobile	B.S. Biology	8
Jeff Curran	Laboratory Director	STL Connecticut	M.S. Chemistry	21

Name	Title	Laboratory	Degree	Years of Exp.
Marsha Culik	QA Manager	STL Connecticut	A.A.S. Applied Science	17
Susan Rembert	Laboratory Director	STL Pensacola	B.S. Geology	13
Kelly Hered	QA Manager	STL Pensacola	B.S. Chemistry	13
Michael Spitzer	Laboratory Director	STL Miami	B.S. Chemistry	16
Theresa Giglio	QA Manager	STL Miami	B.S. Biology	12
Janet Pruitt	Laboratory Director	STL Tallahassee	M.P.H. Environmental Health	34
Steven Worrell	QA Manager	STL Tallahassee	B.A. Interdisciplinary Natural Science	8
Andre Rachmaninoff	Laboratory Director	STL Tampa West	B.S. Biology	21
Kathleen Raftery	QA Manager	STL Tampa West	QA/QC-Food, Pharmaceutical and Environmental	18
Dr. Jack Tuschall	Laboratory Director	STL Savannah	Ph.D. Environmental Engineering Science	28
Kirstin McCracken	QA Manager	STL Savannah	B.A. Geography	7
Mike Healy	Laboratory Director	STL Chicago	B.S. Environmental Biology	17
Raymond Frederici	QA Manager	STL Chicago	M.B.A; B.S. in Environmental Biology	18
John O'Donnell	Acting Lab Director	STL Baltimore	Science Course Work & Work Experience	18
Mimi Uhlfelder	QA Manager	STL Baltimore	M.S. Chemical Oceanography	31
Dr. Michael Wheeler	Laboratory Director	STL Billerica and STL Westfield	Ph.D. Inorganic Chemistry	13
Peter Bowser	QA Manager	STL Billerica and STL Westfield	M.S. Environmental Resource Management	7
Michael Urban	Laboratory Director	STL Edison	B.S. Chemistry	24
Madhuri Dave	QA Manager	STL Edison	B. S. Microbiology / Chemistry	16
Susan Tinsmith	Laboratory Director	STL Buffalo	B.S. Chemistry	11
Chuck Huber	QA Manager	STL Buffalo	B. A. Chemistry	6

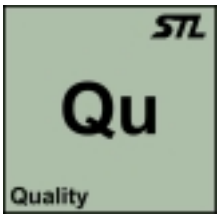
Name	Title	Laboratory	Degree	Years of Exp.
Lou Cercone	Laboratory Director	STL Newburgh	B.A. Biology	22
Patricia Chany	QA Manager	STL Newburgh	B.S. Environmental Chemistry	19
Chris Ouellette	Laboratory Director	STL Burlington	B.S. Biology	9
Kim Watson	QA Manager	STL Burlington	B.S. Environmental Engineering	18
Peter Law	Laboratory Director	STL On-Site Technologies Division	B.S. Environmental Sciences	18
Emanuel Hignutt	Laboratory Director	STL Anchorage	B. A. Chemistry	15
Dr. Rolland Grabbe	QA Manager	STL Anchorage	Ph.D. Chemistry	20
Sharon Mertens	Laboratory Director	STL Austin	B. S. Medical Technology	21
Alice Wusterhausen-Colt	QA Manager	STL Austin	B.S. Zoology	25
Chip Meador	Laboratory Director	STL Corpus Christi	M. S. Botany / Ecology	19
Olga McDonald	QA Manager	STL Corpus Christi	B. S. Medical Technology	9
Tim O'Shields	Laboratory Director	STL Denver	B. S. Chemistry	20
Larry Penfold	QA Manager	STL Denver	Chemistry Course Work & Work Experience	25
Norm Flynn	Laboratory Director	STL Houston	M. S. Animal Nutrition	20
LaDonna Kibler	QA Manager	STL Houston	B. S. Biology & Chemistry	15
Tom Yoder	Laboratory Director	STL Knoxville	B. S. Business Administration	14
Dr. Chris Rigell	QA Manager	STL Knoxville	Ph.D. Biochemistry	20
Elizabeth Winger	Laboratory Director	STL Los Angeles	B. S. Biological Sciences	8
Sevdâ Aleckson	QA Manager	STL Los Angeles	B. A. Chemistry	19
Christopher Oprandi	Laboratory Director	STL North Canton	B. S. Chemistry	12
Opal Davis-Johnson	QA Manager	STL North Canton	B. A. Chemistry	14

Name	Title	Laboratory	Degree	Years of Exp.
Rusty Vicinie	Laboratory Director	STL Pittsburgh	B. S. Chemistry & Biology	13
Patrick Conlon	QA Manager	STL Pittsburgh	MBA; B. S. Chemistry	18
Kevin Bull	Laboratory Director	STL Richland	M. S. Chemistry	19
Jodie Carnes	QA Manager	STL Richland	B. S. Bacteriology	13
Eric Redman	Laboratory Director	STL Sacramento	M. S. Chemical Physics	20
Pamela Schemer	QA Manager	STL Sacramento	B. S. Chemistry	10
Bill Decklmann	Laboratory Director	STL St. Louis	B. S. Biology	13
Brian Crandall	QA Manager	STL St. Louis	M. S. Health Physics	16
Rich Detar	Laboratory Director	STL Tampa East	B. S. Chemistry	12
Dr. Shibu Paul	QA Manager	STL Tampa East	MBA, Ph.D. Environmental Chemistry	11
Les Arnold	Laboratory Director	STL Valparaiso	Environmental Technologies	21
Linda Moore	QA Manager	STL Valparaiso	B. A. Chemistry	14

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

SECTION 4



QUALITY ASSURANCE

4.1 QUALITY ASSURANCE POLICY

It is STL's policy to:

- provide high quality, consistent, and objective environmental testing services that meet all federal, state, and municipal regulatory requirements.
- generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use.
- provide STL clients with the highest level of professionalism and the best service practices in the industry.
- build continuous improvement mechanisms into all laboratory, administrative, and managerial activities.
- maintain a working environment that fosters open communication with both clients and staff.

4.1.1. Objectives of STL Quality System

The goal of the STL Quality System is to ensure that business operations are conducted with the highest standards of professionalism in the industry.

To achieve this goal, it is necessary to provide STL clients with not only scientifically sound, well documented, and regulatory compliant data, but also to provide the highest quality service experience available in the industry. STL's Quality System is designed to provide a framework for continuous improvement within the organization, minimize systematic error, and to encourage constructive, documented problem solving.

4.1.2 Management Commitment to Quality Assurance

STL management is committed to providing the highest quality data and the best overall service in the environmental testing industry. To ensure that data produced and reported by STL meet the requirements of its clients and comply with the letter and spirit of municipal, state and federal regulations, STL maintains a Quality System that is clear, effective, well communicated and supported at all levels in the company.

The elements that comprise STL's Quality System are outlined in detail in the Quality Management Plan. This document can be obtained by contacting any STL facility.

4.1.3 Proficiency Testing

STL analyzes Proficiency Test (PT) samples as required for accreditation and as outlined in the National Environmental Laboratory Accreditation Conference (NELAC). Each STL facility participates in the PT program semi-annually for each area of testing and matrix (e.g. organics, inorganics, microscopy, radiological, microbiological; aqueous and drinking water) for which it is certified. In addition to the PT program required for NELAC accreditation, STL facilities participate in a number of additional PT programs such as the EPA Contract Laboratory Protocol (CLP) Performance Evaluation program and the Navy and Army Corps of Engineers Laboratory Assessment program.

4.1.4 Double Blind Performance Evaluation

Each STL facility also participates in a double blind performance program annually, which is administered by the Corporate QA Manager. An external vendor is contracted to submit double blind samples to each STL facility. Both the level of customer service and the accuracy of the test results are assessed objectively by the external contractor, who provides a detailed report to the Corporate QA Director and each of the STL facilities. This is administered as a double blind program in order to assess all facets of STL operations.

4.1.5 Client Confidentiality and Proprietary Rights

Data and sample materials provided by the client or at the client's request, and the results obtained by STL, are held in confidence subject to any disclosure required by law or legal process. STL's reports, and the data and information provided therein, are for the exclusive use and benefit of the client, and are not released to a third party without written consent from the client.

4.1.6 Record Retention and Archival

STL has developed a formal record retention policy in its Corporate Quality Management Plan that outlines the period of time various record types must be archived. Archives are indexed such that records are accessible on either a project or temporal basis. Archives are protected against fire, theft, loss deterioration and vermin. Electronic records are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to archives is controlled and documented.

4.2 CERTIFICATIONS

STL is certified/qualified in 50 states and several federal programs. It is important to note that some states only certify in-state labs, but accept results from out-of-state labs as long as they are certified in the state in which they are located and pass WP/WS studies. A listing of all of STL's certifications can be found in Appendix B.

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

SECTION 5



PROJECT EXPERIENCE

5.1 PROJECT DESCRIPTIONS

Although their trading names may have changed, the underlying strength of STL's laboratories, personnel and commitment, has remained the same. STL's laboratories have vast experience with all matrices, methods, protocols and environmental programs working on behalf of industry, commerce and government.

5.1.1 Federal/State/Local Agencies

Client	Project Type	Description
USEPA	Contract Laboratory Program	Several STL facilities were past participants in the USEPA Contract Laboratory Program for both organic and inorganic Statements of Work.
Engineering Firm	Total Environmental Restoration Contract	STL is providing environmental analyses in support of several USACE Total Environmental Restoration Contract (TERC) Programs including Omaha, Kansas, Savannah and New England. Analytical services are being performed on sites in Stratford, CT, Norwood, MA, Red Stone Arsenal, Joliet Army Ammunition Plant and Silresim Superfund Site and include the performance of pest/PCBs, semivolatiles, volatiles, trace explosives and CWM Degradate Agents and wet chemistry methods.
Large Municipality	Permit Monitoring	STL supports a municipality's 301(h) permit monitoring efforts involving the analysis of marine sediments and biota for volatiles, semivolatiles, OP Pesticides/PCBs, trace metals and cyanide. Methods include vacuum distillation for the preparation of samples for volatiles and adaptations of Methods 1624 and 1625 for the analysis of volatiles and semivolatiles by isotope dilution. Additional analytical services include BOD, COD, volatile solids, dissolved sulfide and oil and grease on marine sediments.
Environmental Engineering Firm	NYSDEC – Analytical Services Protocols	STL analyzed waters, soils, leachates, sediments and tars for the entire list of EPA target analytes in accordance with NYSDEC ASP protocols and reporting requirements. This project required close communication with the NYSDEC to establish appropriate clean-up methods for sample extracts to prevent sample dilution due to matrix interferences.
Municipal Airport Authority	Stormwater Monitoring	Beginning in 1993, the EPA mandated a large stormwater monitoring program to determine the source of contamination of a major Florida river. The sampling events take place on both a monthly basis and as-needed basis, utilizing a full range of stormwater parameters.
Airport Authority	Groundwater and Soil Remediation	A large airport was under a mandate to assess and begin remediation of a complete airport facility. Because of the mass amounts of samples, and the limited time frames allowed to sample, all groundwater samples needed to be analyzed within two calendar days and all soils needed to be analyzed within three calendar days. STL was able to supply results consistently utilizing their seven day a week, 16-hour day work schedule. The main constituents analyzed on this project were volatiles, semivolatiles and metals.
United States	Various	STL currently holds a contract to support cooperative projects with the Air Force, Army, Navy, National

Client	Project Type	Description
Geological Survey (USGS) Cooperative Projects		Guard, EPA, BLM, other federal state and local agencies as well as research projects within USGS. Projects include hazardous waste site investigations under RCRA and CERCLA; wastewater and stormwater projects under CWA; drinking water projects under SDWA; ocean dredging and disposal and various research projects. To date, well over 20,000 surface water, groundwater, drinking water, soil, sediment, waste, air and biota samples have been analyzed for USGS. Analyses include VOC, SVOC, pesticides, herbicides, metals, explosives, dioxins, PCB Congeners, PAH, air toxics, radiochemistry and wet chemistry.
NASA	NPDES, RCRA	Performing according to NASA statement of work, STL has performed various on-going projects at sites at the Kennedy Space Center in Florida since 1997.
Consulting Firm	RI at Alabama Arsenal; NPL Sites	Several STL labs provide support of on-going investigations at numerous arsenal sites, some of which were added to the NPL by the EPA in 1994. In total, the labs analyze samples from more than 90 sites. Some of these sites were known chemical warfare agent sites. Samples are analyzed using SW-846 methods for a wide variety of parameters including: VOC, SVOC, Pesticides/PCBs, explosives, Metals, TCLP, GRO, DRO, TPHC, Thiodiglycol, White Phosphorous and wet chemistry parameters. Full Deliverable package includes EDD.
Environmental Consulting Firm	Site Cleanup of Major Chemical Agent Disposal Facility	This high-profile and politically sensitive project, under the intense scrutiny by the ACOE, USEPA and the state of Utah, was awarded to STL because they were one of the few laboratories in the country best qualified to handle the scope and complexity of the project. This includes incineration testing experience, technical expertise in performing all tests required and unmatched capacity and is based on STL's specialization in air toxics, dioxin and explosive analyses and leadership in MS and LCMS analyses.

5.1.2 Department of Defense

Client	Project Type	Description
Consulting Firm	U.S. Navy – Pearl Harbor	STL supported a consulting firm and the US Navy on a large scale study at Pearl Harbor. As part of this project approximately 110 sediment, 30 waters and 45 tissues were analyzed for Alkyl Tin, Chlorinated Herbicides, Metals, Low-level SVOA, SIM PAH, P/PCB, Triazines/OP Pesticides, Explosives, acid volatile sulfides and simultaneously extracted metals (AVS/SEM), TOC and Carbamates.
Engineering Firm	U.S. Navy CLEAN – Atlantic Division	STL has performed analyses from 12 different Naval facilities, for a total of 24 phases. Services provided have included full CLP analyses, explosives, wet chemistry and geotechnical support. Electronic Data Deliverables (EDDs) have also been supplied.
Engineering Firm	U.S. Army Corps of Engineers	STL has been supporting a USACE project at Plattsburgh Air Force Base for two years. STL has performed full organic and inorganic CLP analyses on soil, groundwater and liquid matrices.

Client	Project Type	Description
U.S. Army Corps of Engineers	Quality Assurance Laboratory	STL has been recognized by the USACE as a Quality Assurance laboratory since 1993. USACE QA laboratories are chosen by a technical committee based on the laboratory's ability to meet a rigorous validation process that exceeds the requirements set for standard USACE validation.
U.S. Army Corps of Engineers	Landfill Closure	As part of a clean-up from a major hurricane, the U.S. Army Corps of Engineers contracted a national environmental firm to sample, analyze, and have a final report to them within 30 calendar days. A full range of parameters needed to be analyzed on this project. STL was able to provide all the required analytical services in 14 calendar days.
Engineering Firm	AFCEE	STL has provided support during several different project phases at Pease AFB. Many of the initial project samples were analyzed for GC/MS volatiles, semivolatiles, EDB, metals, PAHs, pesticides and wet chemistry analyses for nitrate, sulfate, alkalinity and free CO2. A groundwater remediation process for Site 32 was conducted on a rapid turn basis to monitor a treatment system. Analyses were conducted on volatiles, semivolatiles, pentachlorophenol, TSS and metals on a 48 hour turn basis for 6 weeks.
Engineering Firm	AFCEE	STL has provided analytical support for a confidential Air Force Plume Response Project since November 1996. Over 20,000 inorganic and organic analyses have been performed on water and sediment matrices including GC/MS volatiles and semivolatiles, EDB, pesticides/PCBs, herbicides, metals and more than 20 wet chemistry parameters.
Consulting Firm	U.S. Navy CLEAN II	STL has performed a full suite of organic and inorganic analyses following CLP and SW-846 protocols at over 40 Naval facilities on the west coast. Fully validatible and summary packages have been provided including EDDs in customized formats.
Consulting Firm	U.S. Navy BRAC – Northern Division	STL has been providing analytical services to a major consulting firm under an ID/IQ contract with the Navy Northern Division at over 30 Naval installations. Analyses performed have included a full suite of organic and inorganic constituents on aqueous, solid and tissue matrices in support of RI/FS, IRP, and Risk Assessment Programs.
Consulting Firm	AFCEE	STL has been providing analytical services in support of an RI/FS at Andersen AFB, Guam since 1994. All analyses have followed the strict guidelines of SW-846 protocols and the Andersen AFB Basewide Sampling and Analysis Plan (which STL has assisted with development). Matrices of a very complex nature, including monitor lizard tissue, have been analyzed under strict quality control limits. All data generated has been provided in "CLP-like" packages and electronically uploaded into ERPIMS.
Consulting Firm	AFCEE	Under guidance of the Hickam AFB Basewide Quality Assurance Plan and the AFCEE IRP Manual, STL has provided analytical support at several Air Force Bases in the Pacific Rim. Analyses of organic and inorganic constituents were performed on soil, sediment, groundwater and surface water samples.
U.S. Army Corps of Engineers	Various	STL has provided analytical support for the U.S. Army Corps of Engineer's site investigations nationwide since 1986. STL has routinely evaluated various sample matrices such as water, soil/sediment, plant, animal and fish tissues and air from over 16 USACE sites.

Client	Project Type	Description
Consulting Firm	AFCEE - RFI and Risk Assessment	Analytical support was provided to the RFI and Risk Assessment efforts that were performed simultaneously at Altus AFB. This was one of the largest funded projects for fiscal 1999. AFCEE protocols were followed and modified for APX IX analytes. Most of the 2,250 samples were analyzed for VOC, SVOC and metals, with Full deliverable data packages in hard copy and CD-ROM formats. EDDs were submitted in AFCEE's ERPIMS 4.0 format.
Major Consulting Firm	Permit to Continue Operations at Chemical Agent Disposal System	STL supported analysis at a hazardous waste treatment facility for the destruction of the chemical agent munitions stockpile in the South Pacific. The disposal system was designed to dispose of nerve agents, blister agents, drained munitions, contaminated refuse, bulk containers, liquid wastes, explosives, propellant component and other agent related waste. STL provided analytical support for the incinerator trial burns for the purpose of obtaining a new permit to continue operations that included liquid incinerator, metal parts furnace, dunnage incinerator and the deactivation furnace system. Analyses includes Dioxin/furans by Method 23, 8280 and 8290, PCB congeners by High resolution GS/MS, VOC, SVOC. Total chromatographic organics, metals by ICP/MS, Hexavalent Chromium by Method 0061, HCl and HF.

5.1.3 Industrial

Client	Project Type	Description
Fortune 100 Chemical Company	Remedial Investigation – Superfund Site	STL analyzed over 900 soil samples for TCL/TAL parameters over a nine-month period using CLP and SW-846 methodologies. Several types of extract clean-up procedures were required on many samples due to high levels of contamination. STL continues to provide analytical support to the company for on-going remediation and groundwater monitoring.
Oil Refiner	Discharge and Groundwater Monitoring	STL has provided analytical support at a major East Coast refinery. The bulk of the project has required the analysis of discharge water and groundwater for organics and inorganics using EPA 600 series methods. Sludges have been analyzed for TCLP parameters utilizing SW-846 methods.
Electrical Utility	Emergency Response	STL operates a program with a northeastern electrical utility to provide sampling and analysis for any type of spill or environmental incident. Over the last five years, STL has analyzed over 1,000 soil and oil samples for PCBs, and other samples for PAH, volatiles, semivolatiles and GC Fingerprinting.
Semiconductor Manufacturer	Permit Analysis	STL analyzes daily samples for a variety of organic and inorganic parameters that are permitted under SPDES and POTW. Emergency turnaround times of 24 hours or less are frequently required.
Railroad Company	Contract Laboratory	STL performs sampling and analyses on groundwater remediation sites, as well as analyses for consultants on hazardous spills, UST's and site investigations.

Client	Project Type	Description
Oil Company	Contract Laboratory	STL analyzes samples from gas station sites, bulk terminals, production facilities and property transfers. Samples are also analyzed for bioattenuation, air sparging and other remedial techniques. STL works with over 50 consultants on site assessments and investigations.
Southeastern Utility	Sampling and Analysis	STL provides sampling and analyses for a major southeastern utility company. Over 2000 monitoring wells are sampled and tested for metals and volatiles per year.
Soup Company	NPDES Monitoring	STL coordinates the environmental sampling schedule and provides analytical services for approximately 50 sites nationwide for a soup company. The scope of work includes wastewater sampling and analysis and potable water sampling and analysis. Project "Site Summaries" are provided to each site prior to each sampling event providing instructions, parameters, methods, detection limits, reporting requirements, and any specific details that are important to the successful execution of the program. At the completion of each project, the project manager reviews the analyses. If any permit levels are exceeded, immediate notification is given to the client.
Ordnance Manufacturer	Analytical Support	STL provides comprehensive analytical support for a major ordnance manufacturing facility. STL has experience with, and knowledge of, the methods and regulatory requirements associated with explosive residues and other materials common to this facility. EPA and state approved methods for the analysis of water, soil and waste materials are employed.
Chemical Company	Environmental Testing	STL has provided analytical support services in response to programs in support of RCRA, CERCLA, NPDES/CWA and CAA programs. A variety of matrices have been evaluated for environmental compliance, including water, soil/sediment and waste. Routine monitoring programs typically evaluate these matrices for volatile, semivolatile, and inorganic parameters for Appendix IX, TCL/TAL, Priority Pollutants and other target analytes of concern.
Major Steel Maker	Sampling & Analysis, NPDES, RCRA	STL coordinates and performs all aspects of this client's NPDES monitoring including sampling, analysis, data reduction and data reporting to agencies. This NPDES permit is the most extensive in the state of Indiana. STL coordinates all aspects of the company's waste characterization for over 30 waste streams within the facility. STL has developed electronic formats for all agency reporting requirements. STL works with various consulting and engineering firms who provide site investigations and process monitoring.
Major Electronics Company	Waste Characterization & Disposal	Over the past five years, STL has provided extensive waste characterization analysis and disposal decision assistance for a major electronics company. STL has provided customized electronic deliverables to facilities throughout the state of Texas. The laboratory was awarded that company's '*Superior Excellence Award"*

5.1.4 Mixed Waste

Client	Project Type	Description
Engineering Firm	Department of Energy – Mixed Waste	STL had been contracted to provide organic, inorganic and radiochemistry analyses on soil, aqueous, vegetation and air matrices on a weekly basis for waste and radiochemistry classification.
Environmental Engineering Firm	Department of Energy – Mixed Waste	STL was under contract with a large environmental consulting firm for the analysis of mixed waste samples collected from a DOE site in New York under a removal action plan. Approximately 80 samples were submitted for analysis within a two week period. The project included the analysis of soils and waters for CLP organics and inorganics (TCL/TAL); TCLP; and radiological parameters including Gross Alpha/Beta, Gamma Spectroscopy, Strontium 90 and Tritium. Data packages including comprehensive quality assurance information, were reported within four weeks and were fully validated by the contractor's quality assurance team.
Engineering Firm	U.S. Navy CLEAN	STL has analyzed soil, aqueous, waste and fish tissue matrices for organics, inorganics, wet chemistry and radiological analyses. Compliance with CLP and Navy CLEAN analytical protocols and deliverables is mandatory.

5.1.5 Air

Client	Project Type	Description
Oil Company	Air Stripper Monitoring	STL is under contract with an oil company for the analysis of tedlar bags and canisters for BTEX & TPH from UST sites throughout the United States. This work is done in support of air stripper monitoring.
Confidential	Groundwater Vapor Extraction	STL provides monthly canister analyses of a carbon vapor extraction system for Method 8260 volatile organic compounds at an Air Force Base in Ohio. Level III data deliverables are provided.
Confidential	Exposure Monitoring, Risk Assessment	STL provided air analyses for metals and TSP utilizing 8x10" QMA filters, PUF/XAD-2 cartridges for semivolatiles and Summa canisters for volatiles. These analyses were conducted in support of remediation at a Superfund Department of Defense site in Utah.
Major Engineering Firm	Long Term GW Monitoring Program	STL has provided air analysis in support of an AFCEE program that required TO14 and Method 18 analyses since 1993. QAPP development and weekly were reports provided to the consultant. Lab provided AFCEE I and II deliverables.
Major Consulting Firm	Trial Burn Demo Project - Site-Specific Risk Assessment	STL was under contract to evaluate and perform modifications to EPA methods on samples exhibiting highly acidic matrix. STL demonstrated that methods applied produced defensible and validatable results. STL assisted in the sample collection of off-gas from the combustion of high level radioactive, nitric acid solution containing hazardous metals. The following sampling trains were employed to collect the off-gas samples: Method 0030/0031, TO-14A, Method 0040 for VOC analysis, Method 0060 for metals, including Mercury; Modified Method 5 (0023A/0010) for PCDD/PCDF and PAH by high resolution

Client	Project Type	Description
		GC/high resolution MS; Modified Method 0010 for SVOCs.

5.1.6 Geotechnical

Client	Project Type	Description
Engineering Firm	Field Investigation	STL analyzed subsurface soil samples for grain size, bulk density and porosity in support of a groundwater containment field investigation. In addition to the geotechnical parameters, soil samples were also analyzed for TOC, sulfate and cation exchange capacity. Groundwater samples were analyzed for TCL volatiles, TAL metals, cyanide, TSS, BOD, TOC and chloride.
Engineering Firm	Site Investigation	STL is currently providing analytical support to an engineering firm for their work at a site on the Housatonic River. As part of this project, STL is analyzing approximately 140 soils per month for particle size using ASTM Method D422.

5.1.7 On-Site Technologies Division

Client	Project Type	Description
Military Installation	DOD Site Investigation	STL's 45-foot mobile laboratory, equipped with GC/MS and a dual ECD GC, provided on-site analysis at a DOD site investigation. Analyses consisted of volatiles by 8260 and EDB by 504.1 to determine site assessment and plume delineation activities conducted at this military installation. STL's mobile laboratory provided data on 1 hour and 24 hour turnaround times. A complete CLP-type data package and electronic deliverables were provided every seven days. Average sample volume was 25 samples per day, with some days requiring the analysis of up to 50 samples.
Groundwater Pump & Treat Facility	USEPA NPL Superfund Site Remediation Project	STL is responsible for the management of this USACE validated on-site laboratory at a Superfund Pump & Treat Facility. Staffed with six full-time chemists, analyses of volatile and semivolatile organic compounds by GC/MS, pesticides by GC/ECD, metals by ICAP and selected wet chemistry parameters are completed. All results are reported with five days of sample receipt.
New England Municipality	USEPA NPL Superfund Site Monitoring	This two-phase project consisted of STL's on-site laboratory conducting soil analysis for PCBs, lead and copper on a 48-hour turnaround time. Over 3,500 samples were completed during this phase. The second phase was divided between STL's fixed laboratory and mobile laboratories and consisted of 24-hour turnaround time for lead and copper by XRF and GC/FID for PCB analysis.
Engineering Firm	AFCEE	STL's mobile laboratory provided analyses for EPA methods 8270 and 8021 at Griffis Air Force Base. Additional services included the electronic production of AFCEE 1996 QAPP 1.1 reporting forms.

Client	Project Type	Description
Incineration Site	On-Site Characterization	STL provided the on-site characterization of drums, sludge and soils at an incineration site in Swartz Creek, Michigan. Soils were analyzed for organic constituents to verify the remediation effectiveness. STL also provided on-site air analyses for the duration of the project.
Engineering Firm	Sample Acquisition	STL completes all required environmental sampling activities for this regional engineering firm. Services provided include sampling of groundwater, including low-flow technique; soil and sediments; wastewater, including time and flow-proportional composite sampling; surface waters; air and surface wipes. A range of on-site analyses is also performed and all work is completed under strict chain-of-custody protocols.
Various Industrial Firms	Waste Characterization	STL has supported various industrial firms in waste characterization utilizing their mobile laboratory facilities. STL analyzed samples from 13,000 drums of unknown waste materials at a Nashua, New Hampshire site and 15,000 drums of waste materials at a Toms River, New Jersey site.

5.1.8 Aquatic Toxicity

Client	Project Type	Description
Consulting Firm	Superfund Site Monitoring	STL was contracted by a regional environmental consulting firm to provide chronic toxicity testing services as final stage, confirmatory analyses at a remediation project in Massachusetts. Working with the consultant, STL assisted in designing the overall scope of work, including some modified testing protocols specifically designed for this project's needs, which were used to evaluate the remediation efforts recently completed at the project site. All testing protocols utilized for this project were reviewed and approved by the U.S. EPA.
Paper Company	Toxicity Identification (TIE) and Toxicity Reduction Evaluation (TRE) Studies	STL provided an international paper company with a variety of bioassay and chemical analyses relative to Toxicity Identification (TIE) and Toxicity Reduction Evaluation (TRE) studies. Both chronic and acute toxicity testing protocols were utilized and over 1000 screening toxicity tests were performed to monitor process changes within the facility.

5.1.9 Dredged Material Evaluation

Client	Project Type	Description
Consulting Firm	U.S. Army Corps of Engineers and New York/New Jersey Port Authority	STL was contracted to provide analytical support for proposed upland disposal of dredged material to coal pits in Pennsylvania and West Virginia. Analytical services included a full suite of parameters including PCB congeners and SPLP waste characterization on bulk sediment prior to and following stabilization. All activities were performed under the strict quality control guidance of the *Management and Regulation of Dredging Activities in New Jersey's Tidal Waters (Draft, March 1996).*

Client	Project Type	Description
U.S. Army Corps of Engineers	River Bottom Sediments	STL was contracted with the USACE for the analysis of river bottom sediments for pesticides/PCBs and a selected group of metals. Because these materials were bioaccumulative, STL modified methods of sample preparation to meet the USACE's low detection limits.
Consulting Firm	U.S. Army Corps of Engineers	STL developed a Quality Assurance Project Plan and performed chemical analyses of Chesapeake Bay sediment proposed for dredging in 1996 and 1997. Sediment, site water, and elutriate water were analyzed following procedures and target detection limits consistent with EPA guidelines provided in the *Inland Testing Manual (1994)* for dredged material disposal in inland waters.
Consulting Firm	U.S. Army Corps of Engineers	STL provided analytical support at Castle Astoria, New York for proposed ocean disposal of dredged material. Sediment, site water, elutriate water, and tissue matrices were analyzed for metals, pesticides, PCB congeners, PAHs, and TOC following the strict protocols of the *Green Book* and *EPA Region II Regional Implementation Manual.* For this project, STL developed a chelation extraction procedure for metals determination in site and elutriate water to meet all project required detection limits.
Consulting Firm	New York/New Jersey Port Authority	STL, in association with a major consulting firm, was tasked with finding acceptable disposal alternatives for natural red clay found in the navigation channels of the Newark Bay and Staten Island Kills Complex. Biota analyses were performed after a 28-day bioaccumulation period. All analyses were performed in accordance with the EPA Region II Regional Implementation Manual and on an accelerated turnaround.
U.S. Army Corps of Engineers	Dredged Material Evaluation	The U.S. Army Corps of Engineers requested that STL develop the data necessary to determine the continued suitability for ocean disposal of material proposed to be dredged during the construction of the Bayou Casotte Turning Basin and Pass Christian Harbor, Mississippi. Physical characteristics, chemical contamination, and bioaccumulation potential were compared to a reference area in the Mississippi Sound to determine similarity. A full suite of organic and inorganic analyses including AVS/SEM was performed on sediment and tissue matrices.

5.1.10 Tissue Analyses

Client	Project Type	Description
U.S. Army, Rocky Mountain Arsenal	Biota Analysis	Under this contract, STL developed PMRMA approved analytical protocols for the low level determination of organochlorine pesticides, mercury, and arsenic on plant and animal tissue. Electronic data deliverables were provided in accordance with USACE IRDMIS requirements.

Client	Project Type	Description
Consulting Firm	U.S. Navy CLEAN II – Ecological Risk Assessment Support	STL performed chemical analyses on sediment, soil, eluate, elutriate, water, small mammal, and animal tissue samples in support of an onshore ecological risk assessment of the Navy's Mare Island, California facility. This was a very challenging project due to the variety and complexity of matrices and large sample volume received within a very short window. All project and data quality objectives were met within the required timeframe.
Consulting Firm	U.S. Army Corps of Engineers	STL provided analytical services to a major consulting firm in support of a removal action at Fort Totten, New York. Fish, oyster, and mussel tissue were tested for mercury, one of the main contaminants of concern at the site.
U.S. Air Force, Armstrong Laboratories	Public Health & Ecological Risk Assessment Support	STL was contracted to provide analytical services in support of an investigation of the water, sediment, and biotic population quality of Leon Creek, which flows through Kelly AFB. Tissue samples from selected trophic levels in the stream were tested for a full suite of organic and inorganic constituents to calculate Biological Concentration Factors as part of the ecological risk assessment.
Consulting Firm	Ecology Risk Assessment Support	STL performed tissue analysis of fish, earthworms, and small mammals. Strict homogenization procedures were followed in preparation of small mammal analysis due to biohazard potential. Electronic data transfer files were provided in accordance with USAEC IRDMIS requirements.
Consulting Firm	Fish Tissue Analysis	STL evaluated concentrations of arsenic, mercury, PCBs, and organochlorine pesticides in the edible portions of fish from the Potomac and Anacostia River. Analyses were performed as part of the current NPDES permit for the Blue Plains WWTP.
Consulting Firm	U.S. Army Corps of Engineers	STL analyzed fish and shellfish for PCBs and metals to determine exposure to predators and estimate risk to human health. Additionally, tissue analyses were performed after a 28-day bioaccummulation exposure period in support of sediment evaluation activities.
Chemical Company	Fish Samples	STL's client needed to analyze thousands of fish samples of varying species and lipid content over a relatively short period of time. This project required the participation of a large number of laboratories and the client wanted to demonstrate and maintain comparability between all the laboratories' results. STL developed and validated a fish/biological tissue method specifically for this project, including the production of a fish tissue standard reference material for use by all the participating laboratories.
Environmental Firm	Analysis of Tissue	The analytical work provided for this project included the preparation and analysis of tissue from various plants and animals. The animal tissue consisted of that from the whole body homogenization of frogs, earthworms and voles. The analyses included pesticides and PCBs by Method 8080, semivolatile organics by Method 1625 and trace metals by Methods 6010/7471.

Client	Project Type	Description
Consulting Firm	Phase II and IIA Analytical Studies	STL has concluded Phase II and IIA analytical studies for PCB congeners in fish, tissue, soil and water on the Hudson River with a consulting firm as part of the Hudson River PCB Reassessment RI/FS under an ARCS contract with EPA Region II. Dual column capillary GC/ECD was used to quantify close to one thousand samples for over one hundred target congeners. High resolution coring was used in the project and PCB patterns were correlated to historical events on the river.
Various Clients	Various Tissue Analysis	Thousands of tissue samples have been analyzed for various clients over the past ten years. Tissue samples have included various animal tissue and organs from worms, birds, reptiles, caddis fly larvae, bovine blood, human blood serum bovine milk, plant tissue and insects.

5.1.11 Dioxin Analysis

Client	Project Type	Description
Major Paper Mill	Routine Monitoring and Process Control	STL conducts dioxin/furan analyses by Method 1613A and Method 8290 on sediment, influent, effluent, pulp, paper and fish tissues. Analysis of fish tissue has been done annually for a major paper company at various locations since 1988. In general, STL provides more than 100 dioxin/furan analysis on fish tissue sample for several pulp/paper clients throughout the United States.
Wastewater Treatment Plants	Monitoring Programs	STL provides dioxin/furan analysis to various municipalities and sanitation agencies utilizing methods 1613, 8280 and 8290 on aqueous, sludge, ocean sediment matrices. STL's history with some clients date back to 1990.
Consulting Engineers	Air Force Base	STL has provided routine analytical testing including dioxin/furan analysis by Method 8290 on soils, aqueous and liquid waste samples since 1992. All data provided included validatable data packages (CLP-like) and electronic deliverables in IRPMS format.
US Geological Survey	On-going Monitoring	STL provides dioxin analysis using Method 8290 to support on-going monitoring at Lake Tahoe, CA and Lake Mead, NV since 1995. Sediments, waters and semi-permeable membrane dialysates are analyzed.
EPA	Monitoring	STL has provided support for dioxin/furan analyses to Region IX EPA since 1995. Most projects conformed to project-specific QA/QC requirements with CLP-like forms presented in a validatable data package. Method 1613A for soils and waters used.
USDA	Analysis of Livestock Feed	STL provided analytical support to numerous poultry plants, catfish farms and a clay manufacturer involved in an investigation of potentially contaminated livestock and livestock feed. Over 100 samples were analyzed on a quick TAT (2 – 5 days) for 2,3,7,8-TCDD by Method 1613A. Samples included feed, clay, catfish filets, fish oil, eggs, poultry tissue and poultry fat.

5.1.12 Radiochemistry

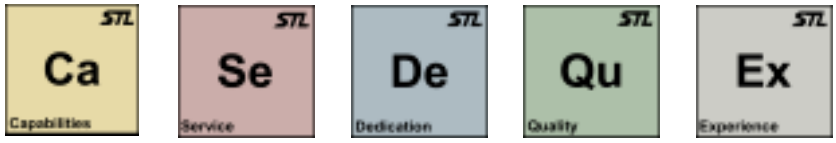
Client	Project Type	Description
U. S. Army Corps of Engineers	FUSRAP	STL, as an MRD validated and NRC approved laboratory, provides analytical support of three FUSRAP sites in St. Louis. In addition to the mixed waste analysis for VOC, SVOC, the laboratory also performs Gamma Spectroscopy and Isotopic Thorium. CLP-like data packages and ASCII data file electronic deliverables were reported. Period of performance: 1991 though 2001.
Department of Energy	Radiochemcial Bioassay and Environmental	DOE administers the bioassay program in support of remediation work for all contractors at the Hanford Site in Richland, Washington and the environmental program in support of site monitoring at Hanford. STL performs analysis for (routine and rapid TAT) of urine and fecal samples for the following isotopes: Americium, Curium, Plutonium, Strontium, Thorium, Tritium and Uranium (Isotopic and Total). STL also conducts analysis of air particulates, water, soil/sediment, milk, wine, vegetation, wildlife, vegetables and silica gel for the following additional isotopes: Carbon, Gamma isotopes, Gross Alpha & Beta, Iodine, Radium and Technetium.
Department of Energy	Radiochemistry Bioassay	STL supports a bioassay program in support of activities at several mid-western sites. The lab performs analysis of urine samples for routine turnaround times for Americium, Curium, Neptunium, Plutonium, Strontium, Technetium, Thorium, Tritium and Uranium (Isotopic and Total).
Department of Energy	Radiochemical Environmental and Waste Characterization	STL performs analysis of water, soil/sediment, building materials, concrete and waste for routine and rapid TAT for the following isotopes: Americium, Carbon, Curium, Gamma Isotopes, Gross Alpha and Beta, Iodine, Neptunium, Plutonium, Radium, Strontium, Technetium, Thorium, Tritium and Uranium (Isotopic and Total). This analyses is in support of the remediation work at the Hanford site in Richland, WA.

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

APPENDIX A

Table of Analytical Services



SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
Volatiles by GC/MS - PP/TCL/TTO																															
Volatiles, Drinking Water	EPA 524.2					x	x	x	x		x	x			x	x	x	x				x		x	x	x	x	x	x	x	x
Volatiles	EPA 624		x		x	x	x	x	x	x	x	x	x			x	x	x	x		x	x			x	x	x	x	x	x	x
Volatiles	SW 5030B / 8260B		x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
Volatiles	SW 5035B / 8260B		x	x	x	x	x	x	x	x	x	x		x	x	x	x		x	x	x	x		x	x	x	x	x	x	x	
Volatiles, Appendix IX List	SW 8260B		x	x	x	x	x	x	x	x	x			x		x		x	x		x	x			x	x	x		x	x	
Volatiles	EPA SOW OLM03.2				x	x	x	x	x			x		x					x		x	x			x	x	x				
Volatiles	EPA SOW OLM04.1 ****					x	x	x											x			x									
Volatiles, Low Level	CLP SAMLC (OLCO2.1)				x	x	x	x				x		x					x			x			x	x	x	x			
Tentatively Identified Compounds (TICs)	EPA 624, SW 8260B, EPA 524.2		x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x	x		x	x		x	x	x	x	x
Volatiles, Isotope Dilution	1624						x																	x	x						
Volatiles, Vacuum Distillation	1624						x																								
Soil Volatile Methanol Prep	State Specific		x	x	x	x	x		x	x		x				x	x	x		x	x	x		x	x		x	x	x	x	x
Volatiles by GC																															
Volatiles, Drinking Water	EPA 502.2					x	x											x													
VOC Purgeable Halocarbons	EPA 601					x	x				x	x	x				x	x	x		x				x		x	x	x		x
VOC Purgeable Halocarbons	SW 5030B / 8021B					x	x			x	x	x				x	x	x	x	x	x			x	x		x		x	x	x
VOC Purgeable Halocarbons	SW 5035 / 8021B					x	x*			x	x					x			x	x	x			x	x					x	
VOC Purgeable Aromatics (BTEX/DCB/CB only)	EPA 602		x		x	x	x	x		x	x	x	x				x	x	x		x			x	x	x	x	x	x		x
VOC Purgeable Aromatics (BTEX/DCB/CB only)	SW 5030B / 8021B		x		x	x	x	x		x	x	x			x	x	x	x	x	x	x			x	x		x	x	x	x	x
VOC Purgeable Aromatics	SW 5035 / 8021B		x			x	x*			x	x				x	x			x	x	x			x	x					x	
VOC (in Series)	601 / 602 / SW 5030B / 8010B / 8021B					x	x				x		x			x	x	x	x		x			x	x		x	x	x		x
Full Method List (1)	SW 5030B / 8021B					x	x									x		x	x	x	x			x	x						
Full Method List (1)	SW 5035 / 8021B					x	x*									x			x	x	x			x	x						
BTEX and GRO (Plus MTBE)	Mod. SW 5030B / 8015B / 8021B			x	x	x	x	x			x				x	x	x	x	x	x	x			x	x	x	x	x	x	x	
BTEX and GRO (Plus MTBE)	SW 5035 / 8015 / 8021B				x	x	x*				x				x	x			x	x	x			x	x	x				x	

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
BTEX (Plus MTBE)	SW 5030B / 8021B			x	x	x	x	x		x	x	x	x		x	x	x	x	x	x	x			x	x	x	x	x	x	x	x
BTEX (Plus MTBE)	SW 5035 / 8021B				x	x	x*			x	x				x	x			x	x	x			x	x	x					
Gasoline Range Organics (GRO) CA LUFT	Mod. SW 5030B / 8015			x	x	x	x	x			x	x	x		x				x		x			x		x		x			x
Gasoline Range Organics (GRO)	Mod. SW 5035 / 8015			x	x	x	x*				x	x				x				x	x			x	x			x		x	x
Gasoline Range Organics (GRO)	SW 5030B/8015B																			x				x		x		x			
Gasoline Range Organics (GRO) Alaska	AK101		x		x										x									x							
Gasoline Range Organics (GRO) WA, OR	NWTPH-GX				x																										
Gasoline Range Organics (GRO) Maine	4.2.17				x																										
Gasoline Range Organics (GRO) Tennessee																												x			
Volatile Petroleum Hydrocarbons	MADEP VPH (2)						x														x				x		x				x
Volatile Petroleum Hydrocarbons (Iowa)	OA-1				x						x															x					
DBCP & EDB	EPA 504.1				x	x	x	x							x	x	x	x		x	x	x		x	x	x	x	x	x		
DBCP, TCP and EDB	SW 8011					x	x									x					x					x		x			
DBCP & EDB	SW 8011				x	x		x								x	x			x	x				x	x	x	x	x			
GC Fingerprint Scan	Mod. SW 8015		x		x	x			x	x		x				x	x	x	x	x	x			x	x		x	x	x	x	x
VOC-DAI-Direct Aqueous Injection	SW 8015B			x		x			x		x	x				x	x	x			x			x	x						x
VOC-DAI - Appendix IX List	SW 8015B			x		x			x							x	x								x						
VOC-DAI (HAPS)	NCASI																x								x						
Alcohols (Select List)	SW 8015B			x			x	x	x		x					x			x						x	x				x	
PHC	QAM-025-10/91																															
Soil Volatile Methanol Prep, each	State Specific		x	x	x	x	x		x	x		x				x	x	x		x	x			x	x	x	x	x	x	x	x
MN - VOC	EPA 465E & 465C, State 465D																		x													
Semivolatiles by GC/MS - PP/TCL/TTO																																
Semivolatiles	EPA 525					x										x	x	x														
Semivolatiles	EPA 625 / SW 8270C	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x	x
Semivolatiles, Appendix IX List	SW 8270C			x	x	x	x	x	x	x	x			x		x		x	x		x	x		x	x	x	x			x	

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
Semivolatiles	EPA SOW OLM03.2				x	x	x	x	x			x		x					x		x	x			x	x	x				
Semivolatiles	EPA SOW OLM04.1 ****					x	x	x											x			x									
Semivolatiles, Low Level	CLP SAMLC (OLCO2.1)					x	x	x	x			x		x					x			x			x		x				
Semivolatiles, Isotopic Dilution	1625						x																								
Polynuclear Aromatic Hydrocarbons (PAHs)	EPA 625 / SW 8270C	x	x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
PAHs GC/MS SIM Low Level	Ion Trap Spy Inject GC/MS													x		x															
PAHs GC/MS SIM Low Level	STL Method	x	x				x																	x							
PAHs GC/MS Scan Low Level	SW 8270C	x	x											x								x		x	x	x					
Tentatively Identified compounds	All Methods	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x		x
Dioxins / Furans	SW 8280													x										x	x						
TCDD	EPA 613													x										x	x						
Semivolatiles by GC																															
Diesel Range Organics (DRO)	Mod. SW 3510/3550/8015B		x	x	x	x	x	x	x	x	x	x	x			x	x	x	x	x	x			x	x	x	x	x	x	x	x
Diesel Range Organics (DRO) CA LUFT	CA LUFT			x	x		x	x			x		x		x				x		x			x		x		x			
Diesel Range Organics (DRO)	WI DNR (13)				x	x													x												
Diesel Range Organics (DRO) Alaska	AK102		x		x										x									x							
Diesel Range Organics (DRO) WA,OR	NWTPH-DX				x																										
Diesel Range Organics (DRO) Maine	4.1.25				x																										
Total Chromatographable Organics (TCO)	Mod. SW 8015			x												x					x			x							
Alaska Residual Range Organics	AK103		x		x										x									x							
Florida Petroleum Residual Organic	FL-PRO (3)				x											x	x		x		x				x		x	x	x		
Texas Total Petroleum Hydrocarbons	TNRCC Method 1005				x					x			x								x				x						
Phenols	EPA 604/SW 8040												x													x					
Polynuclear Aromatic Hydrocarbons	SW 8100				x	x				x						x	x			x	x				x		x		x		
Extractable Petroleum Hydrocarbons	TN EPH (4)					x		x													x				x		x				
Extractable Petroleum Hydrocarbons	MADEP EPH (5)						x														x				x		x				x
Extractable Petroleum Hydrocarbons (Iowa)	OA-2				x						x															x					

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
Alkyltins	STL Method						x																								
Percent Lipids	STL Method																		x					x							
Immunoassay Screen (Herb)	EPA 4015																		x	x											
Phenols	SW8041																					x									
Pesticides / PCBs																															
Pesticides/PCBs Drinking Water	EPA 505															x		x													
Organochlorine Pesticides / PCBs	EPA 608 / SW 8081A/8082		x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x
Organochlorine Pesticides / PCBs, Apx IX List	SW 8081A/8082			x	x	x	x	x	x	x	x			x		x	x	x	x		x	x		x	x	x	x	x	x	x	
Organochlorine Pesticides / PCBs	EPA SOW OLM03.2				x	x	x	x	x			x		x					x		x	x			x	x	x				
Organochlorine Pesticides / PCBs	EPA SOW OLM04.1****					x	x	x											x			x									
Organochlorine Pesticides / PCBs	CLP SAMLC (OLCO2.1)					x	x	x	x													x			x		x				
Organochlorine Pesticides	EPA 608 / SW 8081A		x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
Polychlorinated Biphenyls (PCBs)	SW 8082		x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
Polychlorinated Biphenyls (PCBs) in oil	SW 3580A/8082			x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	
Triazine Pesticides	EPA 619						x				x					x	x								x		x				
Organophosphorous Pesticides	SW 8141A			x	x	x	x		x	x	x	x				x	x		x		x	x			x		x	x			
Organophosphorous Pesticides, Apx IX List	SW 8141A			x			x	x	x		x					x	x	x	x		x	x			x		x				
PCB Congeners	GC/MS Method 680			x								x												x	x						
PCB Congeners	STL Method (8082)				x		x																		x						
PCB Coplanars	STL Method				x		x																	x							
Herbicides																															
Chlorinated Herbicides, Drinking Water	EPA 515.1					x										x	x	x				x			x		x		x		
Chlorinated Herbicides	EPA 615						x				x					x	x		x		x				x		x		x		
Chlorinated Herbicides, Appendix IX List	SW 8150B							x			x					x	x							x	x	x	x		x		
Chlorinated Herbicides	SW 8151A			x	x	x	x		x		x	x	x			x	x	x	x		x	x		x	x	x	x	x	x		
Chlorinated Herbicides (exc. MCPP/MCPA)	SW 8151A			x	x	x	x	x			x					x	x		x			x		x	x	x	x	x	x		
Chlorinated Herbicides, Appendix IX List	SW 8151A			x	x	x	x	x	x		x					x	x	x	x		x	x		x	x	x	x		x		

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
GC / GC/MS Clean Up																															
Alumina Cleanup	SW 3610B	x					x	x	x	x				x		x			x					x							
Alumina Cleanup	SW 3611B									x		x		x			x							x	x		x				
Florisil Cleanup	SW 3620B				x	x	x	x	x	x	x	x		x		x	x	x	x	x	x	x		x	x	x	x			x	x
Silica Gel Cleanup	SW 3630C						x		x	x				x		x		x	x	x	x			x	x	x	x				x
GPC	SW 3640A			x	x	x	x	x	x		x	x		x		x		x	x	x	x	x		x	x	x	x				x
Acid-Base Partition Cleanup	SW 3650B (Mod.)			x		x	x		x			x		x		x		x	x	x	x	x		x	x		x				x
Sulfur Cleanup	SW 3660B			x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
Acid Cleanup	SW 3665A		x		x	x	x	x	x	x	x			x	x	x	x		x	x		x		x	x	x	x	x	x	x	
High Performance Liquid Chromatography - HPLC																															
Polynuclear Aromatic Hydrocarbons (PAHs)	EPA 610				x		x	x			x								x		x	x			x	x	x	x		x	
Polynuclear Aromatic Hydrocarbons (PAHs)	SW 8310				x		x	x			x								x		x	x		x	x	x	x	x		x	
Explosives	SW 8330				x		x	x			x			x										x		x	x				
Explosives	CHPPM				x																			x							
Carbamates	EPA 531.1					x																									
Carbamates	EPA 632					x	x									x											x				
INORGANIC FRACTIONS - Metals																															
PP Metals (13)	200.7, 245.1 / SW 6010B, 7470A/7471A			x	x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
TAL Metals (23)	200.7, 245.1 / SW 6010B, 7470A/7471A			x	x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
TAL Metals (23)	EPA SOW ILMO4.0				x	x	x	x	x			x		x			x		x		x	x			x	x	x		x		
RCRA Metals (7)	200.7, 245.1 / SW 6010B, 7470A/7471A			x	x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x		x	x	x	x	x	x	x	x

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
ICP Metals	EPA 200.7 / SW 6010B			x	x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
ICP Metals	EPA SOW ILMO4.0				x	x	x	x	x			x		x			x		x		x	x			x		x		x	x	
Furnace Metals	EPA 200 Series, 7000 Series			x	x	x		x	x	x	x		x			x	x	x		x	x	x		x	x	x	x	x	x	x	x
Furnace Metals	EPA SOW ILMO4.0				x	x		x	x								x	x			x	x			x	x	x		x		
Furnace Metals	EPA 200.9				x	x		x	x							x	x	x			x				x		x		x	x	
Furnace Metals	SM1311B										x																				
Mercury-Hg (cold vapor)	EPA 245.1, SW 7470A/7471A			x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
Mercury-Hg (cold vapor)	EPA SOW ILMO4.0				x	x	x	x	x			x		x			x		x		x	x			x	x	x		x		
Metals Preparation	SW 3050B			x	x	x	x	x	x	x	x	x		x		x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
Metals Preparation	SW 3005A, SW 3010A, SW3020A			x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
Metals Preparation	SW3051, CEM, SW3052			x										x																	
Metals Preparation	SM3030C, SM3030E										x													x							
TCLP	SW 1311			x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
SPLP	SW 1312			x	x	x	x	x	x	x	x			x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
CA W.E.T. Test	CA Title 22			x	x			x	x		x			x	x									x		x					
Metals, Appendix IX	SW 6010B, 7000 Series			x	x	x	x		x	x	x			x	x		x		x			x		x	x	x	x	x	x		
Metals, Appendix II	SW 6010B, 7000 Series			x	x	x				x	x			x	x		x		x			x									
MEP (Multiple Extraction Procedure)						x																x		x	x	x	x	x	x		
SPECIAL TEST CATEGORIES																															
Air Analysis																															
NIOSH Methods:																															
GC/MS Confirmation of GC Components	Mod. SW 5030B / 8260						x									x					x			x	x						x
GC/MS Screen (10 TICs) Non-quantitative	Mod. SW 5030B / 8260															x				x	x			x	x						x
Aldehyde Screen	NIOSH 2539																														x
GC/FID Screen (non-quantitative)																				x					x						x
BTEX	NIOSH 1501																								x						x

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
BTEX and TPH	Mod. SW 8015B / 8021B, EPA 18															x					x			x	x						
Naphthas	NIOSH 1550																														x
Pesticides (GC-ECD)	NIOSH 1550																														x
Chlordane	NIOSH 5278																														x
PCBs in Air, Oil or Wipe	NIOSH 5503																							x							x
Polynuclear Aromatic Hydrocarbons (PAH)	NIOSH 5515																														x
Phenols	NIOSH 3502																														x
Metals by ICP	NIOSH 7300																				x				x						x
Metals by Graphite Furnace	NIOSH 7300																				x				x						x
Mercury	NIOSH 6009																				x				x						x
Formaldehyde	NIOSH 3508																														x
Cyanide	NIOSH 7904																								x						x
Nuisance Dust	NIOSH 0500, PM10																							x							x
Ambient Air																															
Volatile Aromatic Hydrocarbons	TO-1 (6)								x						x						x				x						
Volatiles	TO-2								x						x	x									x						
BTEX & MTBE	TO-3														x																
Pesticide by GC	TO-4			x					x					x		x	x							x	x						
Polyaromatic Hydrocarbons by GC/MS	TO-13 (6)			x					x				x	x		x	x				x			x	x						
Polyaromatic Hydrocarbons by SIM	TO-13 (6)															x	x				x			x	x						
Canister Sample by GC/MS	Mod. TO-14 (6)			x			x						x	x	x	x					x				x						
Canister Sample by GC/MS - SIM	Mod. TO-14 (6)														x	x					x				x						
Tentative Identified Compounds (TICs + 10)	Mod. TO-14 (6)			x			x						x	x	x	x					x				x						
Canister Sample by GC/MS (Polar/Non-Polar)	TO-15						x																								
Rental - 6 liter Summa Canisters	-			x			x						x	x	x	x					x				x						
Purchase of TENAX tubes, tedlar bags	-						x		x				x	x	x	x					x				x						
Gases																															
Dissolved Gases	RSK-175						x								x										x						

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
Methane & Fixed Gases (CO, CO2, CH4, N2, O2)	STL Method / ASTM D1946			x			x								x						x				x						
Fixed Gases	ASTM D1946			x			x							x	x																
Specialty Organics by HRGC/HRMS																															
Dioxins/Furans	8280													x										x							
Dioxins/Furans	8290													x										x							
Dioxins/Furans	1613													x										x							
PCBs (Congeners)	1668 (Mod)													x										x							
Pesticides	STL Method													x										x							
Polynuclear Aromatic Hydrocarbons	CARB 429 (Mod)													x										x							
Specialty Organics by HPLC/MS																															
Triazines		x																						x							
Carbamates		x																						x							
Explosives		x																						x							
Chemical Warfare Degradates																								x							
Impurity identification under GLP		x																													
HPLC/MS method development		x																													
Source Testing																															
Volatile Organic Sampling Trains	0030/5041A/8260B													x																	
Semi-Volatiles	0010/3542/8270C													x										x							
Pesticides	0010/3542/8081A													x										x							
PCBs (Congeners)	0010/3542/1668 (Mod)													x										x							
Dioxins/Furans	0023A/8290													x										x							
Dioxins/Furans	Method 23													x										x							
Polynuclear Aromatic Hydrocarbons	0010/3542/CARB 429 (Mod)													x										x							
Total Chromatographical Organics/Gravimetric	0010/3542/EPA TCO/Grav													x										x							
Multi-metals train	0060/6010B/7471A													x										x							
Multi-metals train	Method 29/6010B/7471A													x										x							

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
Anion Impinger Solutions (Cl, F, Br)	0050/9056/9057													x										x							
Air train particulate Filters	PM-10													x										x							
Media preparation	VOST, XAD, filters													x										x							
Asbestos Analysis / Industrial Hygiene																															
Polarized Light Microscopy (PLM)	EPA 600/M4-82-020																				x										x
Phase Contrast Microscopy (PCM)	PCM NIOSH 7400																				x										x
Transmission Electron Microscopy (TEM)	EPA Phase II 600/4-83-042																														x
Non-Friable Organically Bound	PLM/TEM																														x
Chemical Degradation Agents																															
Thiodiglycol	STL Method				x																			x			x				
DIMP/DMMP	STL Method				x																			x							
IMPA/MPA	STL Method				x																			x							
Organosulfur Compounds	STL Method				x																			x							
BZ	STL Method				x																			x							
Nitroguanidine	STL Method													x										x							
Nitrocellulose	STL Method													x										x							
Perchlorates (in process)	STL Method													x										x							
Microscopy Services																															
Scanning Electron Microscopy (SEM)	SEM																														x
SEM with X-ray Spectroscopy	EDX, WEDX																														x
TEM with X-ray Spectroscopy	TEM-EDX																														x
Computer Assisted Microscopy:																															
PSA	TEM 200+, SEM, Light Microscopy																														x
Fourier Transfer Infra Red Spectroscopy	FTIR																														x
Hardness Testing																															x

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
ICP-MS	EPA 200.8, SW 6020					x					x													x		x					
Radiological Services																															
Water / Wastewater																															
Americium-Isotopic (Am-241)	Mod. HASL-300 (10)																					x			x						
Carbon-14 (C-14)	Mod. EPA C-01 (8)																					x									
Curium-244 (Cm0244)	HASL-300 Mod.																								x						
Carbon-14 (C-14)	Mod. EPA C-01 (7)																								x						
Curium-Isotopic (Cm-242, 244)	Mod. HASL-300 (10)																					x									
Gamma Spectroscopy	Mod. EPA 901.1 (7)																								x						
Gamma Spectroscopy	ASTM D3649-91																					x									
Gross Alpha / Gross Beta	Mod. EPA 900.0 (7)																								x						
Gross Alpha / Gross Beta	EPA 9310																					x									
Neptunium-237 (Np-237)	Mod. HASL-300 (10)																					x									
Plutonium-Isotopic (Pu-238, 239/240)	Mod. HASL-300 (10)																					x									
Radium-226	EPA 903.1 (7)																					x			x						
Radium-228	Mod. EPA 904.0																					x			x						
Radium-Total Alpha Emitters	EPA 903.0 (7)																								x						
Radium-Total Alpha	Mod. ASTM D2460																					x									
Strontium-90	Mod. DOE RP500 (9)																								x						
Strontium-90	Mod. HASL-300 (10)																					x									
Technetium-99 (Tc-99)	Mod. DOE RP550 (9)																								x						
Technetium-99 (Tc-99)	Mod. HASL-300 (10)																					x									
Thorium-Isotopic (Th-228, 230, 232)	Mod. HASL-300 (10)																					x									
Tritium	Mod. EPA 906.0 (7)																					x			x						
Isotopic Plutonium (Pu-238, 239. 240)	HASL-300 (10)																								x						
Isotopic Thorium (Th-228, 230, 232)	HASL-300 (10)																								x						
Uranium-Isotpoic (U-234, 235, 238)	Mod. HASL-300 (10)																					x			x						
Uranium-Total (U)	ASTM D5174-91																					x									

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
Solids																															
Americium-Isotopic (Am-241)	Mod. HASL-300 (10)																						x			x					
Carbon-14 (C-14)	Mod. EPA C-01 (8)																						x								
Curium-Isotopic (Cm-242, 244)	Mod. HASL-300 (10)																						x								
Gamma Spectroscopy	HASL-300 (10)																									x					
Gamma Spectroscopy	ASTM D3649-91																						x								
Gross Alpha / Gross Beta	DOE RP 720																									x					
Gross Alpha / Gross Beta	EPA 9310 / SM16																						x								
Neptunium-237 (Np-237)	Mod. HASL-300 (10)																						x								
Plutonium-Isotopic (Pu-238, 239/240)	Mod. HASL-300 (10)																						x								
Radium-226	HASL-300																									x					
Radium-226	ASTM D3649-91																						x								
Radium-228	Mod. EPA 904.0																									x					
Radium-228	ASTM D3649-91																						x								
Radium-Total Alpha	Mod. ASTM D2460																						x								
Strontium-90	Mod. DOE RP500 (9)																														
Strontium-90	Mod. HASL-300 (10)																						x								
Technetium-99 (Tc-99)	Mod. DOE RP550 (9)																									x					
Technetium-99 (Tc-99)	Mod. HASL-300 (10)																						x								
Thorium-Isotopic (Th-228, 230, 232)	HASL-300 (10)																						x								
Tritium	Mod. PNL-ALO-441																														
Tritium	Mod. EMSL-LV-0539-17																						x								
Isotopic Plutonium (Pu-238, 239. 240)	HASL-300 (10)																									x					
Isotopic Thorium (Th-228, 230, 232)	HASL-300 (10)																									x					
Uranium-Isotopic (U-234, 236, 238)	HASL-300 (10)																						x			x					
Uranium-Total (U)	ASTM D5174-91																						x								
Drinking Water																															
Gamma Spectroscopy	EPA 901.1																						x								

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
Gross Alpha / Gross Beta	EPA 900.0 (7)																						x			x					
Radium-226	EPA 903.1																						x			x					
Radium-228	EPA 904.0																						x			x					
Radium-Gross Alpha	Mod. ASTM D2460																						x								
Radon-222 (Rn-222)	EPA 913.0																						x								
Strontium-90	EPA 905.0 (7)																									x					
Strontium-90	Mod. HASL-300 (10)																						x								
Tritium	Mod. EPA 906.0 (7)																						x			x					
Soil Tests																															
Grain Size	ASTM D442						x																								
Soil Compaction	ASTM D698						x																								
Specific Gravity	ASTM D854						x									x															
Specific Gravity	ASTM D 1298							x								x															
Moisture Content	ASTM D2216			x	x		x			x						x															
Moisture Content	SM 2540G							x		x																					
Soil Permeability	ASTM D2434						x																								
Soil Classification	ASTM D2487/2488						x																								
Atterburg Limits	ASTM D4318						x																								
Hydraulic Conductivity	ASTM D5084						x																								
Toxicology																															
Freshwater Whole Effluent Toxicity	ceriodaphnia & promelas pimephales																														x
Saltwater Whole Effluent Toxicity	Sheepshead minnow & mysids																														x
INORGANIC FRACTIONS																															
General Chemistry																															

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
Acidity	EPA 305.1				x	x		x	x	x	x	x	x			x	x				x				x	x	x		x	x	
Acidity	EPA 305.2				x		x				x					x	x							x	x		x		x		x
Acidity	SM18 2310B				x			x				x				x	x				x				x		x		x		
Alkalinity, Hydroxide	EPA 310.1 / SM 2320B			x	x		x	x		x	x				x	x	x		x					x	x		x		x	x	
Alkalinity, Total	EPA 310.1			x	x	x	x	x	x	x	x	x	x		x	x	x		x		x	x		x	x	x	x	x	x	x	
Alkalinity, Total	EPA 310.2					x																									
Alkalinity, Total	SM18 B 2320B			x	x		x	x		x	x	x			x	x	x	x	x		x			x	x		x		x	x	x
Alkalinity, Bicarbonate	EPA 310.1			x	x	x	x	x	x	x	x				x	x	x		x		x	x		x	x	x	x	x	x	x	
Alkalinity, Bicarbonate	SM 406C				x				x		x					x	x								x		x		x		
Alkalinity, Bicarbonate	SM18 B 2320B				x			x		x	x	x			x	x	x	x	x					x	x		x		x	x	
Alkalinity, Carbonate	EPA 310.1			x	x	x	x	x	x	x	x				x	x	x		x		x	x		x	x	x	x	x	x	x	
Alkalinity, Carbonate	SM18 B 2320B			x	x			x		x	x	x			x	x	x	x	x					x	x		x		x	x	
Anion Exchange Capacity	SW 9080								x																x		x				
Anions, by IC (Br, PO_4, SO_4, NO_3, Cl, F)	EPA 300.1 / SW 9056				x	x					x			x		x			x						x		x			x	
Anions, by IC (Br, PO_4, SO_4, NO_3, NO_2,Cl, F)	EPA 300.0 / SW9056			x	x	x	x	x		x	x			x		x		x	x					x	x	x	x	x		x	
Anions, by IC (Br, PO_4, SO_4, NO_3, NO_2, Cl)	SM 4110B				x	x		x							x										x		x				
AOX	1631																							x	x						
Ash	ASTM D-482					x		x		x							x								x	x	x		x	x	
Ash	SM 2540G							x		x						x	x								x		x		x	x	
Ash	ASTM D-3174							x								x	x								x		x		x		
ASTM Leaching Procedure	ASTM D3987				x	x			x			x					x		x			x			x		x		x	x	
BOD5	EPA 405.1				x	x	x	x	x	x	x	x				x	x	x	x		x	x		x	x	x	x	x	x	x	x
BOD5	SM18 5210B				x		x	x		x	x	x			x	x	x	x	x		x	x		x	x		x		x	x	x
CBOD5	SM18 5210B				x	x	x	x		x	x					x	x	x	x		x	x		x	x		x	x	x	x	x
Bomb, Prep for Hazardous Waste	SW 5050						x	x								x									x					x	
Bottom Sediment & Water	ASTM D-4007							x																							
Bromide	SW 320.1					x			x							x	x														
Bromide	Method 300.0					x																		x							
BTU	ASTMD 240-87					x	x									x		x							x	x					
Cation Exchange Capacity	SW 9080			x					x	x															x						

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
Cation Exchange Capacity	SW 9081			x	x		x	x				x							x					x	x						
Chloride	EPA 300			x		x				x	x			x	x	x			x					x	x			x		x	
Chloride	EPA 325.2				x	x	x	x	x	x	x						x		x			x		x	x		x	x		x	
Chloride	EPA 325.3				x						x		x						x		x			x	x	x			x		x
Chloride	SW 9250						x				x					x														x	
Chloride	SW 9251				x	x		x			x						x		x			x			x		x	x		x	
Chloride	SW 9252A												x						x		x				x				x		x
Chloride	SW 9253									x		x				x															
Chloride	SM18 4500-CL B			x								x				x		x												x	
Chloride	SM18 4500-Cl C																				x				x				x	x	x
Chloride	SM18 4500-Cl E				x		x	x									x								x		x			x	
Chloride (Emissions)	SW 9057/Method 26a			x																											
Chlorine (O2 Bomb)	SW 5050 (followed by Cl Analysis)			x			x	x								x								x	x					
Chlorine Demand	SM 2350B					x			x			x																		x	
Chlorine Residual	EPA 330.4				x	x		x	x	x		x	x			x								x						x	
Chlorine Residual	EPA 330.5											x				x			x		x									x	
Chlorine Residual	SM18 4500Cl F				x			x				x																			
Chlorine Residual	SM18 4500Cl G						x											x			x										
COD	EPA 410.1						x									x	x								x	x	x	x	x		
COD	EPA 410.4			x	x	x			x		x	x	x			x		x	x		x	x		x		x		x		x	x
COD	SM18 5220B						x																								
COD	SM185520C																											x			
COD	SM18 5220D			x			x					x			x				x		x			x							x
COD	Hach 8000					x		x		x								x						x						x	x
Coliform, Fecal	SM18 9222-D									x						x	x	x			x				x		x		x	x	x
Coliform, Fecal	(DW) SM18 9223 UV+(ONPG-MUG)															x					x						x				
Coliform, Strep	SM18 9230-C-a															x															x
Coliform, Total	SM18 9222-B									x						x	x	x			x				x		x		x	x	x
Coliform, Total	(DW) SM18 9223 (ONPG-MUG)															x		x			x						x			x	
Color	EPA 110.1						x				x					x															

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
Color	EPA 110.2				x	x		x	x	x	x	x	x			x	x				x				x	x	x	x	x		
Color (Spectrophotometric)	EPA 110.3												x																		
Color	SM18 2120B				x			x			x	x					x	x			x				x		x		x	x	x
Corrosivity, as pH	SW 9040B, SW 9045C			x	x	x	x	x	x	x	x	x		x	x		x	x	x		x	x		x	x	x	x	x	x	x	x
Corrosivity (NACE)	SW 1110									x							x		x		x										
Conductance, Specific	EPA 120.1/SW 9050A/SM18 2510B			x	x	x	x	x	x	x	x	x	x		x	x	x	x	x		x	x		x	x	x	x	x	x	x	x
Corrosivity (Langlier Index)	SM 2330A+B							x								x	x	x							x		x		x	x	
Chromium, Trivalent	Total Cr - Cr+6			x	x			x	x	x	x						x								x		x		x	x	
Chromium (Hexavalent)	SW 7196A			x	x	x	x	x	x	x	x	x	x		x		x		x		x	x		x	x	x	x	x	x	x	x
Chromium (Hexavalent)	SM 3500D				x		x	x			x					x	x	x	x			x		x	x		x		x	x	x
Chromium (Hexavalent)	USGS I1230-85-01032											x																				
Chromium (Hexavalent, in Soil)	SW 3060A / 7196A (NJ Mod)						x			x		x				x															
Chromium (Hexavalent, in Soil)	1005				x			x								x									x						
Chromium (Hexavalent)	SW 7195					x																									
Chromium (Hexavalent, Emissions)	SW 0061/SW 7199			x																											
CTAS	SM5540																x														
Cyanide, Amenable to Chlorination	EPA 335.1			x	x	x	x	x	x	x	x		x	x		x	x		x		x			x	x	x					
Cyanide, Amenable to Chlorination	SM18 4500-CN G						x	x		x	x						x	x			x				x					x	
Cyanide, Amenable to Chlorination	SW 9010B / 9014					x		x			x										x				x						
Cyanide, Amenable to Chlorination	SW 9012A			x	x				x		x	x	x	x			x		x			x		x	x	x				x	
Cyanide, Amenable to Chlorination	Lachat 204001A															x	x							x						x	x
Cyanide, Extractable	SW 9013									x		x									x			x	x						
Cyanide, Total (Colorimetric)	EPA 335.2				x	x		x		x	x	x	x		x	x	x	x	x					x	x	x		x			x
Cyanide, Total	ILM04.0				x	x	x	x	x			x		x				x	x		x	x			x	x					
Cyanide, Total (Automated)	EPA 335.3			x	x				x		x	x				x			x		x	x			x						
Cyanide, Total	EPA 335.4				x		x		x		x	x		x		x	x		x		x				x						
Cyanide, Total (prep only)	SW 9010B							x		x	x				x	x	x				x			x	x			x		x	x
Cyanide, Total	SW 9012A			x	x		x		x		x	x	x	x	x	x	x		x			x		x	x	x					x
Cyanide, Total	SW 9010B / SW 9014					x		x		x	x					x		x			x			x	x			x			x
Cyanide, Total	SM4500-CN.A-E				x		x				x	x				x	x	x	x						x					x	x

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
Cyanide, Total	SM 4500 CNC E							x			x					x	x		x						x	x				x	
Cyanide, Total	SM 4500 CN G										x																				
Cyanide, Free	EPA 335.2												x																		
Cyanide, Free	SM 18 4500-CN I					x			x	x	x							x	x		x	x		x	x			x		x	
Cyanide, Free	Lachat 204001A																														x
Cyanide, Free	SM4500-CN.A-E										x					x									x					x	x
Cyanide, Physiologically Available CN	MA DEP								x																						x
Cyanide, Weak & Dissociable	SM 4500CN I				x			x		x	x					x		x				x			x					x	
Density	SM18 2710F						x	x									x								x		x		x		
Density	ASTM D1298							x		x						x	x								x		x		x		
Density	ASTM 5057							x																						x	
Dissolved Inorganic Carbon	EPA 415.1 M				x		x	x			x						x								x		x	x			
Dissolved Organic Carbon	EPA 415.1				x		x	x			x				x	x	x								x		x	x		x	
Dissolved Oxygen	EPA 360.1				x	x							x											x		x		x			
Extractable Organic Halides (EOX)	SW 9020B									x	x					x		x							x						
Extractable Organic Halides (EOX)	SW 9023					x				x	x	x													x	x					
Ferrous Iron	SM18 3500B					x			x									x													x
Ferrous Iron	SM19 3500FE-D				x	x	x	x			x	x				x	x	x	x						x		x		x		
Flashpoint	SW 1010				x	x	x	x	x	x	x				x	x		x	x		x	x			x	x		x		x	x
Flashpoint	SW 1020A		x	x								x				x								x		x					
Flashpoint	SW 1030								x			x					x								x		x		x		
Flashpoint	ASTM D-92							x		x						x														x	
Flashpoint	ASTM D-93							x			x					x			x												
Fluoride (probe)	EPA 340.2			x	x	x	x	x	x	x	x	x		x		x	x	x		x	x		x	x		x	x	x	x	
Fluoride (probe)	SM18 4500-F C				x		x	x		xx	x	x				x	x		x		x				x	x	x		x		x
Fluoride (distillation probe)	EPA 340.2 / SM18 4500-F C				x			x								x					x					x					
Fluoride (distillation probe)	SW 9214											x									x										
Fl Fluoride (IC)	EPA 300			x		x					x			x		x			x					x	x	x	x	x		x	
Fluorine	SW 5050 (followed by F analysis)							x																	x						
Formaldehyde	NYSDOH APC-44								x																						

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

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Formaldehyde	NIOSH 3500Mod.											x				x												x			
Free Carbon Dioxide	SM 4500 CO2D							x			x						x								x		x		x		
Glycol	NYSDOH APC-44								x																						
Hardness (EDTA Total as CaCO3)	EPA 130.2			x	x	x	x	x		x	x	x	x			x	x	x	x			x		x	x	x	x	x	x		
Hardness (EDTA Total as CaCO3)	SM18 2340B						x	x	x		x	x		x			x		x			x		x	x		x		x		x
Hardness (EDTA Total as CaCO3)	SM19 2340C			x	x		x	x				x					x							x	x		x	x	x		
Hardness (calculation)	EPA 200.7 / SW 6010B			x		x	x	x	x		x	x		x	x	x	x	x	x		x	x		x	x		x	x	x		
Heavy Metals	USP, 22 Ed 231, Md. II							x																							
Hydrazine	ASTMD1385																											x			
Ignitability	SW 1010				x	x	x	x	x	x	x					x		x	x		x			x	x	x				x	x
Ignitability	SW 1020A		x	x								x				x										x					
Ignitability	SW 1030								x								x								x		x		x		
Ignitability	ASTM D-4982A							x																							
Ignitability	SW Sec. 7				x			x			x					x			x			x			x			x			
MBAS, Surfactants	EPA 425.1				x	x	x	x	x	x			x			x	x	x			x			x	x	x	x	x	x		
MBAS, Surfactants	SM18 5540C				x		x	x							x	x	x	x			x				x		x		x		
Moisture, Percent (%)	EPA 160.3			x	x	x	x		x	x	x	x	x		x	x	x	x	x		x	x		x	x	x	x	x	x	x	x
Moisture, Percent (%)	SM 2540G M							x		x	x						x		x						x		x		x	x	
Ammonia, Nitrogen	EPA 350.1			x	x	x					x	x				x	x		x		x	x			x	x	x			x	
Ammonia, Nitrogen / Distillation	EPA 350.1			x							x								x			x			x					x	
Ammonia, Nitrogen / Distillation	EPA 350.2						x	x				x				x					x							x		x	
Ammonia, Nitrogen / Distillation	EPA 350.3								x	x			x		x	x			x					x				x			
Ammonia, Nitrogen / Distillation	SM 4500 NH3B+C							x																						x	
Ammonia, Nitrogen / Distillation	SM18 4500-NH_3 E															x		x			x									x	x
Ammonia, Nitrogen / Distillation	SM18 4500-NH_3 H				x							x			x	x														x	
Ammonia, Unionized	FL DEP SOP																											x			
Nitrate-Nitrogen	EPA 300			x		x					x			x	x	x			x					x	x		x	x		x	
Nitrate-Nitrogen	353.2, SM18 4500NO_3F			x	x	x	x	x		x	x	x	x				x				x	x			x	x	x		x	x	
Nitrate-Nitrogen	Lachat 107041A																x	x						x	x		x		x		x
Nitrite-Nitrogen	EPA 300			x		x					x			x	x	x			x					x	x	x	x	x		x	

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
Nitrite-Nitrogen	EPA 353.2			x	x	x	x	x	x	x	x	x	x				x		x		x	x		x	x	x	x	x	x	x	
Nitrite-Nitrogen	EPA 354.1						x	x			x		x						x		x								x		
Nitrite-Nitrogen	SM18 4500NO_2B				x		x	x			x										x								x		x
Nitrate/Nitrite	EPA 300			x		x					x				x	x			x					x	x			x		x	
Nitrate/Nitrite	EPA 353.2			x	x	x	x	x	x		x	x	x				x		x		x	x		x	x	x	x	x	x	x	
Nitrate/Nitrite	SM18 4500NO_3F						x	x			x	x					x	x			x				x		x		x	x	
Nitrate/Nitrite	Lachat 107041A																x							x	x		x		x		x
Nitrogen, Total Kjeldahl (TKN)	EPA 351.2			x	x	x	x		x	x	x	x	x			x	x	x			x			x	x		x			x	
Nitrogen, Total Kjeldahl (TKN)	EPA 351.3 / SM18 4500-NH_3E						x			x						x			x		x									x	
Nitrogen, Total Kjeldahl (TKN)	EPA 351.3 / SM18 4500-NH_3E															x			x		x							x		x	
Nitrogen, Total Kjeldahl (TKN)	EPA 351.3 / SM18 4500NorgC							x								x														x	
Nitrogen, Total Kjeldahl (TKN)	Lachat 107062D																x								x		x				x
Nitrogen, Total Organic (TON)	EPA 351.2				x				x		x	x					x								x		x			x	
Nitrogen, Total Organic (TON)	EPA 350.1, EPA 351.2					x					x	x				x	x								x		x			x	
Nitrogen, Total Organic (TON)	EPA 350.1, EPA 351.3						x				x					x		x			x										
Nitrogen, Total Organic (TON)	EPA 351.3 / EPA 350.2							x			x					x			x							x		x			
Nitrogen, Total Organic (TON)	SM 4500NorgC / 4500NH3B+C							x			x																				
Nitrogen, Total	EPA 351.3/353.2																							x				x			
Odor	EPA 140.1					x		x	x	x						x	x				x				x		x		x		
Odor	SM 2150B							x									x								x		x		x		x
Oil & Grease (IR)	EPA 413.2			x	x		x	x	x			x			x	x	x		x		x	x			x		x		x	x	
Oil & Grease (Gravimetric)	EPA 413.1				x	x	x	x	x	x		x	x		x	x		x	x		x	x				x				x	
Oil & Grease (Gravimetric)	SW 9070				x	x	x	x				x	x			x			x		x					x				x	
Oil & Grease (Gravimetric)	SW 9071A				x	x	x	x				x	x			x			x		x									x	
Oil & Grease (Gravimetric)	SM18 5520B				x			x											x		x									x	
Oil & Grease (IR-Waters)	SW 5520C							x									x		x						x		x		x	x	
Oil & Grease (Gravimetric-Soxhlet)	SW 5520D							x																						x	
Oil & Grease	1664			x	x	x			x		x		x	x		x	x		x					x	x		x		x	x	
Oxidation / Redox Potential	SM18 2580				x							x													x						
Oxidation / Redox Potential	ASTM D1498				x		x	x	x	x															x						x

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
Oxygen, Dissolved	EPA 360.1				x	x		x			x	x				x	x				x	x		x	x		x	x	x		
Oxygen, Dissolved	EPA 360.2				x		x	x								x	x		x					x	x		x		x		
Oxygen, Dissolved	SM18 4500-O C				x		x	x	x		x					x	x	x			x				x		x		x	x	x
Oxygen, Dissolved	SM18 4500-O G				x			x				x				x	x								x		x		x		
Paint Filter Test	SW 9095A			x	x	x	x	x	x	x	x	x				x	x	x			x	x		x	x	x	x	x	x	x	x
Petroleum Hydrocarbons-IR (TPHC)	EPA 418.1			x	x	x	x	x	x	x		x	x			x	x	x	x	x	x	x			x		x		x	x	
Petroleum Hydrocarbons-IR (TPHC)	SW 9073					x										x	x				x				x		x		x		
Petroleum Hydrocarbons (TPHC)	NYSDOH 310.13					x												x													
Petroleum Hydrocarbons (Gravimetric)	SM18 5520 F				x				x										x											x	
PH	EPA 150.1 / SW 9040B / 9045C/ 4500H+B			X	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x		x	x		x	x	x	x	x	x	x	x
pH	SW 9041A							x			x	x				x	x		x						x		x		x		
Phenols, Total	EPA 420.1				x		x			x	x					x	x	x	x		x				x					x	x
Phenols, Total	EPA 420.2			x		x		x	x		x	x	x			x						x		x		x		x			
Phenols, Total	SW 9065				x		x			x	x					x	x		x		x			x	x						x
Phenols, Total	SW 9066			x		x		x	x		x	x	x			x						x				x		x		x	
Phenols, Total	SM18 5530C						x				x													x							x
Phosphate (Ortho)	EPA 365.2			x		x	x	x	x	x	x	x				x	x		x		x				x		x		x	x	
Phosphate (Ortho)	EPA 365.3				x						x		x			x		x	x					x						x	
Phosphate [Ortho]	SW 9056										x						x								x		x	x			
Phosphate (Ortho)	EPA 365.4																x								x		x				
Phosphate (Ortho)	EPA 300										x			x	x	x			x							x		x		x		
Phosphate (Ortho)	SM18 4500-P E						x	x	x		x	x					x				x				x		x		x	x	x
Phosphorus (Total)	EPA 365.1						x									x					x	x				x		x			
Phosphorus (Total)	EPA 365.2			x		x		x	x			x				x								x						x	
Phosphorus (Total)	EPA 365.3				x						x		x					x	x												
Phosphorus (Total)	SM18 4500-P BE						x					x									x			x						x	x
Phosphorus (Total)	SM18 4500-P E							x																						x	
Purgeable Organo Halides (POX)	9021										x																				
Reactivity (Cyanide/Sulfide)	SW846,Chapter7			x	x	x	x	x	x	x	x	x	x			x	x		x		x				x	x				x	x

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
Salinity	SM18 2520B						x	x	x																x						x
Salinity	SM17 201C																				x				x						
Silica	EPA 370.1				x	x			x	x						x										x					
Silica	SW 6010B (Mod)						x	x			x			x		x						x		x	x						
Silica	SM 4500-Si-D								x							x															
Sludge Volume Index	SM 2710 D								x																					x	
Solids, Percent	SM 2540G							x		x	x						x								x		x		x		x
Solids, Percent	EPA 160.3 MOD																							x				x			
Solids, Settleable	EPA 160.5				x	x	x	x	x	x	x	x	x			x	x	x	x		x	x		x	x	x	x		x		
Solids, Settleable	SM18 2540F				x		x	x	x	x	x	x					x	x			x				x		x		x		x
Solids, Total	EPA 160.3			x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x		x	x		x	x	x	x	x	x	x	
Solids, Total	SM18 2540 B			x	x		x	x		x	x	x					x	x			x				x		x		x	x	x
Solids, Total Fixed & Volatile	SM19 2540G					x	x			x	x	x					x	x							x		x		x	x	
Solids, Total Fixed & Volatile	SM18 2540G							x		x	x						x	x							x		x		x	x	x
Solids, Total Dissolved	EPA 160.1			x	x	x	x	x	x	x	x	x	x		x	x	x	x	x		x	x		x	x	x	x	x	x	x	x
Solids, Total Dissolved	SM18 2540 C				x		x	x		x	x	x					x	x			x				x		x		x	x	x
Solids, Total Suspended	EPA 160.2			x	x	x	x	x	x	x	x	x	x		x	x	x	x	x		x	x		x	x	x	x	x	x	x	x
Solids, Total Suspended	SM18 2540 D				x		x	x		x	x	x					x	x			x				x		x		x	x	x
Solids, Total Volatile	EPA 160.4 SM 18 2540 E				x	x	x	x	x	x	x	x					x	x	x		x	x			x		x		x	x	x
Solids, Volatile Suspended	EPA 160.4 SM 18 2540 E				x		x	x	x	x	x	x	x				x	x			x				x		x		x	x	x
Specific Gravity	SM18 2710 F			x		x	x	x			x						x		x						x		x	x		x	
Specific Gravity	ASTM D-1298							x		x	x					x	x							x	x		x				
Sulfate, as SO_4	EPA 300			x		x				x	x			x	x	x			x					x	x	x	x	x			
Sulfate, as SO_4	EPA 375.2								x							x														x	
Sulfate, as SO_4	EPA 375.4			x	x	x	x	x		x	x	x	x				x	x			x	x		x	x	x	x	x		x	x
Sulfate, as SO_4	SW 9038			x	x	x	x	x		x	x	x	x				x				x				x		x	x		x	
Sulfate, as SO_4	SM 4500-$SO_4$2.A-E						x											x												x	x
Sulfate, as SO_4	SM 4500-$SO_4$2 E							x									x								x		x			x	
Sulfide, as S	EPA 376.1			x	x	x	x	x	x			x	x			x	x		x		x	x		x	x	x		x		x	x
Sulfide, as S	EPA 376.2			x			x			x	x					x	x								x					x	

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
Sulfide, as S	SM18 4500-S2.A-E						x										x				x				x					x	x
Sulfide, as S	SM18 4500-S2 E				x			x									x								x					x	
Sulfide, as S (Distillation)	SW 9030B						x		x		x	x	x				x				x	x		x	x	x				x	
Sulfide, Acid Soluble	SW 9030B/9034			x		x	x	x		x	x	x					x				x				x						
Sulfide, Acid Insoluble	SW 9030B/9034					x	x			x											x										
Sulfite, as SO_3	EPA 377.1 SM18 4500-SO_3 B				x	x	x			x	x	x	x				x	x			x				x		x		x	x	
Sulfur	SW 5050 (Followed by SO4 Analysis)							x								x									x						
Total Carbon	EPA 415.1 M							x			x					x	x								x	x	x	x		x	
Total Inorganic Carbon	EPA 415.1 M / SM 5310C M				x			x			x					x	x		x						x	x	x	x		x	
Total Organic Carbon (TOC)	EPA 415.1			x	x	x	x	x			x	x	x		x	x	x		x					x	x		x	x		x	
Total Organic Carbon (TOC)	EPA 415.2								x							x					x					x						
Total Organic Carbon (TOC)	SM18 5310 B											x					x				x				x		x					
Total Organic Carbon (TOC)	SM18 5310 C							x																								
Total Organic Carbon (TOC)	SW 9060			x	x	x	x	x			x	x	x		x	x	x		x					x	x	x	x	x			
Total Organic Carbon (TOC)	Lloyd Kahn (Region II)			x	x		x		x			x																				
Total Organic Carbon (TOC)	Walkley Black				x			x		x			x						x		x	x		x	x							
Total Organic Halides (TOX)	EPA 450.1					x	x										x		x					x	x	x						
Total Organic Halides (TOX)	SW 9020B					x	x	x		x	x	x	x				x		x		x			x	x	x						
Total Organic Halides (TOX)	SW 5050/9253																				x											
Total Organic Halides (TOX)	SM18 5320B							x				x					x								x							
Total Organic Halides (TOX) (Waste)	Cl2 - Cl							x									x								x							
Total Organic Halides (TOX) (AOX)	1650										x						x							x	x							
Turbidity	EPA 180.1 SM18 2130 B				x	x	x	x	x	x	x	x	x		x	x	x	x	x		x			x	x	x	x	x	x	x	x
Unionized Hydrogen Sulfide	SM4500 S-F																					x										
Viscosity, Brookfield	ASTM D-2196					x		x																								
Viscosity, Kinematic	ASTM D-445							x																								
Volatiles, Percent	SM 2540G							x		x	x																					
Water, Percent (Dean Stark)	ASTM D95							x																								
Water, Percent (Karl Fischer)	ASTM D4377							x		x																						

Analytical Fraction	Methods	STL AASG (Denver)	STL Anchorage	STL Austin	STL Baltimore	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL On-Site Technologies	STL Pensacola	STL Pittsburgh	STL Richland	STL Sacramento	STL Savannah	STL St. Louis	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield & STL Billerica+
OTHER SERVICES																															
Field Services						x	x					x		x		x	x	x	x	x	x				x		x	x	x	x	x
Diskette Deliverables		x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
Mobile Laboratory						x								x	x					x											x
Quality Assurance Plan Writing/Review				x	x		x	x			x			x	x		x		x	x		x			x		x	x	x	x	
Report Deliverables														x																	
Level 1 - Data Summary		x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
Level 2 - w/ QC		x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
Level 3 - Reduced Format		x	x	x	x	x	x	x	x	x	x	x	x	x	x	x		x		x	x	x		x	x	x	x	x		x	x
Level 4 - Full/CLP				x	x	x	x	x	x		x	x	x	x	x			x	x	x	x	x		x	x	x	x	x			x

+ All analyses conducted at STL Westfield with the exception of Microscopy Services
* Only as Methanol Preserved
****Exception to Forms.
(1) Lab must be notified in advance
(2) Method for determination of Volatile Petroleum Hydrocarbons, MADEP-VPH-98-1, Revision 0, January 1998
(3) Method for Determination of Petroleum Range Organics, FL DEP.
(4) Method for the Determination of Extractable Petroleum Hydrocarbons, By GC/FID, Tennessee DEC.
(5) Method for the Determination of Extractable Petroleum Hydrocarbons, MADEP-EPH-98-1, Revision 0, January 1998.
(6) Methods from EPA / 625 / R-96/101b, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air
(7) Methods reference EPA-600/4/80-032, Prescribed Procedures for Measurement for Radioactivity in Drinking Water, Aug. 1990.
(8) Methods reference EPA-600/4/75-001, Prescribed Procedures for Measurement for Radioactivity in Drinking Water, Aug. 1990.
(9) Methods Compendium
(10) Methods from Environmental Measurements Laboratory (EML) Procedures Manual, 27th Edition, November 1990.
SM16 = Standard Methods for the Examination of Water and Wastewater, 16th Edition
SM17 = Standard Methods for the Examination of Water and Wastewater, 17th Edition
SM18 = Standard Methods for the Examination of Water and Wastewater, 18th Edition

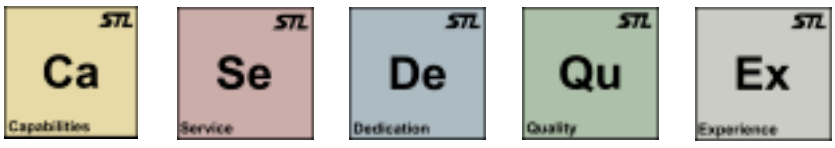
SM19 = Standard Methods for the Examination of Water and Wastewater, 19th Edition
SW = USEPA Test Methods for Evaluating Solid Wastes, SW-846, 3rd Edition
ASTM = American Society for Testing & Materials

SEVERN TRENT SERVICES

SEVERN TRENT LABORATORIES

APPENDIX B

Certifications



Certifying State/Program	Program Type	STL Anchorage	STL Austin	STL Baltimore	STL Billerica	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL Pensacola	STL Pittsburgh	STL Richland	STL St. Louis	STL Sacramento	STL Savannah	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield
Alabama	DW			X				X			X					X	X		X	X	x					X	X			
	MICRO																X													
Alaska	**DW**																													
	UST	**X**		**IP**							**X**				**X**									**x**						
Arizona	DW-Asbestos				X																									
	DW					X		X			X				X									X						
	WW					X		X			X				X					X				X						
	HW					X		X			X				X					X				X						
	Air														X									X						
	Radiological																													
Arkansas	**DW**					**X**		**X**			**X**																			
	WW		**X**			**X**		**X**			**X**			**X**		**X**				**X**			**IP**	**X**	**X**					
	HW		**X**			**X**		**X**			**X**			**X**		**X**				**X**			**IP**	**X**	**X**					
	MICRO																													
	Soil		**X**			**X**		**X**			**X**			**X**		**X**				**X**			**IP**	**X**	**X**					
California	DW						X	X						X	X									X						
	WW		X	X		X	X	X			X		X	X	X				X	X			X	X						
	HW		X	X		X	X	X			X		X	X	X				X	X			X	X						
	Radiological																					X	X							
Colorado	**DW-Asbestos**				**X**																									
	DW			**X**				**X**		**IP**	**X**				**X**						**X**	**X**								
Connecticut	General			X		X	X	X	X		X	X		X		X		X	X	X	X		X	X			X			X

Certifying State/Program	Program Type	STL Anchorage	STL Austin	STL Baltimore	STL Billerica	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL Pensacola	STL Pittsburgh	STL Richland	STL St. Louis	STL Sacramento	STL Savannah	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield
	DW					X		X			X	X		X					X		X			X	X					
	DW-Asbestos				X																									
	Asbestos																													
	Radiological																						X							
Delaware	**DW**			**X**				**X**												**X**					**X**					
	Superfund DNREC			**X**								**X**																		
Florida	DW						X	X						X		X	X			X				X	X	X	X	X		
	WW			X			X	X						X		X	X		X	X	IP			x	X	X	X	X		
	HW			X			X	X			X			X		X	X		X	X	IP			x	X	X	X	X		
	Air														X															
	DW-Asbestos				X																									
Georgia	**DW**			**X**				**X**						**X**			**X**								**X**	**X**	**X**			
	MICRO																**X**								**X**	**X**				
Hawaii	DW			X				X						X										x						
	MICRO																													
Idaho	**DW-Asbestos**				**X**																									
	DW			**X**				**X**			**X**											**X**									
Illinois	DW							X															X							
	WW							X																					IP	
	HW							X																					IP	
	MICRO																													
Indiana	**DW**			**X**				**X**						**X**											**X**	**X**			**X**	
	MICRO																												**X**	

Certifying State/Program	Program Type	STL Anchorage	STL Austin	STL Baltimore	STL Billerica	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL Pensacola	STL Pittsburgh	STL Richland	STL St. Louis	STL Sacramento	STL Savannah	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield
Iowa	DW							X																	X					
	WW							X																	X					
	UST			X																										
Kansas	**DW**							**X**								**X**							**X**							
	WW		**x**	**X**		**X**		**X**			**X**					**X**				**X**			**X**							
	HW		**X**	**X**		**X**		**X**			**X**					**X**				**X**			**X**			**X**				
	Radiological																													
Kentucky	DW			X		X		X					X	X		X			X	X	X				X		X			
	DW-Asbestos				X																									
	UST			X		x		X					X												X					
Louisiana	**DW**			**X***			**X***	**X***								**X***				**X***							**X***	**IP**			
	GW/WW/HW		**X***			**X***	**X***	**X***		**IP**	**X***			**X***	**X***		**X***			**X**				**X***	**X***	**X***					
Maine	DW			X			X		X																		X			
	DW-Asbestos				X																									
	Asbestos																													
Maryland	**DW**			**X**		**X**		**X**			**X**			**X**	**X**	**X**			**X**	**X**	**X**			**X**	**X**		**X**				
	DW-Asbestos				**X**																									
	Radiological																													
Massachusetts	DW			X		X	X	X	X					X		X		X	X		X				X	X	X			X
	WW			X	X	X	X	X	X					x				X	X	X	X				X	X	X			X
	DW-Asbestos				X																									
	Asbestos																													
Michigan	**DW**			**X**				**X**												**X**					**X**					

Certifying State/Program	Program Type	STL Anchorage	STL Austin	STL Baltimore	STL Billerica	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL Pensacola	STL Pittsburgh	STL Richland	STL St. Louis	STL Sacramento	STL Savannah	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield
	DW-Asbestos				X																									
Minnesota	DW							X			X								X											
	WW					X		X			X								X						X					
	HW					X		X			X								X						X					
Mississippi	**DW**							**X**									**X**									**X**	**X**	**X**		
Missouri	DW							X											X				X							
	MICRO																													
Montana	**DW**			**X**				**X**																						
Nebraska	DW						X	IP																						
Nevada	**DW**							**X**							**X**							**X**		**X**						
	WW							**X**							**X**								**X**	**X**						
New Hampshire	DW			X		X	X	X	X												X									X
	WW			X		X	X	X	X											X	X									X
New Jersey	**DW**		**X**			**X**	**X**	**X**	**X**			**X**		**X**				**X**						**IP**	**X**					
	DW-Radiological																													
	WW		**X**	**X**		**X**	**X**	**X**	**X**			**X**	**X**	**X**				**X**	**X**	**X**	**X**			**IP**	**X**					
	Hazardous Waste		**X**	**X**		**X**	**X**	**X**	**X**			**X**	**X**					**X**	**X**	**X**	**x**			**IP**	**X**					
	CLP						**X**		**X**			**X**																		
New Mexico	DW-Asbestos																													
	DW - Dioxin													X																
New York	**DW**					**X**	**X**	**X**	**X**			**X**		**X**				**X**					**X**	**X**	**X**					**X**
	DW-Radiological																						**X**							
	WW		**X**	**X**		**X**	**X**	**X**	**X**			**X**		**X**				**X**	**X**	**X**	**X**		**X**		**X**					**X**

Certifying State/Program	Program Type	STL Anchorage	STL Austin	STL Baltimore	STL Billerica	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL Pensacola	STL Pittsburgh	STL Richland	STL St. Louis	STL Sacramento	STL Savannah	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield
	WW-Radiological																						**X**							
	HW		**X**	**X**		**X**	**X**	**X**	**X**			**X**		**X**				**X**	**X**	**X**	**X**		**X**		**X**					**X**
	CLP			**X**		**X**	**X**		**X**			**X**						**X**	**X**		**X**									
	Air/Emissions		**x**			**X**	**X**		**X**					**X**																
	DW-Asbestos				**X**																									
	Asbestos																													
North Carolina	DW					X		X											X						X	X				
	DW-Radiological																													
	DW-Asbestos				X																									
	WW/LC		X	X		X		X	X		X			X		X	X			X	X				X	X	X			
North Dakota	**DW-Asbestos**																													
	DW					**X**		**X**																**X**	**X**					
	HW					**X**		**X**			**X**			**X**					**X**	**X**				**X**						
	WW			**X**		**X**		**X**			**X**								**X**	**X**				**X**						
Ohio	VAP							X						X					X		IP									
Oklahoma	**WW**		**X**			**X**		**X**			**X**		**X**	**X**		**X**				**X**										
	DW							**X**																						
	HW		**X**			**X**		**X**			**X**		**X**	**X**		**X**				**X**										
Oregon																						X		x						
Pennsylvania	**DW**			**X**		**X**	**X**	**X**				**X**						**X**	**X**	**X**	**X**				**X**	**X**				
	DW-Radiological																						**IP**							
	DW-Asbestos				**X**																									
Rhode Island	DW			X			X	X			X	X						X								X	X			X

Certifying State/Program	Program Type	STL Anchorage	STL Austin	STL Baltimore	STL Billerica	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL Pensacola	STL Pittsburgh	STL Richland	STL St. Louis	STL Sacramento	STL Savannah	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield
	WW						X	X	X		X	X	X					X								X	X			X
	Asbestos																													
South Carolina	**DW**							**X**						**X**					**X**			**X**	**X**	**X**	**X**	**X**	**X**			
	WW		**X**	**X**		**IP**		**X**						**X**		**X**	**X**		**X**	**X**	**IP**			**X**	**X**	**X**	**X**			
	HW		**X**	**X**		**IP**		**X**			**X**			**X**		**X**	**X**		**X**	**X**	**IP**			**X**		**X**	**X**			
	DW-Asbestos				**X**																									
South Dakota	DW			X																										
Tennessee	**DW**			**X**		**X**		**X**			**X**			**X**		**X**	**X**		**X**	**X**					**X**		**X**			
	DW-Asbestos				**X**																									
Texas	DW							X																	X	X				
	MICRO																													
	TNRCC			X						X																				
Utah	**DW/HW/WW**			**X**				**X**	**IP**		**X**		**X**	**X**					**IP**			**X**	**X**	**X**						
	DW/HW/WW-Radiological																					**X**	**X**							
	UST							**X**							**X**															
	DW-Asbestos				**X**																									
Vermont	DW						X	X																						X
	Asbestos																													
	DW-Asbestos				X																									
Virginia	**DW**			**X**		**X**		**X**						**X**				**X**		**X**	**X**			**X**	**X**		**X**			
	DW-Radiological																													
	DW-Asbestos																													
	Asbestos																													

Certifying State/Program	Program Type	STL Anchorage	STL Austin	STL Baltimore	STL Billerica	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL Pensacola	STL Pittsburgh	STL Richland	STL St. Louis	STL Sacramento	STL Savannah	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield
Washington	DW																					X		X						
	WW			X		X		X	X		X			X	X					X		X	X	X	X					
	DW-Asbestos				X																									
	HW			X							X				X					X		X	X	X						
West Virginia	**DW**							**X**																						
	WW					**X**													**X**	**X**	**X**				**X**					
	HW						**X**												**X**	**X**	**X**				**X**					
Wisconsin	General					X			X					X					X											
	DW																							IP						
	WW		x					X			X													x	X					
	HW		x					X			X																			
	UST																													
Wyoming	**DW-Asbestos**				**X**																									
	DW-Radiological																													
	DW							**IP**																						
	UST																								**X**					
Puerto Rico	Drinking Water			X																						X				
	Power Authority																									X				
American Industrial Hygiene Association (AIHA)	Asbestos, Metals, Solvents																			X										
US Army Corps of Engineers	**Organics**	**X**	**X**	**X**			**X**	**X**	**X**		**X**		**X**	**X**	**X**				**X**	**X**	**X**		**x**	**x**	**X**	**X**	**X**	**X**		

Certifying State/Program	Program Type	STL Anchorage	STL Austin	STL Baltimore	STL Billerica	STL Buffalo	STL Burlington	STL Chicago	STL Connecticut	STL Corpus Christi	STL Denver	STL Edison	STL Houston	STL Knoxville	STL Los Angeles	STL Miami	STL Mobile	STL Newburgh	STL North Canton	STL Pensacola	STL Pittsburgh	STL Richland	STL St. Louis	STL Sacramento	STL Savannah	STL Tallahassee	STL Tampa East	STL Tampa West	STL Valparaiso	STL Westfield
	Inorganic	X	X	X			X	X	X		X		X	X	X				X	X	X		x	x	X	X	X	X		
	Air														x															
	Radiochemistry																													
US Department of Agriculture	**Foreign Soil Permit**	X	X	X		X	X	X	X		X	X	X		X	X	X	X	X	X	X	X	X	x	X	X	X	X		
USEPA Office of Groundwater & Drinking Water	**ICR Certificate**																X			X										
Air Force Center for Environmental Excellence	AFCEE	IP	x	X		X		X	X		X				X					X				X	X		x			
Naval Facilities Engineering Services Center	**NFESC**			X			X	X						X	X				X	X	X			x	X	X				
A2LA	ISO Guide 25			X		X		X					X	X			X								X					
	Environmental Testing			X		X		X						X																
	Lead Testing			X				X																						
A2LA	**ISO 9001**																													
ACIL-IAETL	Seal of Excellence					X	X													x				x						
Stds. Council of Canada	**General - MRA**					X		X																						
State of Colorado	Radiology Material License										X																			
US Nuclear Regulatory Commission	**Material License**								X													X	X					X		

The codes utilized for program types are described below:

DW Drinking Water certification

HW Hazardous Waste certification

WW Wastewater certification

X Laboratory has some form of certification under the specific program. Many states certify laboratories for specific parameters or tests within a category. The information in the table indicates the lab is certified in a general category of testing. Please contact your Account Executive or Project Manager if parameter specific certification information is required.

X* Laboratory has Interim status.

IP Laboratory is in the process of obtaining certification. Please contact laboratory for most current status.

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Appendix B-4

Level II Data Validation Checklist

DATA VALIDATION CHECKLIST

Sample Identification(s):

Sample Date(s):

Sample Team:

Sample Matrix:

Analyzing Laboratory:

Analyses:

QA Reporting Level:

Laboratory Report No.

ARCADIS Geraghty & Miller, Inc.
88 Duryea Road
Melville
New York 11747
Tel 631 249-7600
Fax 631 249-7610

Environmental

Project:

Project Number:

ANALYTICAL DATA PACKAGE DOCUMENTATION
GENERAL INFORMATION

	Reported		Performance Acceptable		Not
	No	Yes	No	Yes	Required
1. Sample results		X		X	
2. Parameters analyzed		X		X	
3. Method of analysis		X		X	
4. Reporting limits of analysis		X		X	
5. Sample collection date		X		X	
6. Laboratory sample received date		X		X	
7. Sample preparation/extraction date		X		X	
8. Sample analysis date		X		X	
9. Copy of chain-of-custody form signed by lab sample custodian		X		X	
10. Narrative summary of QA or sample problems provided		X		X	

QA - quality assurance

Comments: An ARCADIS G&M level II review was conducted on the data packages and any qualification of the data was determined using ,"USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review" (October 1999), "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review" (February 1994), and ARCADIS G&M professional judgment. Field data, field notes, and sampling logs were not reviewed.

INORGANIC ANALYSES
TOTAL METALS

	Reported		Performance Acceptable		Not
	No	Yes	No	Yes	Required
1. Holding times		X		X	
2. Blanks		X		X	
A. Preparation and calibration blanks		X	X		
B. Equipment rinsate blanks					X
C. Field blanks					X
3. Laboratory control sample (LCS) %R		X		X	
4. Matrix spike (MS) %R		X		X	
5. MS duplicate %R		X		X	
6. Laboratory duplicate RPD		X	X		
7. Field duplicate comparison					X

%R - percent recovery
%D - percent difference
RPD - relative percent difference
MSD - matrix spike duplicate
NA - not applicable or not analyzed
FAA - furnace atomic absorption
ICP - inductively coupled plasma atomic emission spectroscopy

Comments:
Performance was acceptable, with the following exceptions and notes.

INORGANIC ANALYSES
WET CHEMISTRY METHODS

	Reported		Performance Acceptable		Not
	No	Yes	No	Yes	Required
1. Holding times		X		X	
2. Reporting limits		X		X	
3. Blanks		X		X	
A. Preparation and calibration blanks		X		X	
B. Equipment rinsate blanks					X
C. Field blanks					X
4. Laboratory control sample (LCS) %R		X		X	
5. Matrix spike (MS) %R		X		X	
6. MS duplicate (MSD) %R		X		X	
7. Laboratory duplicate RPD		X		X	
8. Field duplicate comparison					X

%R percent recovery
LCS - laboratory control sample
RPD - relative percent difference
NA - not applicable or not analyzed
MSD - matrix spike duplicate

Comments:
Performance was acceptable, with the following exceptions and notes.

ORGANIC ANALYSES
VOLATILE ORGANIC COMPOUNDS

	Reported		Performance Acceptable		Not
	No	Yes	No	Yes	Required
GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)					
1. Holding times		X		X	
2. Blanks		X		X	
A. Water blanks (VOCs)		X		X	
B. Equipment rinsate blanks					X
C. Field Blanks					X
D. Trip blanks					X
3. Matrix spike (MS) %R					X
4. Matrix spike duplicate (MSD) %R					X
5. MS/MSD precision (RPD)					X
6. Laboratory control sample (LCS)					X
7. Surrogate spike recoveries		X		X	
8. Internal standard retention times and areas		X	X		
9. Field duplicate comparison					X

VOCs - volatile organic compounds
RRF - relative response factor
% RSD - percent relative standard deviation
TIC - tentatively identified compound
%D - percent drift
%R - percent recovery
RPD - relative percent difference
BS - blank spike
BSD - blank spike duplicate

Comments:
Performance was acceptable, with the following exception and notes.

ORGANIC ANALYSES
SEMIVOLATILE ORGANIC COMPOUNDS

	Reported		Performance Acceptable		Not
	No	Yes	No	Yes	Required
GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)					
1. Holding times		X		X	
A. Extraction holding time		X		X	
B. Analysis holding time		X		X	
2. Blanks		X		X	
A. Water blanks		X	X		
B. Extraction blanks					X
C. Equipment rinsate blanks					X
D. Field Blanks					X
3. Matrix spike (MS) %R					X
4. Matrix spike duplicate (MSD) %R					X
5. MS/MSD precision (RPD)					X
6. Laboratory control sample (LCS)					X
7. Surrogate spike recoveries		X	X		
8. Internal standard retention times and areas		X	X		
9. Field duplicate comparison					X

SVOCs - semivolatile organic compounds
RRF - relative response factor
% RSD - percent relative standard deviation
TIC - tentatively identified compound
%D - percent drift
%R - percent recovery
RPD - relative percent difference
BS - blank spike
BSD - blank spike duplicate

Comments:
Performance was acceptable, with the following exceptions and notes.

DATA VALIDATION CHECKLIST
SUMMARY AND DATA QUALIFIER CODES

ARCADIS Geraghty & Miller, Inc.
88 Duryea Road
Melville
New York 11747
Tel 631 249-7600
Fax 631 249-7610

Project Name
Project Number
Sample Identification(s)
Sample Date(s)
Sample Team
Sample Matrix
Analyzing Laboratory
Analyses
QA Reporting Level
Laboratory Report No.

Sample ID	Analyte(s)	Result	Qualifier	Reason(s)

VALIDATION PERFORMED BY: _____

VALIDATION PERFORMED BY SIGNATURE: _____

DATE: _____

PEER REVIEW BY: _____

PEER REVIEW BY SIGNATURE: _____

DATE: _____

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Appendix C

Health and Safety Plan (HASP)

Appendix C
Johnson Lake Investigation
Health and Safety Plan (HASP)

Owens-Brockway Glass Container, Inc.,
Portland, Oregon

ARCADIS

Infrastructure, buildings, environment, communications

ARCADIS



Katrina Leigh
Environmental Scientist



Phyllis C. Fuchsman
Environmental Scientist



Timothy R. Barber, PhD
Project Manager/Principal Scientist

Appendix C
Johnson Lake Investigation
Health and Safety Plan (HASP)

Owens-Brockway Glass
Container, Inc.,
Portland, Oregon

Prepared for:
Owens-Brockway Glass Container, Inc.

Prepared by:
ARCADIS G&M, Inc.
26451 Curtiss Wright Parkway
Suite 100
Cleveland
Ohio 44143
Tel 216 261 7373
Fax 216 261 7225

Our Ref.:
CL000233.0003

Date:
January 29, 2004

ARCADIS

Table of Contents

1. Introduction 1

2. Health and Safety Responsibility 1

2.1 Health and Safety Coordinator 2

2.2 ARCADIS Field Team Leader 3

2.3 Project Manager 3

2.4 Subcontractor 3

2.5 All On-Site Personnel and Visitors 4

3. Site Description 4

4. Planned Field Activities 5

5. Hazard Evaluation 5

5.1 Physical and Biological Hazards 5

5.2 Chemical Hazards 7

6. Personal Protective Equipment 8

7. Decontamination 8

8. Safe Work Practices 9

9. Emergency Procedures 9

10. Recordkeeping 10

12 References 10

Tables

C-1 Emergency Telephone Numbers

Figures

C-1 Map to Hospital

ARCADIS

Appendices

C-1 Worker and Visitor Acknowledgement Form

C-2 Daily Health and Safety Record (Tailgate Safety Meeting)

1. Introduction

ARCADIS prepared this site-specific Health and Safety Plan (HASP) for sampling associated with Johnson Lake (site) located in the Columbia Slough, Portland Oregon. This HASP addresses potential hazards to the investigation team and any associated subcontractors during on-site and field activities. The plan addresses applicable regulatory requirements, personnel responsibilities, protective equipment, decontamination procedures, and local medical facilities. This plan also identifies potential hazards associated with site-specific tasks and constituents known or believed to be present at the site.

ARCADIS subcontractors will be given copies of this plan and will be required, at a minimum, to follow the procedures described herein. Subcontractors will also be required to develop more extensive health and safety procedures for those activities for which they are responsible if this HASP does not adequately cover those activities. In this event, the subcontractors and the ARCADIS Health and Safety Coordinator will work together to develop adequate procedures prior to the initiation of that activity. ARCADIS claims no responsibility for its use by others, although other subcontractors working at the Site may use this HASP as a guidance document. In any event, ARCADIS does not guarantee the health and/or safety of any person entering this site. Strict adherence to the health and safety guidelines provided herein will reduce, but not eliminate, the potential for injury at this site. To this end, health and safety becomes the inherent responsibility of personnel working at the site.

This plan has been prepared based upon requirements in the Code of Federal Regulations, Title 29, Part 1910 (29 CFR 1910) pertaining to hazardous-waste site activities, and guidelines contained in the National Institute for Occupational Safety and Health (NIOSH), Occupational Safety and Health Administration (OSHA), U.S. Coast Guard (USCG), and U.S. Environmental Protection Agency (U.S. EPA) manual entitled *Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities* (October 1995).

2. Health and Safety Responsibility

Health and safety responsibility will follow a specific chain of command during all site activities. A description of key personnel and their responsibilities are listed below.

2.1 Health and Safety Coordinator

A Health and Safety Coordinator will be designated for each work task. The Health and Safety Coordinator will be responsible for all aspects of health and safety on the job site. A list of specific responsibilities follows:

- Selects personal protective equipment (PPE) required for site activities;
- Inspects and ensures proper use and operation of PPE;
- Controls entry and exit to the site;
- Ensures adherence to the HASP;
- Monitors the workers for signs of exposure;
- Monitors on-site hazards and conditions;
- Implements emergency procedures and proper decontamination;
- Defines work zones;
- Conducts "tailgate" health and safety meetings;
- Coordinates site activities and provides status reports to the Field Team Leader;
- Manages site activities; and
- Keeps written record of adherence to HASP.

The Health and Safety Coordinator will be responsible for stopping or starting work based on site conditions and/or emergency events. The Health and Safety Coordinator will take any additional measures necessary to provide a safe working environment, and will report to the Field Team Leader regarding work status, emergency events, and guidance in health and safety matters.

2.2 ARCADIS Field Team Leader

The Field Team Leader will be responsible for project completion and will have ultimate control over site activities. The Field Team Leader will prepare and direct all scopes of work, compile and coordinate the work team, choose the Health and Safety Coordinator, evaluate site conditions, and report to the project manager (PM).

2.3 Project Manager

The PM will be responsible for defining project objectives, approving scopes of work, and determining the ultimate chain of command.

2.4 Subcontractor

All ARCADIS subcontractors associated with this project are responsible for assigning specific work tasks to their employees. Each subcontractor's management will provide qualified employees and allocate sufficient time, materials, and equipment to safely complete assigned tasks. In particular, each subcontractor is responsible for equipping its personnel with any required PPE.

ARCADIS considers each subcontractor to be an expert in all aspects of the work operations for which they are tasked to provide, and each subcontractor is responsible for compliance with those regulatory requirements, which pertain to those services. Each subcontractor, in close coordination with ARCADIS health and safety personnel, is expected to perform its operations in accordance with its own unique safety policies and procedures, to ensure that hazards associated with the performance of the work activities are properly controlled. Copies of any required safety documentation for a subcontractor's work activities will be provided to ARCADIS for review by the Health and Safety Coordinator and the ARCADIS Field Team Leader prior to the start of on-site activities, if required. In the event that subcontractor procedures/requirements conflict with requirements specified in this HASP, the more stringent guidance will be adopted after discussion and agreement between the subcontractor and ARCADIS project health and safety personnel.

Hazards not listed in this HASP but known to the subcontractor, or known to be associated with the subcontractor's services, must be identified and addressed to the ARCADIS PM, Field Team Leader, and the Health and Safety Coordinator prior to beginning work operations. The Task Manager or authorized representative has the authority to halt the subcontractor’s operations, and to remove the subcontractor or

subcontractor employee from the site for failure to comply with established health and safety procedures or for operating in an unsafe manner.

2.5 All On-Site Personnel and Visitors

All personnel working for ARCADIS or its subcontractors are required to read and acknowledge their understanding of this HASP. All visitors to controlled areas of the site must receive a health and safety briefing from the Health and Safety Coordinator explaining applicable health and safety requirements and document their understanding of those requirements. All personnel are expected to abide by its requirements and cooperate with site supervision to ensure a safe and healthful work site. Personnel must immediately report any of the following to the Field Team Leader or Health and Safety Coordinator:

- Accidents and injuries, no matter how minor;
- Unexpected or uncontrolled releases of any hazardous substances;
- Any symptoms of exposure to a hazardous substance;
- Any unsafe or malfunctioning equipment; and
- Any changes in site conditions that may affect the health or safety of project personnel.

All personnel working on-site will be required to complete all sections (as appropriate) of the Worker and Visitor Acknowledgement Form located in Appendix C-1.

3. Site Description

Johnson Lake lies immediately to the north of the Owens Brockway facility, located at 5850 NE 92nd Drive, Multnomah County, in Portland, Oregon. The lake is within the Colombia Slough system. The Whitaker Slough runs immediately to the north of Johnson Lake, and the Columbia Slough is approximately 500 feet north of the Whitaker Slough (see site maps in Johnson Lake Investigation Work Plan). The Portland International Airport is located north of the Columbia Slough, between the slough and the Columbia River.

4. Planned Field Activities

This HASP is meant to encompass all field activities that will be performed by ARCADIS at the site. This includes but is not limited to collecting surface sediment and biota samples from Johnson Lake.

5. Hazard Evaluation

The following physical, biological, and chemical hazard evaluation is based upon a review of available site-specific information and upon available information regarding the toxicity and physical properties of the known constituents of interest.

5.1 Physical and Biological Hazards

Physical and biological hazards exist at the site because work will be conducted outdoors and may be performed in or around surface water and on overgrown areas and/or on steep terrain. In most cases common sense should rule to prevent accidents; however, the specific items listed below should be given particular attention.

Weather

It is recognized that hazards may result from adverse weather. A decision to discontinue sampling or other activity because of severe or threatening weather conditions (e.g. lightning, strong wind, heavy rain, very hot temperatures) will be made by the Health and Safety Coordinator.

Heat Stress

Heat stress is usually caused by a number of factors such as meteorological conditions, PPE, workload, and individual characteristics of site personnel. In order to minimize the potential for heat stress to occur, appropriate actions must be taken to prevent the onset of heat stress. Water for potable use will be made available during all site activities. Frequent rest periods should be taken in shaded areas to minimize fatigue and exposure to hot temperatures.

The Health and Safety Coordinator should monitor personnel for any signs of heat stress. These signs may include any of the following: impaired performance, loss of coordination, skin eruptions (red face or rashes), nausea, headaches, and cramps. If these signs are observed, the worker should be relieved of duties and made to rest until he/she can adequately recover, or appropriate medical attention should be administered as needed.

Cold Stress

Symptoms of cold stress should be monitored at temperatures below 50 degrees Fahrenheit. Two general categories of cold stress hazards include hypothermia and frostbite. Hypothermia occurs when the body cools down and its ability to stay warm fails. This condition is very serious and can cause death. Symptoms of hypothermia include shivering, numbness, glassy stare, apathy, and loss of consciousness. The temperature does not have to be below freezing for hypothermia to occur. If the conditions are right, hypothermia can occur when the temperature is near 50 degrees Fahrenheit. First aid for hypothermia includes removing any wet clothing, wrapping the victim in blankets and placing them in a warm place, and if the victim is conscious, slowly give them warm liquids. Avoid warming the victim too quickly since this can cause complications with the heart. Workers suffering from hypothermia should seek medical attention.

Frostbite is also a serious cold stress hazard and is caused by freezing of body parts, especially fingers and toes. Symptoms of frostbite include lack of feeling in the affected area, skin that appears waxy, is cold to the touch, or is discolored (flushed, white, yellow, or blue). To care for frostbite, the affected area should be placed in warm water (not exceeding 105 degrees Fahrenheit) until the skin looks red and feels warm. Loosely bandage the area with a dry sterile dressing. If fingers or toes are frostbitten, place cotton or gauze between them. Do not break any blisters and get prompt medical attention.

Boat Hazards

A small rowboat will be used to sample sediment and biota in any sampling zone of Johnson Lake that is unreachable by foot. While in a watercraft, it is important to constantly be aware of proper boat safety. Life vests will be worn at all times by workers while in the boat and all safety equipment will be checked prior to use to ensure proper working order. Standing in the boat will not be permitted. A first aid kit and cellular phone will be located on the boat in a plastic bag in the case of injury or other emergencies. Weather conditions will be monitored at all times for any sign of severe weather. If conditions turn severe, personnel will quickly row to shore and will not continue sampling until the threat is over. Workers will be aware of their surroundings and will use common sense at all times while off shore.

A subcontractor will conduct all boat electrofishing on Johnson Lake with oversite by one or more ARCADIS workers. In addition to the contractor's own health and safety protocols, the following procedures will be met: (1) all field workers must be trained in electrofishing safety precautions and unit operation procedures identified by the

electrofishing unit manufacturer; (2) each worker must be insulated from the water and the electrodes; (3) electrofishers/electrodes must be equipped with functional safety switches; (4) field workers must not reach into the water unless the electrodes have been removed from the water or the electrofisher has been disengaged; and (5) it is recommended at least two workers be certified in CPR (cardiopulmonary resuscitation) (U.S. EPA 1999).

Other Physical Hazards

General hazards associated with project work, such as insect or small animal bites, slip, trip and fall hazards, cuts, etc. should be addressed by the Health and Safety Coordinator during the "tailgate" health and safety meeting prior to commencing work each day. Standard first aid and safety precautions will be considered with respect to these items, and a first aid kit will be available on-site. Since areas of the site are overgrown, insects, small animals, thorny vegetation, and poison ivy may be of concern during field activities. Steep terrain and potentially unstable or uneven ground should be given constant attention.

5.2 Chemical Hazards

Polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), metals and pesticides have all been detected historically in Johnson Lake and are suspected to be present in lake sediment. Potential hazards associated with exposure to affected environmental media are less severe than those associated with exposure to the concentrated constituents themselves. The primary potential chemical hazard is exposure to PCBs in the sediment and/or surface water at the sample collection locations. Exposure to PCBs through dermal contact may result in various skin ailments, including chloracne. Ingestion of PCBs may result in liver toxicity. PCBs are carcinogenic to animals. General symptoms of PCB exposure may include skin and eye irritation and chloracne.

Exposure via Ingestion

Chemical exposure via ingestion will be mitigated through the use of the personal hygiene practices listed below:

- Eating, drinking, and/or smoking will be prohibited in the work area; and
- All workers must wash hands and face prior to and after consuming food, beverages, and/or smoking.

If significant PCB ingestion is suspected, medical attention should be provided immediately.

Exposure Via Dermal Contact

Chemical exposure via dermal contact will be mitigated through the use of PPE and common sense when handling liquids or solids within the work zone. The level of protection required will be as needed to prevent dermal contact. All workers will wear Level D PPE (see Section 6).

Eyes should be irrigated immediately if exposure to PCBs is suspected. Skin should be washed immediately if exposure to PCBs is expected.

Exposure Via Inhalation

Chemical exposure via inhalation is not believed to be a viable pathway due to the non-volatile nature of the constituents of interest (i.e., PCBs) and because sampling activities are limited to moist, vegetated, or inundated areas, which are not conducive to the generation of fugitive dusts, associated with dry conditions. Therefore, air monitoring will not be necessary during the sampling event.

6. Personal Protective Equipment

Level D protection will be worn at all times when on the Owens-Brockway property or when conducting off-site sampling. A description of the minimum requirements for Level D are provided below.

Level D

- Safety glasses;
- Latex gloves;
- Safety boots/shoes; and
- Hearing protection, as required.

7. Decontamination

All workers will decontaminate equipment and wash their hands and face prior to leaving the site. Disposable articles should be properly containerized and left in the

work area. Decontamination procedures will be performed in accordance with those developed and recommended by NIOSH, OSHA, USCG, and the U.S. EPA for use at hazardous waste sites. Although no specific procedures are included for Level D PPE decontamination, care should be taken to decontaminate or dispose of gloves and decontaminate boots and clothing as necessary.

8. Safe Work Practices

Workers responsible for conducting the planned sampling activities must be familiar with and should have on-site access to this HASP. All on-site employees will have documented 40-hour Health and Safety training and annual refresher training. All on-site employees will participate in a medical monitoring program.

Daily "tailgate" health and safety meetings will be conducted by the Health and Safety Coordinator prior to starting work to discuss work tasks and hazards as well as emergency procedures. The meeting will be documented in the format shown in Appendix C-2. In the event of an injury or illness caused by work at the site, the OSHA 301 form should be completed by the Health and Safety Coordinator and submitted to the Office Health & Safety Coordinator. Additionally, any unsafe work practices or conditions noted by the workers at the site should be reported to the Health and Safety Coordinator. The Health and Safety Coordinator should then stop work until the problem can be addressed or corrected.

9. Emergency Procedures

A plan for emergency situations has been developed to address immediate responses to existing or imminent hazards or problems. The Health and Safety Coordinator is responsible for investigating all emergency situations. Work will resume after the cause of the accident or hazard has been identified and corrective actions taken. If the emergency situation requires police, fire, and/or emergency rescue personnel, the telephone numbers listed in Table C-1 should be utilized to contact the appropriate emergency personnel. Caution should be used if dialing 911 from a portable or cellular telephone, as the communications systems for these types of telephones frequently connects calls placed to 911 to other cities/states. If mobile or cellular telephones are used, the seven-digit emergency numbers should be used.

Emergency Telephone Numbers

Telephone numbers for fire department, ambulance, police, and medical facilities are shown in Table C-1. In the event of an emergency, the Field Team Leader should be notified as soon as possible.

Emergency Route to Hospital

Eastmoreland Hospital is located at 10800 NE Hancock Street in Portland. The location is mapped in Figure C-1. Travel time from the site is approximately 5 minutes. Directions from the Owens-Brockway facility to the hospital are as follows:

- Take NE 92nd Drive south to NE Columbia Boulevard, and turn left.
- Bear left on NE Sandy Boulevard.
- Turn right on NE 102nd Avenue.
- Proceed 1.6 miles to the hospital.

10. Recordkeeping

The following documentation will be maintained and updated by the site Health and Safety Coordinator:

- Safety and Health Logbook, and forms therein.

Prior to program startup, a safety briefing will be conducted by the site Health and Safety Coordinator covering potential hazards and contents of this Plan.

12 References

U.S. EPA. 1999. Rapid Bioassessment Protocols for Use in Wadeable Streams and Rivers, Second Edition. U.S. Environmental Protection Agency, Office of Water. EPA-841-B-99-002. July 1999.

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Table C-1. Emergency Telephone Numbers, Site Specific HASP, Owens-Brockway, Portland, Oregon

Agency	Telephone Number
Portland City Police	503-823-4800
Wnba Portland Fire	503-797-9622
Ambulance	911
Eastmoreland Hospital	503-257-5500
National Response Center	800-424-8802
Oregon Poison Center	800-222-1222
ARCADIS Project Manager: Tim Barber	216-261-7373 (office) or 440-343-3557 (cell)
Owens-Brockway Plant Engineer: Bob Dolphin	507-251-9415
Oregon DEQ Project Manager: Jennifer Sutter	503-229-6148

Starting from: ❶ 5850 Ne 92nd Dr, Portland, OR 97220-1319

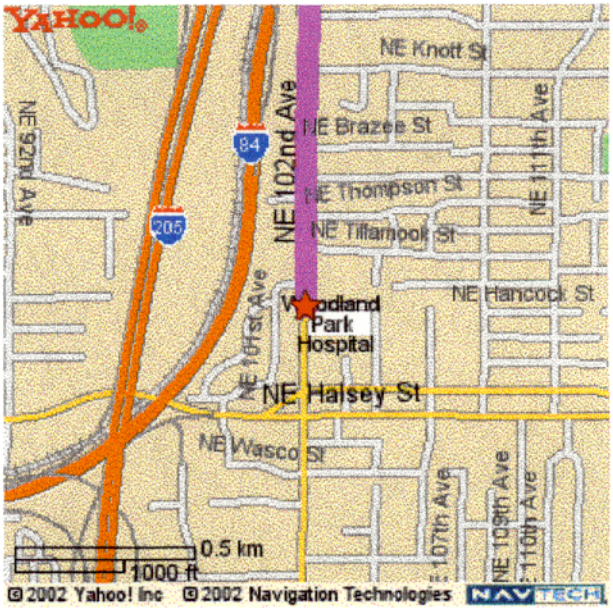
Arriving at: ❷ Eastmoreland Hospital
10300 NE Hancock St, Portland, OR 97220-3831
(503) 257-5500

Distance: 2.4 miles **Approximate Travel Time:** 5 mins

Full Route



Destination



Directions	Miles	
1. Start on **NE 92ND DR**	0.2	↑
2. Turn Left on **NE COLUMBIA BLVD**	0.4	↰
3. Bear Left on **NE SANDY BLVD**	0.2	↖
4. Turn Right on **NE 102ND AVE**	1.6	↱

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Map to Hospital
Owens-Brockway
Portland, Oregon

Date	Project Manager	Drawing Name
8NOV02	T. Barber	Figure 4-1
Drawn By	Lead Design Prof.	Checked
K. Leigh		B. Wiesner
Project Number		Figure Number
CL000233.0001		C-1

Appendix C-2

Worker and Visitor
Acknowledgement Form

HEALTH AND SAFETY PLAN REVIEW RECORD

Owens-Brockway Glass Container, Inc. **Johnson Lake, Portland, Oregon**	**HASP Date:** July 16, 2003 **Revision Number:** 0
	Review Type: Comprehensive

Printed Name	Signature	Employee #	Date Completed

Appendix C-2

Daily Health and Safety Record
(Tailgate Safety Meeting)

TAILGATE SAFETY MEETING

Prepared by _____

Client _____ Project _____

Date _____ Project Number _____

Work Location _____

Type of Work to be Done _____

SAFETY TOPICS PRESENTED

Chemical Hazards: _____

_____

Physical Hazards/Underground Utilities: _____

Protective Clothing/Equipment: _____

Special Equipment: _____

Emergency Procedures: _____

_____

Hospital/Clinic/Phone: _____ Paramedics ( ) _____

Hospital Address: _____

Other: _____

ATTENDEES:

NAME PRINTED	SIGNATURE
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Meeting Conducted by _____ _____

Signature

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Appendix D

Threatened and Endangered Species Review

Appendix D
Johnson Lake Investigation Threatened and Endangered Species Review

Owens-Brockway Glass Container, Inc.,
Portland, Oregon

ARCADIS
Infrastructure, buildings, environment, communications

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Table of Contents

1. Introduction **1**

2. Invertebrates **1**

2.1 Columbia Pebblesnail 1

2.2 Giant Columbia River Limpet 2

3. Reptiles **2**

3.1 Northwestern Pond Turtle 2

3.2 Painted Turtle 3

4. Fish **3**

4.1 Steelhead 3

4.2 Chinook Salmon 4

4.3 Coho Salmon 5

5. Birds **5**

5.1 Bald Eagle 5

5.2 Purple Martin 6

5.3 Western Yellow-Billed Cuckoo 6

6. Mammals **7**

6.1 Pacific Western Big-Eared Bat 7

7. References **8**

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Appendix D
Johnson Lake Investigation
Threatened and Endangered Species Review

Owens-Brockway, Portland, OR

1. Introduction

Records of rare, threatened, and endangered species occurring within two miles of the project area (Johnson Lake and the surrounding slough system) were obtained from the Oregon Natural Heritage Information Center (ORNHIC) on March 6, 2003 to support the selection of receptors of interest for the Ecological Risk Assessment (ERA). The focus of the search was to determine if sensitive species could potentially occur in or obtain prey from Johnson Lake. The species identified in the search are listed in this appendix with a brief description of their habitat and life history.

Observations of eight species were noted within the two-mile radius of the project area. The locations of the observations are confidential; however, none of the eight species were observed in Johnson Lake. In addition, ORNHIC noted three listed anadromous fish species that may be present within the waterways contained in the general area (Lower Columbia River). The ORNHIC does not have comprehensive records available for anadromous fish in Oregon and thus does not provide specific observation records for these species. Overall, most of the species identified by ORNHIC are unlikely to inhabit or obtain a significant portion of their diet from Johnson Lake. However, two turtle species could potentially inhabit the lake. A more focused threatened and endangered species search will be conducted to determine whether these species actually occur in Johnson Lake.

2. Invertebrates

The Columbia pebblesnail (*Fluminicola fuscus*) and the giant Columbia River limpet (*Fisherola nuttalli*) were observed within the two-mile radius of the project area. The Columbia pebblesnail is a federal species of concern but is not listed by the state of Oregon. The giant Columbia River limpet (*Fisherola nuttalli*) is not listed by either federal or state agencies but is tracked by ORNHIC.

2.1 Columbia Pebblesnail

The Columbia pebblesnail, or great Columbia River spire snail, is a member of the Hydrobiidae family, which includes several species of endangered aquatic snails. All species of this family have gills and thus require streams with a high dissolved oxygen content. Suitable streams for hydrobiid snails must be relatively pristine as well, since high turbidity levels may injure or clog their fragile gill tissue. Due to these requirements, the Columbia pebble snail is only found in the colder, clear streams of the Snake-Columbia River system (Hershler and Frest 1996, Neitzel and Frest 1990).

Columbia pebblesnails graze on submerged gravel, scraping algae off the rocks with a toothed appendage called a radula. These snails are quite short-lived, usually reaching sexual maturity within a year, at which time they breed and die. Because about 90 percent of the population turns over annually, a disruption or disturbance during the breeding season can have devastating effects. Due to its preference for cold, clear, well-oxygenated waters, it is highly unlikely that Columbia pebble snails could occur in Johnson Lake.

2.2 Giant Columbia River Limpet

The range of the giant Columbia River limpet or shortface lanx formerly included most of the Columbia River basin, but currently this species is restricted to only a few remaining sites (Neitzel and Frest 1990). The giant Columbia River limpet occurs in streams that are relatively fast moving, clear, cold, and well oxygenated. This species feeds on algal cells and plant matter that are found among the rocks and pebbles underlying these streams. Because of these habitat requirements, the giant Columbia River limpet has been significantly affected by the building of dams along the Snake and Columbia Rivers (Neitzel and Frest 1990). Due to its preference for fast moving, cold, clear waters, it is highly unlikely that the giant Columbia River limpet could occur in Johnson Lake.

3. Reptiles

The northwestern pond turtle (*Emys marmorata marmorata*) and the western painted turtle (*Chrysemys picta*) were observed within the two-mile radius of the project area. The northwestern pond turtle is a federal species of concern and is listed by the state of Oregon as a sensitive-critical species. The western painted turtle is not federally listed, but it is listed as a sensitive-critical species by the state of Oregon.

3.1 Northwestern Pond Turtle

Northwestern pond turtles are found in generally quiet water that ranges in salinity content from fresh to brackish to seawater (Holland 1991). Pond turtles are found near a wide variety of wetlands, including ponds, marshes, lakes, streams, irrigation ditches, and vernal pools. Aquatic habitats with adequate vegetative cover and exposed basking sites are utilized. Northwestern pond turtles are omnivorous generalists and opportunistic predators, eating small insects, aquatic invertebrates, fish, frogs, snakes, birds, and mammals. Wary and secretive, the pond turtle prefers habitats with large areas for cover (logs, algae, vegetation) and basking (logs, boulders). Like painted

turtles, northwestern pond turtles have been observed within 2 miles of the Columbia Slough and could potentially occur in the slow moving waters of Johnson Lake.

3.2 Painted Turtle

Western painted turtles range from western Ontario and British Columbia south to Missouri, northern Oklahoma, eastern Colorado, Wyoming, Idaho and northern Oregon. Isolated populations have been observed in Texas, New Mexico, Arizona, Utah and Chihuahua, Mexico (Cohen 1992). Wild painted turtles prefer slow-moving shallow waters of ponds, marshes, creeks, and lakes with soft, muddy bottoms, suitable basking sites, and ample aquatic vegetation. Painted turtles are diurnal, spending the nights sleeping on the pond bottom or on a partially submerged object. They become active at sunrise and bask for several hours before foraging for food in late morning. They may forage again in late afternoon into early evening (Cohen 1992).

Painted turtles have a varied diet. When they are young, they are mostly carnivorous. At this age they eat mostly maggots, larvae, and beetles. As they mature, they become omnivorous, eating snails, insects, crayfish, leeches, tadpoles, small fish, and many types of plants (Cohen 1992).

Painted turtles have been observed along the Columbia Slough in areas with abundant sunning logs, submergent vegetation and slow moving water. Johnson Lake is accessible from the Columbia Slough and provides adequate habitat for painted turtles. Therefore, western painted turtles may be present in Johnson Lake.

4. Fish

The ORNHIC does not maintain records of specific observations of anadromous fish species, and therefore it is not known whether such species occur within a two-mile radius of the project area. However, three anadromous fish species could potentially occur within this area. Steelhead (*Oncorhynchus mykiss*) and Chinook salmon (*Oncorhynchus tschawytscha*) are federally listed threatened species. Coho salmon (*Onchorhynchus kisutch*) is a candidate species for federal status.

4.1 Steelhead

Steelhead habitat requirements change as they go through different life phases. Adult steelhead need to have access to their natal streams for spawning. This means that streams must be free of barriers to migration, as the majority of spawning occurs in the

upper reaches of tributaries. Adults also need access to spawning gravel in areas free of heavy sedimentation with adequate flow and cool, clear water. Steelhead utilize gravel that is between 0.5 to 6 inches in diameter, dominated by 2 to 3 inch gravel. Escape cover for spawning adults is also important (McEwan and Jackson 2003).

For steelhead eggs and pre-emergent fry, the most important consideration in terms of habitat is cool water with adequate dissolved oxygen. Fine sediment will smother developing eggs, so the area must also be free of silt and the water clear. During their first summer, juvenile steelhead are typically found in relatively shallow areas with cobble and boulder bottoms. They reside at the downstream end of riffles less than two feet deep. Juvenile steelhead prefer areas with woody debris accumulation (logs or tree roots). Cover structures such as boulder clusters and root wads provide both summer and winter rearing habit. Surface turbulence (or white water) provides another source of cover during the summer months. As juvenile steelhead grow, pools become an important habitat component. The best pools for habitat are those with abundant escape cover in the form of large woody debris, undercut banks, root masses, and large boulders (McEwan and Jackson, 2003).

Cool, clear water is essential for the survival of steelhead during all portions of their life cycle. Due to this necessity, it is unlikely steelhead salmon would occur in Johnson Lake, where flow is minimal and waters are warm. Additionally, the warm water temperatures and low oxygen content in the upper slough act as barriers to migration from the lower slough to Johnson Lake. Furthermore, a levee, associated with the Peninsula Drainage Canal, closes off the canal at the southern end and physically separates the upper slough from the lower slough, creating a barrier to migration. When the floodgates of the levee are opened, water flows freely and fish can swim between the upper slough and lower slough. However, this event happens very rarely and usually at times of the year where water levels and water temperatures make salmon travel so far upstream unlikely (personal communication, April 29, 2003, Susan Barthel, Columbia Slough Program Coordinator).

4.2 Chinook Salmon

Chinook salmon are native to the Pacific Coast from southern California to northern Alaska. Chinook spawn in streams that are larger and deeper than other salmon utilize. Freshwater streams and estuaries provide important habitat for Chinook salmon. Fry and smolts usually stay in freshwater from 1 to 18 months before traveling downstream to estuaries. Chinook feed on terrestrial and aquatic insects, amphipods, and other crustaceans while young and are picisivorous when older. Eggs are laid in deeper

water with large gravel and need cool water and good flow to survive. Survival of eggs is poor in areas of high siltation, high water temperatures, low oxygen conditions, and low flow (Streamnet 1998).

Chinook salmon have been observed in the lower Columbia Slough. However, because of their habitat requirements and the prevention of migration by structural and biological factors (i.e., peninsula drainage levee, warm water temperatures, low oxygen), chinook salmon are unlikely to occur in Johnson Lake.

4.3 Coho Salmon

Coho salmon production extends from Point Hope, Alaska to Monterey Bay, California. Wild coho populations have been identified in several Oregon coastal rivers, including the Columbia River. Coho salmon prefer small, relatively low gradient tributary streams with woody debris and shade for spawning and juvenile rearing and may use lakes for rearing where available. They prefer pea to orange size spawning gravel and over-winter in off-channel alcoves and beaver ponds. Survival of eggs is poor in areas of high siltation. Coho fry feed on aquatic insects, zooplankton, and small fish. Adults are piscivorous (ODFW 1996; Streamnet 1998).

Coho salmon have been observed in the lower Columbia Slough. However, it is unlikely they could travel east of the Peninsula Drainage Canal and to Johnson Lake, due to structural and biological barriers.

5. Birds

Three bird species were observed within the two-mile radius of the project area: bald eagles *(Haliaeetus leucocephalus)*, a federal and state threatened species; purple martins (*Progne subis*), a federal species of concern and a sensitive-critical species in the state of Oregon; and western yellow-billed cuckoos (*Coccyzus americanus*), a candidate for federal status but not listed by the state.

5.1 Bald Eagle

The bald eagle is a large predatory raptor that occurs "primarily near seacoasts, rivers and large lakes, breeding in tall trees or on cliffs" (American Ornithologists' Union 1983). They are primarily fish eaters but will take whatever prey is available and easiest to obtain, including birds, invertebrates, turtles, and small mammals. Bald eagles prefer large bodies of open water for feeding and are rarely associated with

smaller streams or ponds (Leighton, Gerrard et al. 1979). Lakes less than 11 km in circumference do not constitute primary breeding habitat for bald eagles; the optimal size is at least 2500 acres (Leighton, Gerrard et al. 1979; Whitfield, Gerrard et al. 1974; USFWS 1986).

Bald eagles have been observed along the Columbia River, which meets the habitat requirements described above. In contrast, Johnson Lake has a surface area of only 6 acres, much less than the optimal size of 2500 acres. Therefore, it is unlikely that bald eagles would nest near or obtain a significant portion of their diet from Johnson Lake.

5.2 Purple Martin

Purple martins are the largest member of the swallow family in North America, measuring 7½ inches long and weighing 1.9 ounces (PMCA 2003). One subspecies, *Progne subis arboricola*, is known to breed along the Pacific coast and in the Rocky Mountains. Purple martins spend the non-breeding season in Brazil and migrate to North America to nest. In the Pacific Northwest, martins are beginning to use gourds and clusters of single-unit boxes for nesting. Martins, like all swallows, are aerial insectivores. They eat only flying insects, which they catch in flight. Their diverse diet includes dragonflies, damselflies, flies, midges, mayflies, stinkbugs, leafhoppers, Japanese beetles, June bugs, butterflies, moths, grasshoppers, cicadas, bees, wasps, flying ants, and ballooning spiders. Since purple martins feed only on flying insects, they are extremely vulnerable to starvation during extended periods of cool and/or rainy weather (PMCA 2003).

Purple martins have been observed along the Columbia River, where nest boxes or other suitable nesting habitat (i.e., pilings) are present. There have been no reports of purple martins around Johnson Lake, and nest boxes and suitable pilings are not present around the lake. Therefore, purple martins are not likely to occur in the vicinity Johnson Lake.

5.3 Western Yellow-Billed Cuckoo

Western yellow-billed cuckoos are obligate riparian nesters, meaning they only breed in streamside forests, especially those dominated by willow and cottonwood stands. The humid, shady environment provided by these forests provides a protective microclimate sheltering nesting birds, eggs, and fledglings from the heat and dryness prevalent in late summer across the western United States. Most nesting in the western United States occurs within relatively large patches of riparian forest, usually 25 to 100

acres in extent (SWCBD 2003). While yellow-billed cuckoos have been known to eat beetles, cicadas, bugs, wasps, flies, katydids, dragonflies, damselflies, praying mantids, lacewings, mosquito hawks, cankerworms, fall webworms, and even tree frogs, more than 75% of their diet is made up of grasshoppers and caterpillars (SWCBD 2003). Thus, adult forms of aquatic insects make up only a small portion of the yellow-billed cuckoo's diet.

Western yellow-billed cuckoos have been observed along the Columbia River but have not been reported near Johnson Lake. It is unlikely cuckoos would occur near Johnson Lake, since the Colombia Slough system does not provide the large acreage of riparian forest cuckoos prefer.

6. Mammals

Pacific western big-eared bats (*Corynorhinus townsendii townsendii*) were observed within the two-mile radius of the project area. These bats are a federal species of concern and a sensitive-critical species in the state of Oregon.

6.1 Pacific Western Big-Eared Bat

Pacific western big-eared bats or Townsend's bats occur in a variety of habitats, from desert shrub to deciduous and coniferous forests at a wide range of elevations. This species is captured or observed in abandoned mines and both unoccupied and actively used old buildings, where it may be considered a pest. It is probable that hollow cavities in large trees or snags may constitute an important undocumented resource for maternity colonies of this species. Population densities of western populations are approximately 1 bat per 343 acres (Idaho Museum of Natural History [IMNH] 2003). Pacific western big-eared bats were observed near a cave on the east side of Portland. Caves have not been observed near Johnson Lake; however hollow tree habitats, which may provide shelter, are likely present along the slough.

The *C. townsendii* diet consists mostly of moths in the family Noctuidae. Beetles and flies and lesser amounts of other insects may also be consumed (IMNH 2003). Thus, although Pacific western big-eared bats could be present near Johnson Lake, they would not feed to a significant extent on aquatic organisms from the lake.

7. References

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Appendix E

Project Management Plan (PMP)

Appendix E
Johnson Lake Investigation Project Management Plan (PMP)

Owens-Brockway, Portland, Oregon

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Table of Contents

1.	Project Management Plan (PMP)	1
2.	Site Investigation Report	1
3.	Risk Assessment	2
4.	Schedule	2

1. Project Management Plan (PMP)

This Project Management Plan (PMP) addresses the reporting and general schedule of events associated with the sampling activities presented in Revision 2 of the Johnson Lake Investigation Work Plan (IWP).

2. Site Investigation Report

Following laboratory and ARCADIS quality assurance and quality control (QA/QC) of the sampling data, a Site Investigation Report will be generated which will include, but not be limited to, the following sections:

- Executive Summary
- Introduction
- Site Background – This section will include a discussion and supporting maps of the Owens-Brockway site and surrounding facilities.
- Site Investigation – This section will include a discussion of the investigative procedures and results for the sediment and biota samples (including sample locations, number of samples, sampling and analytical procedures, analytical data, etc.).
- Summary and conclusions
- Appendix – Detailed information supporting the results of the sampling event will be submitted in the Appendix of the report.

The report will be distributed in the following manner:

1. Two bound and one unbound copy(s) of all work plans and reports will be submitted to Oregon Department of Environmental Quality (DEQ).
2. At the DEQ's request, all copies will be duplex printed on recycled paper, if available.
3. Electronic copies of work plans and reports, including all data and figures, if requested, will be submitted in Microsoft Office or ArcView compatible

format. All photographs will be submitted in both hard copy and electronic file formats.

3. Risk Assessment

As described in the scope of work attached to the Voluntary Cleanup Program letter agreement and in Revision 2 of the Johnson Lake IWP, a risk assessment work plan will be prepared following a preliminary review of the sampling results. The risk assessment work plan will contain a schedule for a human health risk assessment report and an ecological risk assessment report.

4. Schedule

The schedule of events related to the sampling activities in this IWP is presented below. All durations are estimates and may change due to unforeseeable circumstances (e.g., inclement weather). All efforts will be made, however, to adhere to the schedule.

SUBMITTALS	SCHEDULE
Submit Draft Investigation Work Plan (IWP)	To DEQ within 30 days of execution of letter agreement
DEQ Review and Comment on Draft IWP	30 days from receipt of IWP
Final IWP	30 days from receipt of DEQ comments
Initiation of Field Activities	60 days from DEQ approval of Final IWP
Laboratory Analysis and Data Validation	8 weeks from laboratory receipt of samples
Site Investigation Report and Risk Assessment Work Plan	45 days from completion of data validation

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Attachment 1

Oregon DEQ's Comments on the Johnson Lake Investigation Work Plan, dated June 5, 2003

Oregon
Theodore Kulongoski, Governor

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June 5, 2003

Ms. Keely O'Bryan
Thompson Hine
3900 Key Center
127 Public Square
Cleveland, OH 44114

Re: Owens-Brockway Glass Containers, ECSI No. 1311

Dear Keely:

Thank you for providing the draft Investigation Work Plan for Johnson Lake (4/30/03) located within the Columbia Slough watershed in Portland, Oregon. We are impressed with the overall scope of work proposed in this document and believe that it can provide the data necessary to assess the risk posed by contaminants detected in the lake sediments. We have the following comments which should be addressed in the revised investigation work plan for this site. We would be happy to meet with you to discuss these comments. The revised work plan should be submitted no later than July 17, 2003.

Comments

1. This work plan addresses investigation in Johnson Lake only. Since DEQ has identified investigation work that needs to be completed in the upland portion of the facility as well as Johnson Lake, it would be helpful to indicate when the upland work will be completed and how the current effort fits into the overall assessment of the Owens-Brockway facility.

2. The summary of previous investigations provided in Section 2.2 should include the October 2000 bank sediment sampling conducted at Owens Brockway by Kennedy/Jenks Consultants in which PCB Aroclor 1260 was detected at up to 5,270 ppb.

3. The screening evaluation focused only on chemicals detected in lake sediments during previous investigations. It would likely be more efficient to include in addition, any potential contaminants that may have migrated to the lake as a result of upland activities at the Owens-Brockway facility. We do not expect that this would significantly expand the list of chemicals of interest, but it would at least require evaluation of petroleum hydrocarbons.

4. The statement at the top of page 5 indicating that an ecological screening evaluation is expected to be protective of human health is not accurate for compounds that bioaccumulate and pose a threat to human health via fish ingestion as this pathway generally results in a lower screening value in sediments.

5. The equilibrium partitioning approach to identifying sediment screening levels described on pages 6 and 7, appears to generate results consistent with those achieved using DEQ screening values, with the clarification discussed in the following comment. Consequently, it is acceptable to use in this instance.

6. While DEQ sediment screening levels may not be based on a specific dose-response study, they do reflect risk or lack of risk associated with particular concentrations of contaminants, and in that sense they do reflect a cause-effect relationship. The repeated statement, in Section 3.1.2 that this is not the case is misleading. With respect to the conclusions regarding specific contaminants of interest, we generally agree with the summary of chemicals of interest identified in Section 3.1.3 with the addition of petroleum hydrocarbons. However, we note that this screening was conducted on a very small data set and future data collected at the site could result in changes to this list. Since many of the compounds screened out are included in the analyte list for the proposed laboratory methods, we recommend continuing to report concentrations of those compounds (e.g., barium, phenol, endrin ketone). Also, it may be important to the interpretation of bioassay results to include all contaminants that potentially may be present in the sediment.

7. The work plan should include a conceptual site model that identifies potential sources, exposure pathways and receptors. The conceptual model for ecological receptors should clearly identify those receptors selected as assessment entities, as well as the reason for their selection. The analyses that will be conducted should then be linked to the conceptual site model so the reason for conducting each test is clear (in terms of how it will help us interpret risk at the site). It is also important that the sequence for completing the identified evaluations be clarified. Certain evaluations (examples indicated in comments below) may not be necessary if particular exposure pathways are found not to pose an unacceptable risk.

8. The last sentence of Section 3.1.3 states that PAHs, cadmium, lead, and zinc do not biomagnify; however these compounds are included in the list of bioaccumulative compounds developed by U.S. EPA Region 10 for the Dredge Material Management Program. Their presence on this list indicates that they may be expected to accumulate in fish tissue at levels of concern to consumers of fish. This will likely be the exposure pathway that results in the lowest sediment screening level.

9. Further explanation of how the paired surface water and sediment chemistry data, referenced in the first paragraph of Section 3.2, will be collected and how they will be used to interpret sediment toxicity results should be provided.

10. Four different toxicity tests are identified in Section 3.2.1 and the work plan indicates that each test will be used to assess survival, growth and reproduction. The conceptual site model and associated discussion described in comment 7 should provide the basis for

conducting these tests. Note that the surface water toxicity tests identified in the 3rd and 4th bullets are generally only warranted in situations where there is an on-going release directly to the surface water and may not provide information on risks associated with contaminated sediments.

11. The second paragraph of Section 3.2.1 describes how bioassay test results will be evaluated, suggesting that the same interpretive criteria will be used for all 4 tests and all 3 endpoints. The work plan should provide interpretive criteria specific to each test and endpoint. Also, the work plan should indicate whether results will be compared to a control and/or reference sediment sample, or the process for deciding to use one or the other should be described. The percent difference in results indicating an effect will likely be different depending on whether a reference sediment or a control sediment is used.

12. We question the value of collecting surface water samples as indicated in Section 3.2.2 as the detection limits will likely be too high to assess whether contaminants are present at levels of concern. Further discussion of the basis for conducting this sampling should be provided in the Conceptual Site Model and associated discussion. It is unlikely to be warranted unless there is on-going release of contaminants to the lake.

13. The detailed work plan should indicate the number of samples that will be collected for each type of analyses described, sample collection techniques, and sample locations. A map should be provided that identifies sample locations. It will likely be useful to include some biased sampling in the vicinity of outfalls in addition to random samples representing the grids identified in Figure 3-2. Note that, Kennedy-Jenks Consultants experienced difficulty collecting sediment samples due to a "hard pan" layer they encountered during previous sediment sampling at this site. It may be useful to review their observations prior to conducting field work and be prepared with appropriate sample collection tools to ensure that this issue will not prevent collection of sediment samples during this investigation.

14. As we indicated in previous comments (letters dated 12/20/02 and 2/10/03), the PCB homologue analysis described in the first bullet of Section 3.2.2 will not provide data that will allow for determination of PCB toxic equivalency. A PCB congener analysis should be conducted on the 18 NOAA status and trends core congeners (8, 18, 28, 44, 52, 66, 101, 105, 118, 128, 138, 153, 170, 180, 187, 195, 206, 209) using currently available EPA Method 1668 (Revision A) HRGC/HRMS techniques. Note that it will likely be most efficient to conduct this analysis on tissue samples to determine if a complete risk pathway exists. If PCB congeners are not detected at levels of concern in fish tissue and bioassay results do not indicate toxicity, further sediment characterization will not be necessary.

15. Again it will be important to describe the sequence of planned testing and how test results will be used to assess complete exposure pathways identified in the conceptual site

model, in determining the need for some of the testing described on page 14 of the draft work plan. For example, bioassay and tissue sampling results may address the bioavailability of contaminants such that AVS and pore water selenium analyses are not necessary

16. We question whether it will be practical to collect sufficient tissue samples to adequately represent all the categories of biota identified in Section 3.2.4. The conceptual site model and associated discussion should clearly indicate the basis for sampling each category of biota and how the results will be used to estimate risk. It may only be necessary to collect samples for representative species of carnivorous, omnivorous, and bottom-feeding fish and focus on larger fish in these categories, since they are more likely to accumulate higher concentrations of contaminants. The work plan should describe sample collection procedures and numbers of samples that will be collected.

17. A beneficial land use evaluation must be completed to support the exposure scenarios described in Section 4.1. This would provide the foundation for eliminating certain potential land uses such as residential use. Also, the trespasser scenario should more accurately be described as the site visitor scenario with recreational site use described, since much of Johnson Lake will be open to the public. The likelihood that people will swim or wade in Johnson Lake should be addressed in this discussion as well as in the conceptual site model described in comment 7.

18. Note that fish ingestion rates, referenced in the description of the hypothetical angler scenario, page 18, should reflect the Slough-specific data developed in the Technical Memorandum on the Results of the 1995 Fish Consumption and Recreational Use Surveys (Adolfson and Associates, Inc, 12/18/95).

19. The detailed work plan should include a table of the exposure and toxicity factors that will be used in the risk assessment. The associated equations and processes that will be used to calculate risk should also be clearly outlined.

20. The detailed work plan should include a sampling and analysis plan (SAP) including tables with analytical methods and associated detection limits, a health and safety plan (HASP), and a schedule for conducting the proposed work. The SAP and HASP previously submitted may be referenced as appropriate.

If you have any questions, please call me at (503) 229-6148.

Sincerely,

Jennifer Sutter, Project Manager
Voluntary Cleanup/Portland Harbor

Cc: Mark Tussing, Owens-Brockway
Susan Smith, Owens-Brockway
Alicia Voss, VCPH/NWR
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Attachment 2

ARCADIS's Response to the Oregon DEQ's June 2003 Comments on the Johnson Lake Investigation Work Plan

ARCADIS

Response to June 5, 2003 Comments on Investigation Work Plan for Johnson Lake

Comments are provided in italics, followed by ARCADIS' response.

1. This work plan addresses investigation in Johnson Lake only. Since DEQ has identified investigation work that needs to be completed in the upland portion of the facility as well as Johnson Lake, it would be helpful to indicate when the upland work will be completed and how the current effort fits into the overall assessment of the Owens-Brockway facility.

In response to this comment, the following will be added to Section 1.1 of the work plan: "A separate work plan addressing on-site, upland areas that might act as a source of constituents of interest to Johnson Lake will be completed based on the results of the Johnson Lake investigation, as appropriate."

2. The summary of previous investigations provided in Section 2.2 should include the October 2000 bank sediment sampling conducted at Owens Brockway by Kennedy/Jenks Consultants in which PCB Aroclor 1260 was detected at up to 5,270 ppb.

The sampling event referenced in this comment was not included in the work plan, because it involved the collection of soil rather than sediment. As described in response to Comment 3 (below), the revised work plan will consider soil data collected adjacent to Johnson Lake. Therefore, the October 2000 soil data will be included.

3. The screening evaluation focused only on chemicals detected in lake sediments during previous investigations. It would likely be more efficient to include in addition, any potential contaminants that may have migrated to the lake as a result of upland activities at the Owens-Brockway facility. We do not expect that this would significantly expand the list of chemicals of interest, but it would at least require evaluation of petroleum hydrocarbons.

The revised work plan will include a comparison of chemical concentrations in soil adjacent to Johnson Lake with screening values developed for sediment. This comparison will be conducted for the purpose of identifying chemicals of interest in the work plan, because the existing sediment data set for Johnson Lake provides limited spatial coverage. The revised list of chemicals of interest will include PCBs, PAHs, two pesticides (DDT and endosulfan) and their metabolites, and eight metals (arsenic, cadmium, copper, chromium, lead, nickel, selenium, zinc). It is important to note that sediment-dwelling organisms are not exposed to upland soil. The sampling activities proposed in support of the human health and ecological risk assessments will provide sufficient spatial coverage, such that comparisons of soil data to sediment benchmarks will not be retained in the risk assessments.

Petroleum hydrocarbons will not be added as a constituent of interest for Johnson Lake, because petroleum hydrocarbon data cannot be interpreted for risk assessment purposes. Polycyclic aromatic hydrocarbons (PAHs) and benzene, toluene, ethylbenzene, and xylenes (BTEX) are the toxicologically relevant fractions of petroleum hydrocarbons. The BTEX compounds either were never detected in Johnson Lake sediment or on-site soil or were screened out based on comparison

with sediment screening values. Therefore, the analysis of PAHs proposed in the work plan will adequately address any issues associated with petroleum hydrocarbons in Johnson Lake.

4. The statement at the top of page 5 indicating that an ecological screening evaluation is expected to be protective of human health is not accurate for compounds that bioaccumulate and pose a threat to human health via fish ingestion as this pathway generally results in a lower screening value in sediments.

The statement will be removed from the revised work plan.

5. The equilibrium partitioning approach to identifying sediment screening levels described on pages 6 and 7, appears to generate results consistent with those achieved using DEQ screening values, with the clarification discussed in the following comment. Consequently, it is acceptable to use in this instance.

No response is required.

6. While DEQ sediment screening levels may not be based on a specific dose-response study, they do reflect risk or lack of risk associated with particular concentrations of contaminants, and in the sense they do reflect a cause-effect relationship. The repeated statement, in Section 3.1.2 that this is not the case is misleading. With respect to the conclusions regarding specific contaminants of interest, we generally agree with the summary of chemicals of interest identified in Section 3.1.3 with the addition of petroleum hydrocarbons. However, we note that this screening was conducted on a very small data set and future data collected at the site could result in charges to this list. Since many of the compounds screened out are included in the analyte list for the proposed laboratory methods, we recommend continuing to report concentrations of those compounds (e.g., barium, phenol, endrin ketone). Also, it may be important to the interpretation of bioassay results to include all contaminants that potentially may be present in the sediment.

It is important that the appropriate uses and limitations of the Oregon DEQ's screening values be clearly stated. Exceedance of these screening values, by itself, does not indicate an unacceptable risk. As described in the work plan, the DEQ's aquatic life values are based on associations between chemical concentrations and various measures of adverse effects, in field-collected sediments containing complex chemical mixtures. They do not represent cause-effect, concentration-response relationships for individual chemicals. Rather, each aquatic life value serves as an indicator of an overall level of contamination (by chemical mixtures) that is intended to be predictive of a lack of toxicity. While this information will be retained in the revised work plan, Section 3.1.2 will be edited to minimize repeated statements to the same effect.

We agree that, where the proposed analytical methods provide results for additional chemicals that were not identified in the work plan as chemicals of interest, all results will be reported and incorporated in an updated screening evaluation. Specifically, all pesticides included in the standard Method 8081 analysis will be evaluated. However, the analysis of a full suite of metals and phenolic compounds would require additional analytical methods not proposed based on the screening analysis presented in the work plan. Although phenolic compounds are included in the standard Method 8270 together with PAHs, the Johnson Lake investigation will use a modified method that is specific to alkylated PAHs and does not include phenolic compounds. Also, barium inherently has a very low potential for toxicity, a fact which will be further clarified in the revised work plan. Concerns regarding the small number of samples used for screening purposes should be adequately

addressed by including soil data in the screening process (see response to Comment 3). Thus, the evaluation of additional constituents will be limited to pesticides and the additional chemicals identified as constituents of interest based on the screening analysis of soil data (see response to Comment 3).

See the response to Comment 3 regarding petroleum hydrocarbons.

7. The work plan should include a conceptual site model that identifies potential sources, exposure pathways and receptors. The conceptual model for ecological receptors should clearly identify those receptors selected as assessment entities, as well as the reason for their selection. The analyses that will be conducted should then be linked to the conceptual site model so the reason for conducting each test is clear (in terms of how it will help us interpret risk at the site). It is also important that the sequence for completing the identified evaluations be clarified. Certain evaluations (examples indicated in comments below) may not be necessary if particular exposure pathways are found not to pose an unacceptable risk.

In response to this comment, a conceptual site model for human health and ecological exposure pathways will be included in the revised work plan. The conceptual site model will include written descriptions, identifying human health exposure scenarios (i.e., on-site worker, trespasser, hypothetical recreator (wader), and hypothetical angler) and ecological receptors (e.g., great blue heron, mink), as well as the basis for their selection. Ecological assessment and measurement endpoints will be identified in the conceptual site model.

See the response to comment 15 (below) regarding sampling sequence.

8. The last sentence of Section 3.1.3 states that PAHs, cadmium, lead, and zinc do not biomagnify; however these compounds are included in the list of bioaccumulative compounds developed by U.S. EPA Region 10 for the Dredge Material Management Program. Their presence on this list indicates that they may be expected to accumulate in fish tissue at levels of concern to consumers of fish. This will likely be the exposure pathway that results in the lowest sediment screening level.

It is well established that fish rapidly metabolize PAHs. In our experience, even fish collected at oil spill sites contain virtually no PAHs. A recent article is attached describing the limited potential for adverse effects on fish consumers due to PAHs. Furthermore, the Dredged Material Evaluation Framework for the Lower Columbia River Management Area (November 1998), a multi-agency plan endorsed by USEPA Region 10 and the Oregon DEQ, establishes bioaccumulation trigger concentrations for fluoranthene and benzo(a)pyrene in sediment that are higher than the maximum *total* PAH concentration measured in Johnson Lake. Bioaccumulation trigger concentrations are not established for any other PAHs, indicating they are not of concern with respect to bioaccumulation. Similarly, bioaccumulation trigger concentrations are not identified for cadmium, lead, or zinc. Indeed, zinc is an essential micronutrient that is biologically regulated (Schmidt-Nielsen 1997), such that zinc concentrations in biota tissue are relatively constant over a wide range of sediment concentrations (Chapman, Allen et al. 1996; Newman 1998). The additional metals identified as chemicals of interest in response to Comment 3 also lack bioaccumulation trigger concentrations or are present in sediment and soil at concentrations below the trigger value. For these reasons, PAHs and metals will not be analyzed in biota tissue.

9. Further explanation of how the paired surface water and sediment chemistry data, referenced in the first paragraph of Section 3.2, will be collected and how they will be used to interpret sediment toxicity results should be provided.

This comment references an introductory overview paragraph. The paragraph will be revised by adding a parenthetical reference to the appropriate subsection containing further explanation. Section 3.2.3 will be clarified to indicate that sediment for both toxicity testing and chemical analyses will be collected as composite samples representing each of the sampling zones designated in Figure 3-2. See the response to Comment 12 (below) regarding surface water sampling.

10. Four different toxicity tests are identified in Section 3.2.1 and the work plan indicates that each test will be used to assess survival, growth and reproduction. The conceptual site model and associated discussion described in comment 7 should provide the basis for conducting these tests. Note that the surface water toxicity tests identified in the 3rd and 4th bullets are generally only warranted in situations where there is an on-going release directly to the surface water and may not provide information on risks associated with contaminated sediments.

As stated in the response to Comment 7, the revised work plan will describe the relationship between toxicity test endpoints and the conceptual site model. See the response to Comment 12 (below) regarding surface water testing. Note also that the proposed test endpoints (survival, growth, and/or reproduction) differ among test species in the work plan; not every test will evaluate the same endpoints as implied by the comment.

11. The second paragraph of Section 3.2.1 describes how bioassay test results will be evaluated, suggesting that the same interpretive criteria will be used for all 4 tests and all 3 endpoints. The work plan should provide interpretive criteria specific to each test and endpoint. Also, the work plan should indicate whether results will be compared to a control and/or reference sediment sample, or the process for deciding to use one or the other should be described. The percent difference in results indicating an effect will likely be different depending on whether a reference sediment or control sediment is used.

Interpretive criteria specific to each test and endpoint will be taken from the Dredged Material Evaluation Framework for the Lower Columbia River Management Area (November 1998). Use of a reference sediment is not proposed at this time; toxicity will be evaluated relative to the laboratory control sediment. However, in the event that significantly reduced survival or growth are observed but do not appear to be due to measured chemical or physical characteristics of the sediment, it may be necessary to conduct a comparative evaluation using a reference sediment. In that event, an appropriate reference area will be selected in consultation with the Oregon DEQ.

12. We question the value of collecting surface water samples as indicated in Section 3.2.2 as the detection limits will likely be too high to assess whether contaminants are present at levels of concern. Further discussion of the basis for conducting this sampling should be provided in the Conceptual Site Model and associated discussion. It is unlikely to be warranted unless there is on-going release of contaminants to the lake.

We agree that hydrophobic organic compounds are unlikely to be detected in surface water. Chemical analyses and toxicity testing of surface water will be removed from the work plan in

response to this comment. As a result, a "swimmer" scenario will not be included in the human health risk assessment. A "wader" scenario will address issues related to direct contact with sediments in Johnson Lake.

13. The detailed work plan should indicate the number of samples that will be collected for each type of analyses described, sample collection techniques, sample locations. A map should be provided that identifies sample locations. It will likely be useful to include some biased sampling in the vicinity of outfalls in addition to random samples representing the grids identified in Figure3-2. Note that, Kennedy-Jenks Consultants experienced difficulty collecting sediment samples due to a "hard pan" layer they encountered during previous sediment sampling at this site. It may be useful to review their observations prior to conducting field work and be prepared with appropriate sample collection tools to ensure that this issue will not prevent collection of sediment samples during this investigation.

As stated in response to Comment 20 (see below), a Sampling and Analysis Plan (SAP) will be submitted and will contain the details of all sampling techniques, including any pertinent sampling maps. The number of sediment samples and their locations will be clarified as described in response to Comment 9. Because sediment sample collection will entail composite sampling to represent each sampling zone (including zones in the vicinity of all outfalls), additional, biased sampling is not needed. As the term implies, biased sampling would skew the data set rather than providing representative data for risk assessment purposes. The inclusion of soil data in the screening process to identify chemicals of interest (see response to Comment 3) is highly conservative and ensures that chemicals of interest will not be overlooked during the representative sampling proposed for Johnson Lake.

Dense clay prevented Kennedy-Jenks Consultants from collecting sediment at depths greater than 6 inches below the sediment surface. The surface sediment reportedly consisted of loose sand or silt, which was readily sampled. The work plan will be clarified to state that sediment sampling will target surface sediment (top 6 inches), because the subsurface clay is not of interest as ecological habitat.

14. As we indicated in previous comments (letters dated 12/20/02 and 2/10/03), the PCB homologue analysis described in the first bullet of Section 3.2.2 will not provide data that will allow for determination of PCB toxic equivalency. A PCB congener analysis should be conducted on the 18 NOAA status and trends core congeners (8, 18, 28, 44, 52, 66, 101, 105, 118, 128, 138, 153, 170, 180, 187, 195, 206, 209) using currently available EPA Method 1668 (Revision A) HRGC/HRMS techniques. Note that it will likely be most efficient to conduct this analysis on tissue samples to determine if a complete risk pathway exists. If PCB congeners are not detected at levels of concern in fish tissue and bioassay results do not indicate toxicity, further sediment characterization will not be necessary.

The Oregon DEQ's published policy on toxicity equivalency factors (TEFs) states that TEFs **may** be used in PCB congener-specific risk assessments, not that such analyses are mandated. The policy also acknowledges that the use of TEFs is an evolving science. Although the science may one day advance to the point where PCB congener data are useful, the current state of the science does not permit a meaningful evaluation of risks associated with PCB congeners, for several reasons, as summarized below:

- Extrapolation of dosing regimes is a significant source of uncertainty in the derivation and application of TEFs. For example, it is assumed that, for fish and birds, responses to single very high bolus doses of PCB congeners via egg injection (i.e., the dosing regime used in the majority of studies upon which TEFs are based) accurately predict responses to chronic low-level dietary and/or maternally transferred exposures as would be experienced in the wild (i.e., the dosing regime relevant to risk assessment).

- Extrapolation among endpoints is another important source of uncertainty. TEFs generally reflect variations in biochemical endpoints (e.g. induction of various enzymes in the liver) and limited systemic health effects among different congeners. Such biochemical responses are not adverse health effects in and of themselves; rather, they are adaptive mechanisms that reflect the body's ability to defend itself. Those studies that yield TEFs based on health effects rather than biochemical responses generally focus on responses to acute, often lethal, doses (i.e., LD50s). When the TEF approach is used for risk assessment purposes, there is the implicit assumption that responses observed in biochemical or lethality endpoints parallel responses in critical health effects, such as effects on reproduction, cancer, and long-term mortality. In fact, the slopes of the dose-response curves for multiple endpoints are expected to differ considerably (De Vito, Ma et al. 1994).

- Recent TEF schemes (i.e., Van den Berg, Birnbaum et al. 1998) reflect a combination of rounding, averaging, and scientific judgment to reconcile sometimes very large variations in responses between studies. TEFs derived for a single congener from different studies, using various endpoints and species, may vary by three or more orders of magnitude. For example TEFs for PCB 189 (3,3',4,4',5,5'-hexachlorobiphenyl) range from 0.00059 to 1.1 (Safe 1994) and those for PCB 77 (3,3,',4,4'-tetrachlorobiphenyl) range from 0.000007 to 0.13 (Safe 1997/1998).

- The relative toxicity of PCB 77 relative to TCDD is highly uncertain for birds, with TEFs in multiple studies ranging from <0.0003 to 0.15 (Van den Berg, Birnbaum et al. 1998). Although Van den Berg, Birnbaum et al. (1998) attribute this variation to differences among bird species, they note elsewhere that "impurity and background contamination (e.g., PCB 126) is an identified problem with this congener." Thus, the selected TEF of 0.05 is essentially arbitrary and may overestimate the toxicity of PCB 77 to birds. According to the National Research Council (NRC 2001), PCB 77 (3,3',4,4'-tetrachlorobiphenyl) accounts for the highest proportion of the TEQ in environmental samples, when calculated using avian TEFs.

- The TEF approach assumes that the contributions of individual components of a mixture to that mixture's overall toxicity are essentially additive (Safe 1998). However, Safe (1994), an originator of the TEF approach, warns "analysis of the results of laboratory animal and wildlife studies suggests that the predictive value of TEQs for PCBs may be both species- and response-dependent because both additive and nonadditive (antagonistic) interactions have been observed with PCB mixtures. In the latter case, the TEF approach would significantly overestimate the toxicity of a PCB mixture." For this reason, Safe (1994) asserts that application of the TEF approach for the risk assessment of PCBs must be used with considerable caution.

- Studies have been undertaken with the objective of addressing this uncertainty by testing the correlation between predicted TEQ toxicity and actual health effects. Safe (1994) demonstrated that, while the TEF approach was useful for estimating "dioxin-like activity" in rats, it significantly overestimated the health effects of PCBs in mice. In salmon, TEQs were not correlated with mortality of eggs and fry from different clutches (Williams, Giesy et al. 1992). Safe (1994) also showed a lack of correspondence between TEQs and cancer potency for Aroclor 1260 and dioxin. Hence, the reliability of the TEF approach is highly dependent on the species and endpoint of interest. Even within the same species, there may be response-specific differences in the reliability of the TEF approach (Safe 1994).

- Numerous published studies have documented the congener composition of PCB Aroclors, and this information can be used to evaluate whether the TEF approach accurately predicts the results of wildlife toxicity studies. As one example, the No Observed Adverse Effect Level (NOAEL) identified for the Great Lakes Initiative based exposure of pheasants to Aroclor 1254 can be expressed as an exposure of 0.021 micrograms TEQ per kilogram body weight per day (µg/kg-d). For comparison, the Great Lakes Initiative identified a NOAEL for pheasants exposed to TCDD of 0.0014 µg/kg-d. Thus, the TEF approach overestimates the toxicity of Aroclor 1254 to pheasants by more than an order of magnitude.

- The USEPA has indicated that because relatively few PCB congeners are "dioxin-like," dioxin toxic equivalence can only account for part of the PCB mixture's toxicity. They further state that a mixture-based approach to cancer risk assessment is necessary to avoid underestimating PCB cancer risks (USEPA 1996). This suggests that PCB cancer risks calculated using a congener approach should be lower than those calculated using the total PCB approach, but often they are higher. The likely reasons for this discrepancy relate to the large degree of uncertainty associated with TCDD toxicity and the uncertainty and conservative bias inherent in the TEF approach itself.

- The basic premise of the TEF approach is that the toxicity of PCB congeners can be indexed to that of TCDD. However, the toxicity of TCDD is itself highly uncertain. Although the USEPA has been working on a reassessment of dioxin toxicity for many years, a revised TCDD CSF has not yet been published on IRIS. Alternative, published CSF values for TCDD range over more than three orders of magnitude (Keenan, Paustenbach et al. 1991; Kimbrough, Falk et al. 1984; USEPA 1985, 1988, 1997, 2000; USFDA 1994). Such extreme uncertainty will result in risk estimates that do not provide meaningful information.

- Similarly, the toxicity of TCDD to wildlife has not been studied as extensively as PCBs. As a result, more uncertainty and extrapolation factors must be applied to derive toxicity reference values for wildlife exposed to TCDD.

As stated succinctly by Giesy and Kannan (1998), "uncertainties of a few orders of magnitude between species and for specific end points are a major drawback in using the TEF approach in risk assessment." Uncertainty in the TEF approach is compounded by the uncertainty in the available toxicity values for dioxin, which spans several orders of magnitude. Hence, risk estimates based on

the TEF approach may deviate from actual risks by as much as six orders of magnitude. Such highly uncertain risk estimates have no utility for risk management purposes. Although there is also uncertainty in estimates of risks posed by total PCBs using the homologue approach, that uncertainty does not approach the uncertainty associated with the TEF approach.

15. Again it will be important to describe the sequence of planned testing and how test results will be used to assess complete exposure pathways identified in the conceptual site model, in determining the need for some of the testing described on page 14 of the draft work plan. For example, bioassay and tissue sampling results may address the bioavailability of contaminants such as AVS and pore water selenium analyses are not necessary.

Samples for sediment chemistry, sediment toxicity testing, and biota tissue chemistry analyses will be collected concurrently. Making chemical analyses contingent on toxicity test results is impractical, because the identification of constituents contributing to toxicity (if any) will need to be performed using chemistry data that are spatially and temporally paired with the toxicity data. Delaying the AVS and porewater analyses until toxicity test results are available is not an option, due to holding time constraints. See the response to Comment 7 regarding the conceptual site model.

16. We question whether it will be practical to collect sufficient tissue samples to adequately represent all the categories of biota identified in Section 3.2.4. The conceptual site model and associated discussion should clearly indicate the basis for sampling each category of biota and how the results will be used to estimate risk. It may only be necessary to collect samples for representative species of carnivorous, omnivorous, and bottom-feeding fish and focus on larger fish in thee categories, since they are more likely to accumulate higher concentrations of contaminants. The work plan should describe sample collection procedures and numbers of samples that will be collected.

The selection of biota types for analysis will be clarified by specifying the wildlife receptors of interest in the conceptual site model. Rather than basing the investigation on sampling convenience, biota types were selected to represent the types of fish and other species consumed by wildlife and humans. ARCADIS has experience successfully collecting these types of biota for chemical analysis. Because different species accumulate PCBs and other chemicals to different extents, the biota sampling program described in the work plan provides a more realistic basis for assessing risks than a program focusing only on large fish.

The number of biota samples to be collected is already described in Section 3.2.4 of the work plan. Sampling methods will be detailed in the SAP.

17. A beneficial land use evaluation must be completed to support the exposure scenarios described in Section 4.1. This would provide the foundation for eliminating certain potential land uses such as residential use. Also, the trespasser scenario should more accurately be described as the site visitor scenario with recreational site use described, since much of Johnson Lake will be open to the public. The likelihood that people will swim or wade in Johnson Lake should be addressed in this discussion as well as in the conceptual site model described in comment 7.

In response to this comment, a land use evaluation will be completed as part of the Risk Assessment Work Plan. The evaluation will be conducted in accordance with the *Final Guidance - Consideration of Land Use in Environmental Remedial Actions, Oregon Department of Environmental Quality, Waste Management and Cleanup Division, 1998.* At the completion of this evaluation, a land use designation will be made and complete exposure scenarios will be finalized. However, based on current zoning designations within the locality of the facility (i.e., mixed industrial and undeveloped; see attached Figure 3), exposure scenarios are likely to include those listed in response to Comment 7.

The trespasser scenario will be retained to account for potential exposure to soil on the Owens-Brockway property. A hypothetical recreator (wader) scenario will be added to account for exposure to sediment in a possible future public recreation area; this scenario encompasses the "site visitor" referenced in this comment. As described in response to Comment 12, swimming will not be evaluated, because surface water will not be analyzed.

18. Note that fish ingestion rates, referenced in the description of the hypothetical angler scenario, page 18, should reflect the Slough-specific data developed in the Technical Memorandum on the Results of the 1995 Fish Consumption and Recreational Use Surveys (Adolfson and Associates, Inc, 12/18/95).

A hypothetical angler scenario will be evaluated for the site, although the current posted fishing ban is presumed to prevent fish consumption at present. Information found in, but not limited to, the *Technical Memorandum on the Results of the 1995 Fish Consumption and Recreational Use Surveys (Adolfson and Associates, Inc, 12/18/95)*, will be used for evaluation purposes following a thorough review of the science and methodology used in the study. We retain the option to present an alternative approach, as appropriate, after reviewing the study

19. The detailed work plan should include a table of the exposure and toxicity factors that will be used in the risk assessment. The associated equations and processes that will be used to calculate risk should also be clearly outlined.

In response to this comment, a Risk Assessment Work Plan will be submitted. This plan will describe exposure and toxicity factors, equations, and evaluation processes as appropriate. However, risk assessment is an iterative process. Conservative, simplifying assumptions are used in the interest of efficiency, if they are sufficient to answer all relevant questions. These default assumptions are replaced with more detailed, realistic, or site-specific information as needed. Thus, it is not possible to identify at this time all of the information that will ultimately be used to estimate risks. The Risk Assessment Work Plan will therefore be submitted after the proposed sampling and a preliminary review of the analytical results have been completed.

20. The detailed work plan should include a sampling and analysis plan (SAP) including tables with analytical methods and associated detection limits, a health and safety plan (HASP), and a schedule for conducting the proposed work. The SAP and HASP previously submitted may be referenced as appropriate.

A SAP and QAPP specific to the proposed sampling will be submitted. The existing HASP for the site will be modified to account for hazards associated with working from a boat.

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REPORT SUMMARY

Management of contaminated sediments poses a complicated, expensive problem, exacerbated by the fact that contamination is often the cumulative result of inputs from numerous sources over many years. Contaminated sediments are of particular concern among owners of former manufactured gas plant sites. This Sediment Guidance Compendium is designed to help site owners faced with remedial challenges determine how to manage contaminated sediments. The compendium discusses the rationale behind risk assessments and provides a complete review of the Natural Resource Damage Assessment guidelines.

Background

Polycyclic aromatic hydrocarbons (PAHs) constitute the primary contaminants of concern in sediments exposed to materials from former manufactured gas plant (MGP) sites. Many former MGP sites are located near or adjacent to rivers, streams, impoundments, lakes, or coastal environments. In the 40-50 years that have passed since these plants were operational, MGP residuals and waste products, including PAHs, may have been degraded, transported, dissipated, or buried by sediments. There is also the potential for MGP residuals to enter surface water through venting of contaminated groundwater, seeps, or runoff from impacted surface soils. Regulatory authorities are now moving forward to address these surface water and sediment issues. Responsible parties holding liability associated with former MGP plants need to develop a clear understanding of the various issues as well as assessment endpoints.

Objective

To increase understanding of issues pertaining to the sampling and cleanup of contaminated sediments.

Approach

This compendium evolved from discussions at the Sediments Seminar II, held October 10-12, 2000, on St. Simon's Island, Georgia. EPRI's MGP Site Management Target members, EPRI project managers, and scientific consultants from across the United States gathered to study the issues pertaining to sediment management at former MGP sites. The compendium presents seven general topics raised by MGP site owners at the seminar and prioritized by EPRI. Each of the authors addressing the Sediments Seminar II issues summarized their perspectives in individual chapters of this compendium.

Results

The first chapter of this compendium discusses PAH chemistry of MGP tar and source identification in sediment. This chapter presents the history of MGPs and the chemistry of MGP waste to explain how MGP signatures differ from other PAHs of multiple origin.

The second chapter addresses the Equilibrium Partitioning Sediment Guidelines and their application to MGP sites.

Chapter three emphasizes the importance of assessing sediments for ecological risk at MGP sites because waste materials may have continued to contaminate sediments long after MGP operations ceased.

The fourth chapter offers a strategic approach to sediment sampling and characterization. The goal of this chapter is to assist in the development of effective field sampling and analyses plans to determine the extent of chemical contaminants in sediments.

Chapter five explores the relationship of contaminant bioavailability to the observed toxicity of aquatic organisms, with particular emphasis on potential MGP contaminants of concern.

Chapter six examines how PAHs in aquatic systems affect fish and other organisms. The most promising approach for assessing risks to fish is a model based on the bioavailability of PAHs in sediment—using fish as a key evaluator for understanding PAH concentration risk in sediments.

Chapter seven reviews the Natural Resource Damage Assessment (NRDA) guidelines—a Federal process for assessing injury to the environment due to uncontrolled releases of hazardous substances. This chapter describes approaches for controlling NRDA costs and integrating NRDA issues into the management of environmental liability at former MGP sites.

EPRI Perspective

This Sediment Guidance Compendium provides a comprehensive look at key issues pertaining to the management, cleanup, and removal of contaminated sediments at former MGP sites. Future work should focus on fingerprinting PAHs and identifying their sources in sediments. In addition, it will be important to understand the toxicological effects of PAHs in aquatic organisms and finfish in light of potential risk assessment findings and the current sediment cleanup guidance.

Keywords

MGP sites
Remediation
Sediments
Equilibrium Partitioning Sediment Guidelines
Natural Resource Damage Assessment

ABSTRACT

Polycyclic aromatic hydrocarbons (PAHs) constitute the primary contaminants of concern in sediments exposed to materials from former manufactured gas plant (MGP) sites. Site owners may leverage their sediment management decisions by conducting risk assessments and collecting sediment and water samples for delineation of contaminants. Such toxicity testing complies with state regulations and promotes scientific understanding of the PAHs biologically available in sediment. Proper assessment of contaminated sediments is necessary if a Natural Resource Damage Assessment (NRDA) is considered as part of the remedy for a contaminated site.

This Sediment Guidance Compendium provides a comprehensive look at key issues pertaining to the management, cleanup, and removal of contaminated sediments at former MGP sites. The seven chapters in this compendium address 1) PAH chemistry of MGP tar and source identification in sediment, 2) Equilibrium Partitioning Sediment Guidelines, 3) assessment of the ecological risks of PAH-contaminated sediments, 4) a strategic approach to sediment sampling and characterization, 5) toxicity testing and bioavailability of contaminants in sediments, 6) ecological assessment of PAHs in fish, and 7) a review of NRDA guidelines.

6
ECOLOGICAL ASSESSMENT OF PAHS IN FISH

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One of the most important classes of chemicals associated with former manufactured gas plant (MGP) sites, and the focus of this compendium, is polycyclic aromatic hydrocarbons (PAHs). Many former MGP sites are located near or adjacent to rivers, streams, impoundments, lakes, or coastal environments. In the 40-50 years that have passed since these plants were operational, MGP residuals and waste products may have been degraded, transported, dissipated, or buried by sediments over time. There is also the potential for MGP residuals to be currently entering surface water through venting of contaminated groundwater, seeps, or runoff from impacted surface soils. Regulatory authorities are moving forward to address these surface water and sediment issues, and responsible parties holding liability associated with former MGP plants need to develop a clear understanding of the assessment endpoints and the advantages and disadvantages of various measures of exposure and effects.

The presence of PAHs in aquatic systems can trigger the need to assess potential ecological impacts. In particular, ecological risk assessment can be used to determine whether clean-up actions are necessary, and if so, to identify appropriate clean-up levels. Natural resource damage claims have also led to assessments of PAH-related effects on aquatic ecosystems. Typically, these assessments involve the evaluation of potential effects on sediment-dwelling (benthic) invertebrates, fish, and aquatic-feeding wildlife (birds and mammals). Because PAHs are only slightly water soluble, surface water quality issues associated with PAHs tend to be minimal and restricted to lighter compounds, such as the 2-ring naphthalene family. PAHs tend to accumulate in sediment, and sediment toxicity issues have been the focus of much research in recent years. The toxicity and bioaccumulation of PAHs is relatively well understood for benthic invertebrates (Di Toro et al., 2000; Tracey and Hansen, 1996; USEPA, 2000a). McGrath et al. (this volume) address sediment issues related to PAHs and describe the state of the art approach to account for the bioavailability of individual PAHs and to develop levels protective of benthic invertebrates. The assessment of fish exposed to PAHs is much more controversial.

Because fish metabolize PAHs, it is very difficult to even establish exposure, much less injury, to highly mobile fishery resources. Metabolism of PAHs by fish diminishes PAH-related risks to fish-eating (piscivorous) wildlife, but it is not entirely effective in protecting the fish themselves from toxicity. Some PAHs are carcinogenic, and high rates of tumors and lesions have been documented in fish populations living near active sources of PAHs, including contaminated sediments and oil spills. In fact, PAHs are suspected as an important cause of most "cancer clusters" observed in fish populations (Baumann, 1998). Much of the research to advance the understanding of the effects of PAHs on fish has been conducted in conjunction with natural

resource damage assessments, related to major oil spills or contaminated sediments. Also, as biological surveys have become more widely used for water quality monitoring purposes, adverse effects on fish have increasingly come to light. PAHs in fish have become an active topic of research in the technical literature and are likely to receive increasing attention from regulatory agencies.

This chapter provides a synthesis of available information on PAHs in fish from a practical risk assessment perspective. Regarding the assessment of PAH-related risks to fish, we discuss (1) exposure characterization, focusing on frequently used exposure measurement methods; (2) effects characterization, focusing on histopathology (i.e., manifestations of disease in fish tissues); and (3) the relationship between exposure and effects, focusing on the ability to predict effects based on exposure data. Regarding piscivorous wildlife, we evaluate the ability to predict PAH concentrations in fish consumed as prey, and the relative risk of effects on wildlife versus aquatic life. The implications of this information for MGP sites are also discussed.

6.1 Fish Exposure to PAHs

PAHs are unlike many other common environmental contaminants in that their effects on fish are not readily assessed based on either tissue residues or surface water concentrations. Tissue residue assessment of fish does not provide an accurate indicator of chronic exposure to PAHs, because fish efficiently metabolize PAHs. Although tissue residues can be used to evaluate acute toxicity due to narcosis (e.g., during an oil spill), long-term exposures such as those associated with contaminated sediments cannot be accurately measured using this approach. Additionally, significant PAH exposures may occur when PAH concentrations in surface water are below detection limits, due to contact with sediment porewater and ingestion of PAH-containing invertebrate prey or sediment.

Multiple approaches are available to characterize fish exposure to sediment-associated PAHs. Four frequently applied methods are discussed below, including analyses of sediment, metabolic enzymes, bile metabolites, and DNA adducts. A brief discussion of exposure pathways and PAH metabolism is provided as background information.

6.1.1 Exposure Pathways

As PAHs enter aquatic systems, they tend to partition quickly from water to organic material, due to their low solubility and highly lipophilic nature. Although a fraction of PAHs may initially remain in the dissolved phase, dissolved PAHs will degrade rapidly via photooxidation and other weathering processes. The fate of a majority of the aquatic aromatic hydrocarbons, then, is adsorption to sediment (including suspended solids and bottom sediment) and biota (Neff, 1979; Eisler, 1987). Once PAH compounds are adsorbed, their stability and resistance to reaction increase (Prahl and Carpenter, 1983; Van Hoof et al., 2001). Higher molecular weight PAHs, which exhibit low mobility because of their larger size, concentrate in sediment near point sources (Baumann, 1998); their distribution decreases logarithmically with the distance from the source (Neff, 1985). However, the distribution of PAHs in sediment is heterogeneous and also depends on factors other than source location, such as sediment particle size (Suffet et al., 1994). Adsorption of PAHs directly from surface water to fish and benthic species results in higher

PAH concentrations in these organisms than in the ambient surface water; however, most aquatic PAHs are associated with sediments, which may contain PAH concentrations several orders of magnitude higher than surface water (Malins and Ostrander, 1991; McElroy et al., 1989).

Fish are exposed to PAHs originating in contaminated sediment primarily via direct contact with the sediment, particularly sediment porewater, and, to a lesser extent, ingestion of sediment-dwelling organisms or sediment. Desorption of PAH compounds from the sediment to interstitial water and/or gut epithelia provides an immediate source of PAHs to benthic organisms, including fish (Stein et al., 1990). Adsorption of PAHs into fish circulatory systems and tissue from interstitial water associated with sediments and surface water can occur across the gills or, to a lesser extent, the skin. Water ventilation is the significant pathway for benthic species due to the large quantities of water that must pass through the gills and the rapidity of equilibrium between body tissue and the medium (de Voogt et al., 1991; USEPA, 2000b). Uptake of PAHs from ingestion of sediment and/or benthos is an indirect route and less significant source of PAH contamination. In an experiment with gizzard shad, Kolok et al. (1996) showed that water ventilation, not sediment ingestion, was the primary exposure route for benzo(a)pyrene (BaP). When fish feed, particulates other than food are blown out of the opercula before swallowing, which will tend to minimize sediment ingestion. For fish species with minimal direct contact with sediment porewater, the relative importance of exposure via ingestion of prey or sediment increases.

Bioavailability and uptake of PAH compounds depends on a myriad of biotic and abiotic factors, including lipid content (of fish), organic carbon content (of sediment and water), physico-chemical properties of different sediments and PAHs (which may be influenced by temperature and salinity), and biological characteristics of different fish species. The lipophilic nature of PAHs allows for easy penetration of biological organisms. As the lipid content of biological membranes increases, the uptake of PAHs increases (Tuvikene, 1995). Increases in dissolved and total organic carbon decrease the bioavailability of PAHs in water and sediment (Kukkonen and Oikari, 1991; Tracey and Hansen, 1996; USEPA, 2000a). The octanol/water partition coefficient (K_{ow}) of individual PAHs describes their tendency to partition to organic matter; PAH compounds with a log K_{ow} between 3 and 6 are taken up most readily from an aqueous environment. At higher log K_{ow} values, low solubility and compound size tend to inhibit biological uptake. Ambient temperature and salinity will also affect the uptake of PAHs into fish tissue (Collier et al., 1978; Seubert and Kennedy, 2000), due in part to their effect on PAH solubility. Specifically, solubility increases with increased temperature and decreases with increased salinity (Neff, 1979). Temperature also affects cell membrane permeability, although fish can regulate this effect physiologically, at least to some extent (Hazel, 1993).

The behavior of the fish themselves can also greatly affect the extent of PAH exposure. Bottom dwelling fish are exposed to sediment simply because of their habitat preference. In addition, many benthic fish species stir up the sediment during regular movement or feeding activities, resuspending PAH material into the interstitial and surface water (e.g., gizzard shad; Kolok et al., 1996). Age, gender, and life stage also determine uptake of PAHs. For example, Lemke and Kennedy (1997) determined that the uptake of BaP significantly increased during the parr-smolt transformation in Coho salmon. Also, the outer membrane (chorion) surrounding developing fish embryos tends to reduce chemical exposures during the embryonic life stage (Villalobos et al., 2000).

6.1.2 Metabolism, Accumulation, and Excretion of PAHs

Once adsorbed into fish, PAHs are immediately distributed throughout the body via the circulatory system, with more compounds delivered to organs with a greater localized blood flow, for example, the kidney and liver. PAHs may be metabolized, stored and/or excreted, with the ultimate goals of reducing toxicity and discharging the xenobiotic substance from the body. Numerous factors influence the fate of PAH compounds within fish, among which are the chemical structure of the parent compound and metabolites, cellular uptake mechanisms, species, sex, and physical condition.

Metabolism and excretion of PAHs is more efficient in fish than invertebrates (Meador et al., 1995). As a result, PAH concentrations in fish are much lower than in invertebrates, and PAHs do not biomagnify through the food web. It is important to note, however, than PAH metabolism in fish is not instantaneous, and an equilibrium will be reached between PAH uptake and metabolism that can result in the presence of detectable parent PAHs in tissue, particularly the more slowly eliminated low molecular weight PAHs (Meador et al., 1995). Concentrations of PAHs in fish tissue can reach equilibrium with the aqueous environment within 24 hours (Law et al., 1991). Although metabolism in invertebrate species is less significant than in vertebrates, PAH metabolites (including deleterious metabolic intermediates) are also formed in invertebrates and have been shown to be available to vertebrates through ingestion (McElroy and Elskus, 1998).

From the circulatory system, a majority of the lower molecular weight PAHs are depurated directly from the body through the gills and, to a lesser extent, the skin of fish (Varanasi et al., 1978). The higher molecular weight PAH compounds are metabolized and can undergo extensive biotransformation. In fish, metabolism occurs primarily in the liver, although several extrahepatic tissues (e.g., kidney, gut, and gills) have been shown to be secondary sites for PAH metabolism (Buhler and Williams, 1989). The intestinal system may play a significant role in the dietary uptake and biotransformation of PAHs, often limiting the quantity of metabolic intermediates distributed to the rest of the fish tissues (McElroy and Elskus, 1998; Sandvik et al., 1998). In the liver, PAHs generally undergo three types of chemical reactions: oxidation, reduction and electrophilic substitution, as discussed below.

There are two stages associated with PAH metabolism, Phase I and Phase II. Phase I involves oxidation, reduction or hydrolyzation of the parent compound to form more reactive polar intermediates. During Phase II, the polar intermediates from Phase I are conjugated to polar endogenous co-factors (i.e., constituents already occurring in the cell) to facilitate excretion. The generalized PAH metabolic process is illustrated in Figure 6-1.

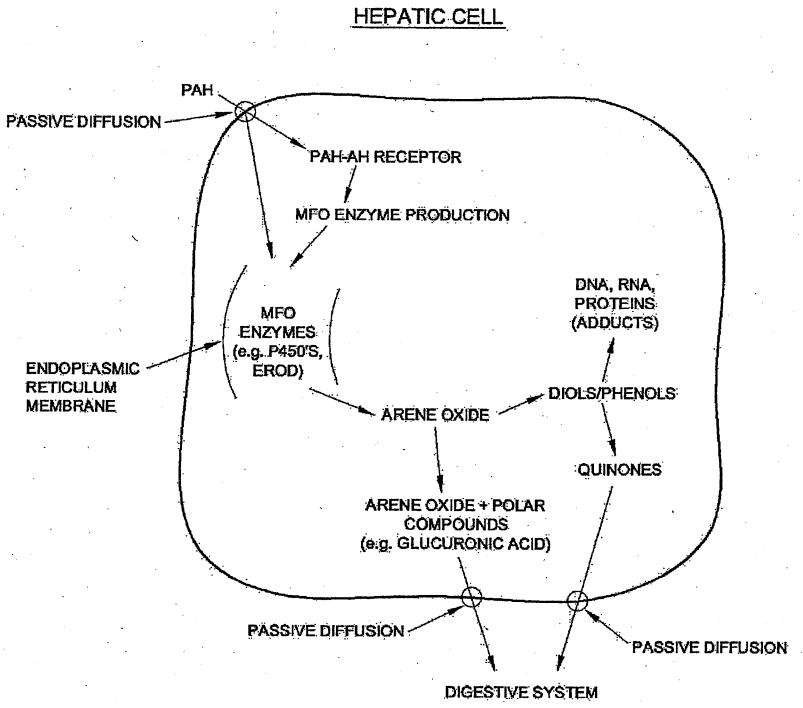


Figure 6-1
Generalized Metabolism of PAHs

During Phase I metabolism, PAHs enter the hepatic cell through passive diffusion and become available for intracellular metabolism via constitutive enzymes of the mixed-function oxidase (MFO) system. This system consists of cellular enzymes, i.e. cytochrome P-450 (P450), NADPH-cytochrome P-450 reductase, flavoprotein monooxygenases, and phospholipids of the endoplasmic reticulum (a cellular organelle), that function to oxidize parent PAH compounds (Buhler and Williams, 1989). Cytochrome P-450 is a family of enzymes concentrated in the membranes of endoplasmic reticulum and is believed to be primarily responsible for PAH metabolism. This group of enzymes in fish is particularly sensitive to xenobiotic (e.g. PAH) chemical invasion (Buhler and Williams, 1989) and can be induced to produce specific forms of cytochrome P-450s via activation of the cytosolic Ah-receptor (a protein that regulates synthesis of xenobiotic-metabolizing enzymes). The activated receptor enters the cell nucleus and enhances transcription of additional metabolic proteins (Klaassen, 1996). One subfamily of P450 that is induced, cytochrome P-450 1A (CYP1A), has been purified from several fish species (e.g. Stegeman, 1989; Williams and Buhler, 1984). CYP1A induction, in turn, stimulates the production of cytochrome P-450 1A1 (CYP1A1). This P450 utilizes the enzymes aryl-hydrocarbon-hydroxylase (AHH), ethoxyresorufin-O-deethylase (EROD), and 7-ethoxycoumarin-O-deethylase (ECOD) to oxidize the PAH compound and create an arene oxide (i.e. epoxide). For PAH compounds that contain a bay region, Phase I metabolism in fish tends to favor bay-region oxidation (Varanasi et al., 1989a) which may make epoxides more chemically reactive (Buhler and Williams, 1989; Klaassen, 1996; Stegeman et al., 1984). Arene oxides are unstable intermediates and can react further to become dihydrodihydroxy compounds (diols) or phenols and ultimately quinones (Stein et al., 1990; Varanasi et al., 1989b). Some

diols and phenols may undergo further reactions and bind to cellular macromolecules such as DNA, RNA, and protein (adduct formation) (Maccubbin, 1994).

Arene oxides may also enter into Phase II of PAH metabolism. Phase II proceeds in the cytoplasm to combine the polar intermediate from Phase I to a polar cellular component, for example, glucuronic acid, glutathione, sulfate, or an amino acid, via conjugating enzymes (Foureman, 1989). The end result is the formation of a highly water-soluble, excretable compound. If Phase I PAH metabolism proceeds faster than Phase II, there may be an increase in toxic metabolites within fish tissue.

Many different pathways, metabolic intermediates, and end products exist for PAH compounds depending, in part, on the parent compound and the species affected. Carcinogenic PAHs are typically a small portion of total PAHs in the environment but have been extensively studied, particularly BaP. A chemical inducer is not needed for BaP to become carcinogenic; therefore it is considered to be a complete carcinogen (Grady et al., 1992). During Phase I of BaP metabolism, BaP-7,8-diol is produced which undergoes further oxidation reactions to become BaP-7,8-dihydrodiol-9,10-epoxide (BaPDE), the suspected carcinogenic metabolite of BaP (Stein et al., 1990). BaPDE forms adducts with DNA which may initiate tumorigenesis.

Excretion and distribution of PAH metabolites within fish follow general patterns depending on the site of metabolism. PAH metabolites are carried from the liver into the gallbladder with the bile. The bile is excreted into the gut and ultimately eliminated with other waste byproducts through the digestive system. Bile typically contains the majority of the overall tissue burden of metabolites (e.g., Goddard et al., 1987). Bile may also be reabsorbed and reprocessed by the liver (i.e., enterohepatic recycling), potentially increasing the exposure of liver cells to highly reactive PAH metabolites. The kidney, a secondary organ for PAH metabolism, sends PAH intermediates into urine. There is also a passive, gradient-dependent loss of PAHs from the body via the gills as the fish moves from a source of exposure to an area of lesser concentration (Neff, 1985).

6.1.3 Sediment Analyses as a Measure of Fish Exposure

Sediment analyses are probably the simplest means of measuring fish exposures to PAHs. Because PAHs are hydrophobic and tend to adsorb to and persist in sediments, measured PAH concentrations in sediment provide an indication of relatively long-term environmental exposures. For benthic invertebrates, the concentrations of PAHs in sediments (when adjusted to account for factors affecting bioavailability) can be used as a surrogate for cumulative PAH exposures via all exposure pathways, including sediment and food ingestion and direct contact (McGrath et al., this volume). This approach has important limitations when applied to fish, however.

The amount of PAH taken up by a fish can be expected to vary significantly among different species exposed to the same PAH-contaminated sediment. Important factors that influence the extent of exposure include:

- *Contact with Sediment:* Bottom-dwelling fish species will experience greater PAH exposure due to increased frequency of sediment contact compared to species that inhabit the water column.
- *Dietary Preference:* Species that feed on benthic invertebrates will experience greater PAH exposures via ingestion than species that feed on other fish, due to differences in PAH metabolism between these types of prey items.
- *Mobility:* Fish species that inhabit a small territory will experience different exposures than species that are more mobile or are seasonally migratory.
- *Habitat Preference:* Species that utilize depositional pools or backwater habitat, where PAHs tend to accumulate in sediment, will most likely experience greater exposure than other species such as riffle specialists or pelagic (water column-dwelling) species.

For risk assessment purposes, therefore, it is especially important that exposure data based on sediment analyses be paired with effects data for fish species that are relevant to the site being assessed, based on habitat characteristics such as salinity, temperature, and size of water body. For example, an effects threshold identified for English sole (Horness et al., 1998) would not be directly applicable to a freshwater site or to marine areas outside this species' range.

Appropriate methods for collecting and analyzing sediment samples are well established. Considerations particular to the assessment of PAH-related risks to fish include sampling depth and the selection of target analytes. To most closely reflect ecological exposures, only surface sediment analyses should be considered. Typically, sampling depths of six inches or less are used for this purpose. There are several analytical methods to identify and quantify PAHs (Boehm et al., this volume). The most common methods are based on gas chromatography/mass spectrometry (GC/MS). EPA's SW-846 Method 8270 focuses on reporting the 16 priority pollutant PAHs. This method has been modified to include an extensive list of alklyated PAHs and to lower detection limits (Lauenstein, 1993). The toxicity and environmental behavior of individual PAHs can vary greatly; therefore, it is important to account for which PAHs are identified and quantified. Other methods are available that may quantify aromatic compounds based on flame ionization or fluorescence detection. These methods may only report total PAHs, not the results for individual compounds or isomeric groups (e.g., C2-phenanthrene).

When analyzing sediments for PAHs, it is important also to analyze for total organic carbon. Organic carbon is a critical factor controlling the bioavailability of PAHs in sediment. Normalizing PAH concentrations to the organic carbon content of the sediment greatly reduces the variability of the relationship between PAH exposure and ecological effects (Di Toro et al., 2000). Soot is another factor affecting bioavailability, because sorption of PAHs to soot reduces PAH partitioning to interstitial water (USEPA, 2000a). Soot concentrations can be quantified (Gustafsson et al., 1997), although the analysis is not routine. An alternative approach to account for the bioavailability of PAHs in sediment is to analyze the concentrations in sediment porewater (Ozretich et al., 1998); again, this approach has not been routinely used for site characterization purposes.

6.1.4 Mixed-Function Oxidase (MFO) Enzymes as a Measure of Fish Exposure

As described above, MFO enzymes can be activated during the metabolism of PAHs by fish. Cytochrome P-450 is a family of isoenzymes which is part of the MFO system of metabolism. These enzymes are located in the membranes of the endoplasmic reticulum and, therefore, appear in the microsomal fraction of cellular preparations. When initiated by exposure to xenobiotic organic chemicals, cytochrome P-450 initiates production of CYP1A which, in turn, increases concentrations of CYP1A1. CYP1A1 causes cellular elevations in EROD and AHH enzymes in order to oxidize PAH compounds.

Measurements of CYP1A, CYP1A1, EROD, or AHH may indicate activation of the general MFO system. Earlier laboratory and field studies of MFO induction measured cytochrome P-450 concentrations indirectly (method by Omura and Sato, 1964, cited in Burns, 1976). More recently, individual isoenzymes of cytochrome P-450 have been identified in scup and rainbow trout species allowing for greater specificity in measurements (Kleinow, 1987). Following microsome preparation from liver, CYP1A can be measured by enzyme-linked immunosorbent assays (ELISAs) (Jorgensen and Wolkers, 1999). Immunochemical localization methods can be used with paraffin-embedded tissue to quantify CYP1A expression (Myers et al., 1998a; Rice et al., 2000). CYP1A1 can be determined from immunoblot (Western blot) analysis of microsomal fractions (Van Veld et al., 1992) or, in the absence of microsomes, from autoradiograms scanned with a laser densitometer (Schoor et al., 1991). EROD can be measured spectrophotometrically (Goddard et al., 1987; Munkittrick et al., 1995; Van Veld et al., 1992; Willett et al., 1995) and fluorimetrically (Aas et al., 2000; Camus et al., 1998; Cormier et al., 2000a; Garrigues et al., 1989). A radiolabelled assay is the standard technique for measuring AHH in fish (e.g., Collier et al., 1995).

A primary advantage of utilizing a constituent of the MFO system as an indicator of PAH exposure is the relative ease of measurement, because most of the analytical techniques are well established. CYP1A may be influenced by certain PAH concentrations in fish tissue in a dose-dependent manner (Stegeman and Hahn, 1994), an imperative for use of the parameter as a measure of exposure. EROD measurement is simple, highly sensitive and highly specific for P4501A1 enzymes (Cormier et al., 2000a). However, Collier et al. (1995) found that the standard EROD assay, in comparison with AHH assays, provided more variable and less consistent results; they suggest the use of AHH assays when measuring induction of CYP1A.

A significant disadvantage of the MFO system as a PAH biomarker is the lack of specificity, in that MFO activity is strongly induced by other chemicals, most notably co-planar PCBs. Contaminated sediments usually contain a mixture of various chemicals, many having the potential to initiate the MFO system. Further, synergistic and antagonistic effects of the chemical mixture can alter induction of the system. Even if PAHs are the only source contaminant, the induction of the MFO system by PAHs in fish varies with temperature, season, acclimation, age, sex, and stress (Buhler and Williams, 1989; Devaux et al., 1998; Jorgensen and Wolkers, 1999; Kleinow et al., 1987; Rice et al., 2000; Vandermeulen and Mossman, 1996). A high degree of variability in EROD activity has also been observed in fish presumably receiving similar PAH exposures (e.g., Vandermeulan and Mossman, 1996). The large number of confounding factors and high variability limit the usefulness of MFO enzymes as a specific measure of exposure to PAHs.

6.1.5 Bile Metabolites as a Measure of Fish Exposure

Measurement of PAH metabolites in bile is more specific that the measurement of metabolic enzymes, which may be induced to metabolize chemicals other than PAHs. As described previously, PAH metabolites are carried from the liver into the gallbladder with the bile. Bile is subsequently excreted into the gut and ultimately eliminated by the digestive system. Many PAHs and their metabolites display strong and characteristic fluorescent properties, and this feature can be used to detect these compounds in bile. Various researchers have developed techniques to estimate the concentration of aromatic hydrocarbons in bile using fluorescence detection (Krahn et al., 1984; Lin et al., 1996). These methods generally consist of diluting the sample in ethanol or water followed by direct injection into a high performance liquid chromatograph (HPLC) equipped with a fluorescence detector. Other methods, such as fixed fluorescence, can also be used to detect and quantify bile metabolites (Lin et al., 1996).

The analysis of biliary metabolites is a screening method; individual metabolites are not identified or quantified. Because of the thousands of different possible PAHs and metabolites, more specific identification of individual PAHs is not practical. Different classes of bile metabolites (e.g., 2-, 3- and 4-ring PAHs) can be quantified, however. The bile is injected directly into the HPLC equipped with a C-18 reversed phase column. The eluent is monitored by flourescence detection at several excitation/emission wavelength pairs. Chromatograms are recorded at each wavelength pair, where parent compounds and metabolites fluoresce. For example, 2-ring PAHs (naphthalene and dibenzothiophenes) are monitored at 290/335 nm, 3-ring PAHs (phenanthrene) are monitored at 260/380 nm, and 4-ring PAHs (BaP) are monitored at 380/430 nm. Suspected metabolites eluting from the column within a specified retention time window (>7 minutes) are summed to yield total fluorescent aromatic hydrocarbons based on equivalents of known amounts of parent compounds. Results are expressed in terms of amount of bile metabolites per total amount of bile (e.g., 100 micrograms of naphthalene equivalents per gram of bile or ppm wet weight) or are normalized to the biliary protein content.

Laboratory studies have demonstrated that the presence of PAH metabolites in fish bile is statistically correlated with PAH exposure (Aas et al., 2000; Hellou and Upshall, 1995; Krahn et al., 1987, 1992; Stratham et al., 1976; van Schanke et al., 2000; Varanasi and Gmur, 1981; Varanasi et al., 1986;). Field studies have corroborated this relationship; fish sampled from contaminated areas contained higher concentrations of bile metabolites than fish sampled from less contaminated areas (Arcand-Hoy and Metcalfe, 1999; Krahn et al., 1984, 1986; McDonald et al., 1992; Pinkney et al., 2001; Willett et al., 1997;). Bile analyses confirmed exposure to PAHs resulting from oil spills (Krahn et al., 1992, 1993; McDonald et al., 1991) and exposure to contaminated sediments in urban environments (Johnston and Baumann, 1989; Malins et al., 1987; Pinkney et al., 2001; Stegeman et al., 1992; Willett et al., 1997). Cormier et al. (2000b) used bile metabolites to detect a longitudinal difference in exposure in the Ottawa River, OH.

Bile metabolites have been used to document exposure to PAHs in a variety of marine and freshwater environments. In general, naphthalene equivalents above 500 parts per million (ppm), phenanthrene equivalents above 50 ppm and BaP equivalents above 1 ppm are indicative of highly-polluted environments; and naphthalene equivalents between 300-500 ppm, phenanthrene equivalents between 10-50 ppm, and BaP equivalents between 0.2-1 ppm are indicative of moderately polluted environments (Sue McDonald, personal communication, as cited in Pinkney et al., 2001). Cormier et al. (2000a) developed exposure criteria values based

on hundreds of analyses from reference sites in Ohio, where a fish bile monitoring program has been implemented. The 95th percentile values for naphthalene and BaP equivalents are 60-80 µg/mg protein and 4 to 5 µg/mg protein, respectively. More than half of the non-reference (i.e., potentially contaminated) sites surveyed in Ohio exceeded these exposure criteria values (Cormier et al., 2000a).

Biliary metabolites have also been statistically correlated to the response of other biomarkers and adverse effects in laboratory and field studies. Van Schanke et al. (2001) reported that bile metabolites are a more sensitive exposure biomarker than EROD activity. Johnson et al. (1993) found a correlation between fluorescent aromatic hydrocarbons and hepatic P450 activity and inhibited ovarian development. The levels of PAH metabolites are dependent on P450 induction status (van Schanke, 2000). The levels of biliary metabolites were higher in fish that were pretreated with the coplanar PCB 126, indicating pre-exposure to Ah-active compounds can increase the biotransformation efficiency in fish. Responses in biliary metabolites have also been associated with liver lesions in fish, as described further below.

PAH metabolites detected in fish bile have been used to differentiate exposure to petrogenic and pyrogenic PAHs (Ariese et al., 1993; Hellou and Payne, 1987; Krahn, 1987; Lin et al., 1996), but this approach is problematic. Two-ring PAHs (expressed as naphthalene equivalents) are used as indicators of petrogenic PAHs, and 4-ring PAHs (expressed as BaP equivalents) are used as indicators of pyrogenic PAHs. This simple approach is inadequate to identify and differentiate multiple sources of PAHs in the environment. In fact, differences in the chromatographic pattern are found in fish exposed to the same source of PAHs (Krahn et al., 1993). Although Krahn et al. (1992) reported a statistical correlation between this screening technique and gas chromatography/mass spectrometry (GC/MS) results near an area impacted by an oil spill, it is not known what specific compounds contributed to the observed fluorescence. McDonald et al. (1992) reported chromatographic peaks where PAH metabolites were not expected based on retention times of known standards. It has been suggested that stress metabolites, associated with the collection of field organisms, can confound the HPLC pattern in bile metabolites (Sue McDonald, personal communication, as cited in Barber et al., 1995). The lack of specificity, the intra- and inter-species variability of the metabolic processes, and the confounding effects of stress metabolites preclude the ability to identify and differentiate sources of PAHs based on bile metabolite analyses.

The level of metabolites in the bile is, in addition to exposure to PAHs, a function of the amount of bile. Bile is produced to aid digestion; therefore, fish that are not feeding tend to have higher bile metabolite concentrations (Collier and Varanasi, 1991). Bile density is a function of feeding status, and is also influenced by size, age, and reproductive status (Collier and Varanasi, 1991; Leadly et al., 1999). Bile metabolite concentrations can be normalized to biliary protein levels to help account for these physiological factors (Collier and Varanasi, 1991; Cormier et al., 2000a). Another factor affecting bile metabolite levels is the exposure duration, where bile metabolite levels initially increase upon exposure to PAHs and then decline over time. French et al. (1996) observed attainment of steady-state concentrations after two weeks, but Camus et al. (1998) did not observe attainment of steady state over a three week exposure. Additionally, bile metabolite levels are not indicative of long-term exposures (Pinkney et al., 2001), such that exposure levels as measured by bile metabolites may not match observations of long-term effects, due to fish mobility or long-term changes in environmental conditions. Finally, factors that affect the level

of activity of metabolic enzymes (described above) will also affect levels of PAH metabolites in bile.

In summary, the analysis of PAH bile metabolites is a rapid, low-cost method to measure exposure of fish to PAHs in the environment. However, the inability to resolve specific metabolites and the confounding effects of fish condition and stress metabolites limit the development of a meaningful causal relationship between exposure and the occurrence of adverse effects. Bile metabolites are highly variable as a result of reproductive status, sex, age, diet, and mobility. Furthermore, the ability of fish to rapidly eliminate PAHs limits the ability to develop a quantitative pattern of exposure.

6.1.6 DNA Adducts as a Measure of Fish Exposure

A longer term measure of PAH exposure in fish is available through the analysis of DNA adduct levels. At the completion of Phase I metabolism of PAHs in fish, arene oxides are formed. These compounds are unstable and may undergo transformation into diols or phenols. Diols and phenols, through a series of reactions, have the ability to bind to cellular macromolecules, such as DNA, RNA and protein, thus forming an adduct. The covalent binding of constituents to genetic material typically defines an adduct, although Maccubbin (1994) has suggested that an adduct may be any modification to the structure of DNA resulting from chemical or physical agent exposure.

DNA adducts have been widely studied in liver tissue (for a review, see Maccubbin, 1994). Recently DNA adducts also have been isolated in extrahepatic tissue, including the intestine, indicating that PAH contamination during oral exposure may be a significant inducer of DNA adducts (Ericson et al., 1998b; Ericson et al., 2000). Carcinogen-damaged DNA (e.g., from adduct formation) in a dividing cell can cause inheritable DNA mutations, which, when promoted, can initiate tumor formation (Telli-Karakoc et al., 2001). DNA adducts in hematopoietic (blood-forming) tissues and blood may be associated with disruption of physiological functions such as immunosuppression and hematopoiesis (Faisal et al., 1998; Rose et al., 2000; 2001).

Measurement of adducts in fish is a relatively recent advancement. The dominant and most sensitive technique for assessing DNA adducts in fish is the ^{32}P-postlabelling assay (Aas et al., 2000; Maccubbin, 1994; Stein et al., 1990). This assay involves ^{32}P labeling of DNA followed by thin-layer chromatography and autoradiography to quantify ^{32}P decay (Reddy and Randerath, 1986). Numerous studies have used this measurement technique to identify and quantify DNA adducts in both laboratory and field-collected fish species (e.g., Ericson et al., 1998a,b; Lyons et al., 2000; Myers et al., 1998b; Noaksson et al., 1998; Ploch et al., 1998; Potter et al., 1994; Rose et al., 2000; Varanasi et al., 1989d; Willett et al., 1997). In addition to DNA, PAH intermediates can form adducts to albumin, a blood protein. Studies of albumin adducts utilize HPLC and fluorescence to measure exposure (Padros and Pelletier, 2000). Albumin adducts appear to be short-lived, however (Padros and Pelletier, 2000), and are of less relevance to carcinogenesis.

It has been determined that fish can serve as models for metabolism and adduct formation in vertebrates (Telli-Karakoc et al., 2001). With that in mind, induction of the MFO system initiates a myriad of chemical processes each with a unique effect on the cell, tissue, or

organism. Because of the direct involvement with genetic material, DNA adduct formation may provide the most direct link to the processes associated with carcinogenesis in fish. Therefore, quantification of adduct formation as a result of exogenous chemical exposure in fish may indicate the carcinogenic potential of a compound (Maccubbin, 1994; Stein et al., 1990). In addition, DNA adducts have proven to be a more consistent biomarker with less variability than EROD and CYP1A1 (Devaux et al., 1998; Willett et al., 1997) and may be detected at low levels (Aas et al., 2000). Levels of DNA adducts have been shown to correlate with levels of PAH exposure at multiple contaminated sites (Varanasi et al., 1989c).

Adduct formation to DNA causes damage to the genetic material and is, therefore, subject to repair mechanisms which are poorly understood (Maccubbin, 1994). Despite the potential for DNA repair, DNA adducts are relatively persistent in fish tissue (Rose et al., 2001; Myers et al., 1998b). Hepatic DNA adducts exhibit a half-life of 40-100 days (Myers et al., 1998b), and unrepaired adducts can persist for at least 84 days (Stein et al., 1993). Rose et al. (2001) found that DNA adducts in liver, kidney, and spleen tissue persisted at high levels for at least 96 days, although adduct levels in blood decreased sharply after 32 days (probably due to the shorter life-span of blood cells).

Differences in adduct formation and repair exist among species (Ericson et al., 1998b; Ploch et al., 1998), and these processes may also be influenced by complex mixtures of chemicals acting synergistically and antagonistically (Stein et al., 1990). Fish migration can complicate the relationship between DNA adduct formation and contamination at the site of capture (Lyons et al., 2000). Finally, differences in labeling efficiency can lead to variation in adduct measurements among procedures and laboratories (Ericson et al., 1998b).

The analysis of DNA adducts represents the most ecologically relevant biomarker analysis currently available for fish. The formation of DNA adducts represents an alteration of the organism's genetic material. The DNA can repair itself, however, and the link between DNA adducts and the development of cancer in free ranging fish has not been established. Although progress is being made on elucidating the chemical structure of individual metabolites and identifying compound-specific DNA adducts, it is not possible to identify and differentiate different sources of PAHs forming DNA adducts in free ranging fish.

6.2 Effects of PAHs on Fish

In examining effects of PAHs on fish, it is important to distinguish between ecologically adverse effects and effects that may not influence overall organism health and reproductive fitness:

> "The fact that very low levels of hydrocarbons in sediment, water, or foods may produce biological responses in fish attests to the importance of developing criteria for assessing the toxicological significance of sublethal effects or otherwise defining genuine harmful effects in contrast with effects per se (Payne et al., 1988)."

In an ecological risk assessment context, adverse effects are defined relative to the assessment endpoint selected as part of the risk assessment process, i.e., the ecosystem characteristic to be protected. Assessment endpoints for fish often relate to population maintenance and community

structure and function (e.g., fish species composition, diversity of feeding guilds, etc.) (USEPA, 1998). In a natural resource damages context, specific regulations apply to the definition of biological injury. Under Department of Interior regulations (43 CFR Part 11.62(f)(1)), injury to living resources is defined as:

- Death,
- Disease,
- Behavioral abnormalities,
- Cancer,
- Genetic mutations,
- Physical deformations, and
- Physiological malformations.

Under Superfund, injury to a natural resource is defined less specifically as a measurable adverse change in the chemical or physical quality or viability of that resource (CERCLA Section 101 (16)).

A variety of adverse effects have been observed in fish exposed under controlled laboratory conditions to oil, creosote, coal tar, and individual PAHs. Effects include mortality (DeGraeve et al., 1982; Edsall, 1991; Marty et al., 1997; Tadokoro et al., 1991; Van den Hurk et al., 1998; Vines et al., 2000), decreased growth (DeGraeve et al., 1982; Moles et al., 1981), behavioral abnormalities (Ostrander et al., 1990; Woodward et al., 1983), delayed spawning (Khan, 1991), decreased hatching success (Hose et al., 1981; 1982; Kocan et al., 1996a; Vines et al., 2000; Winkler et al., 1983), developmental abnormalities, such as defects of the eyes, skeleton, nervous system, and skin, fluid accumulation in the abdomen or around the heart, and lack of body pigment (Hannah et al., 1982; Hose et al., 1981; 1982; 1984; Kocan et al., 1996a; Marty et al., 1997; Vines et al., 2000; Winkler et al., 1983), cell death due to PAH-induced expression of viral hemorrhagic septicemia virus (Marty et al., 1999), enlarged liver (Payne et al., 1988), fin erosion (Sved et al., 1997; Woodward et al., 1983), and lesions and tumors (Hawkins et al., 1988; 1990).

Many of these effects have also been observed at sites contaminated with PAHs. Site-specific effects have been evaluated using several approaches, including laboratory exposures to sediments collected from the field (e.g., Fabacher et al., 1991; Kocan et al., 1996b), fish community surveys (e.g., Kuehn et al., 1995; Yoder and Rankin, 1995), and histopathological evaluation of wild-caught fish (i.e., examination of tissues for signs of disease), discussed in greater detail below. Site-based field studies have the advantage of reflecting actual environmental conditions, but the specific cause of observed effects can be unclear if multiple chemicals or other stressors co-occur at a site. As described further below, epidemiological techniques have been used to positively identify PAHs as a major contributor to certain adverse effects in fish, most notably in Puget Sound and the U.S. Pacific coast (Collier et al., 1997; Myers et al., 1998b) and the Great Lakes (Baumann and Harshbarger, 1995).

Among the various effects of PAHs on fish, histopathological effects have received particular attention in biomonitoring efforts, including those related to natural resource damage claims. Histopathology investigations have identified three major types of effects associated with PAHs

in sediment: (1) liver lesions, tumors, and other abnormalities; (2) external lesions and tumors; and (3) reproductive system effects. These effects are described below.

6.2.1 Liver Lesions, Tumors, and Other Abnormalities

In a review of the literature, Baumann (1998) identified 25 separate zoological epidemics (termed epizootics) of liver tumors (neoplasms) in North American fish. The suspected toxicants include PAHs in all but three cases, although causality often has not been rigorously demonstrated. Surveys of liver lesions and tumors in bottom-feeding fish are increasingly recommended for monitoring of aquatic environmental quality (e.g., Pinkney et al., 2001). The prevalence of liver lesions is strongly influenced by age, and in some species, by sex (Myers et al., 1994; Baumann et al., 1987); therefore, these characteristics should be recorded in monitoring surveys.

Liver lesions (including tumors) have been of primary interest in fish histopathology studies, because the liver exhibits lesions in field-collected fish more often than other organs (Myers et al., 1990). The particular susceptibility of the liver is not surprising, because the biotransformation of PAHs to electrophilic metabolites is a key step in carcinogenesis and occurs primarily in the liver (Stein et al., 1990). Metabolites of some PAHs can form DNA adducts (as discussed previously), and DNA adducts in turn can cause mutations due to faulty DNA repair or replication (Klaassen, 1996). The result is a series of lesions progressing toward tumors.

A spectrum of multiple, co-occurring liver lesions is found in susceptible fish species (e.g., English sole), closely resembling the series of lesions induced in rodents and fish through controlled chemical exposures in the laboratory (Myers et al., 1990). Table 6-1 lists 16 lesion types identified by Myers et al. (1990) and Stehr et al. (1998) for environmental monitoring purposes, together with an explanation of the associated terminology. Early lesions are small, affecting individual cells or cell clusters (termed foci of cellular alteration). Liver tumors are usually large and readily observable upon gross examination of the liver, with multiple nodules often forming the bulk of the liver (Myers et al., 1990). In addition to liver lesions, liver hyperplasia (increased number of cells) or increased liver weight relative to body weight (liver somatic index) have been observed in laboratory and field exposures to PAHs (Payne et al., 1988; Ericson et al., 1998b).

Table 6-1
Liver Lesions Found in English Sole and Other Fish Species (adapted from Myers et al., 1990 and Stehr et al., 1998)

Lesion type/category	Description	Explanation
Hepatocellular necrosis/degeneration	Nonspecific, degenerative - includes spongiosis hepatis	Necrosis refers to dead or dying liver cells; spongiosis hepatis is characterized by spaces in liver tissue filled with pale material
Nuclear pleomorphism	Unique, degenerative	Enlarged or multiple cell nuclei; precursor to megalocytic hepatosis
Megalocytic hepatosis	Unique, degenerative	Enlarged cells and nuclei, with specific characteristics (e.g., dark nuclei, transparent cytoplasm)
Hepatocellular regeneration	Proliferative, non-neoplastic	Liver cell growth, replacing tissue damaged by megalocytic hepatosis; regenerating cells can become tumorous
Clear cell focus of cellular alteration	Preneoplastic	Lesion that can become a tumor; named for appearance after standard staining techniques
Eosinophilic focus of cellular alteration	Preneoplastic	Lesion that can become a tumor; named for appearance after standard staining techniques
Basophilic focus of cellular alteration	Preneoplastic	Lesion that can become a tumor; named for appearance after standard staining techniques
Liver cell adenoma	Benign neoplasm	Non-invasive tumor originating from liver cells
Hepatocellular carcinoma	Malignant neoplasm	Invasive tumor originating from liver cells
Cholangioma	Benign neoplasm	Non-invasive tumor originating from bile duct cells
Cholangiocellular carcinoma	Malignant neoplasm	Invasive tumor originating from bile duct cells
Mixed hepatobiliary carcinoma	Malignant neoplasm	Invasive tumor originating from both liver and bile duct cells
Cholangiofibrosis	Proliferative, non-neoplastic	Fibrous lesion (scar tissue) on bile duct
Hemosiderosis	Storage disorder	Iron overload
Steatosis/fatty change	Storage disorders	Fatty liver
Hydropic vacuolation	Unique, unknown if degenerative or proliferative	Swollen, fluid-filled vacuoles; observed in species less susceptible to tumors than English sole

6.2.2 External Tumors and Other External Abnormalities

Baumann (1998) identified 18 reported epizootics of skin tumors in North American fish. The suspected toxicants include PAHs in 11 of these cases. Other suspected causes listed for skin tumor epizootics are chlorine, PCBs, radiation, pulp waste, and "toxics" (Baumann, 1998). Viruses and UV light have also been suggested as potential causes of external tumors in fish. Freshwater bottom-feeders, including black bullhead, brown bullhead, and white sucker, appear to be particularly susceptible to external tumors (Baumann, 1998). Pinkney et al. (2001)

identified two categories of tumors in brown bullhead collected from PAH-contaminated sites in Chesapeake Bay, namely cancerous (invasive) squamous carcinomas and non-cancerous epidermal papillomas. The tumors were generally found in the mouth area and were fleshy-pink colored, round or oval, solid in texture, and at least in some cases of significant size (Pinkney et al., 2001; Figure 6-2). Fish age and sex strongly affect the likelihood of external tumors in brown bullhead (Baumann et al., 1987).

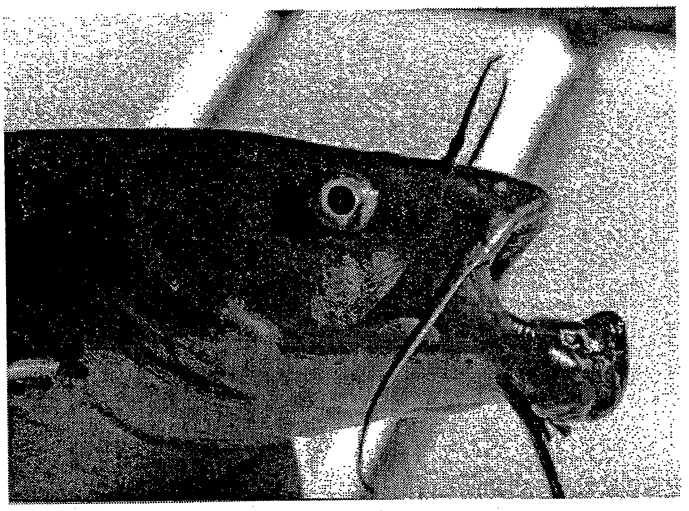


Figure 6-2
External Tumor on Brown Bullhead. (Photo by J. Forester, reproduced courtesy of F. Pinkney, originally presented by Pinkney et al., 2001.)

External anomalies in fish are often recorded during fish community surveys, particularly those conducted using the Index of Biotic Integrity (IBI), a multimetric fish assemblage assessment approach originally developed by Karr (1981) and frequently modified based on regional considerations (Simon and Lyons, 1995). Most versions of the IBI include a metric assessing the percentage of individuals exhibiting grossly observable external anomalies. A large database of information on the prevalence of such anomalies is available from the Ohio Environmental Protection Agency, which is recognized as a leader in the monitoring of fish community quality. Ohio's version of the IBI includes a metric addressing the percentage of fish exhibiting deformities, eroded fins, lesions, and tumors (DELT anomalies). Yoder and Rankin (1995) evaluated patterns of IBI metric values among various pre-determined impact types and identified elevated levels of DELT as the most diagnostic characteristic of "complex toxic" impacts. Especially high DELT levels were observed in the Ottawa River (Sanders et al., 1998; Yoder and Rankin, 1995), in an area where sediments contain highly elevated PAH concentrations (Cormier et al., 2000b).

It is clear from laboratory experiments that PAHs have the potential to cause deformities, eroded fins, external lesions, and tumors, although deformities have most often been reported in fish larvae rather than adults (perhaps due to poor survival of affected individuals). However, these types of abnormalities can also occur due to other factors. For example, although Fabacher et al. (1991) observed fin erosion in fish exposed to crude extracts of contaminated sediments, the incidence of fin erosion was much lower upon exposure to redissolved fractions containing PAHs, indicating that this effect may have been caused by other contaminants. Thus, elevated

DELT levels may sometimes be due to PAHs, while low levels of DELT indicate the absence of a PAH-related epizootic of external anomalies.

6.2.3 Reproductive System Disorders

Successful reproduction of a species is imperative to maintain a viable population. Among the life stages of fish populations, embryos and larvae are the most sensitive to toxicant exposure (Marty et al., 1997; Varanasi et al., 1989a) and are key to the growth of the population; however, few studies have focused on this area of research. The effects of PAH contamination on fish reproduction may be acute (e.g., hatching success) or chronic (e.g., decreased steroid production, reduced gonadal size). Shortnose sturgeon embryos and larvae exposed to high concentrations of PAH compounds in coal tar suffered 95% mortality (Kocan et al., 1996b). A decrease in hatching success was observed in sand sole treated with BaP (Hose et al., 1982). Pacific herring exposed to petroleum from the Exxon Valdez oil spill had a lower percent hatch rate than herring from an uncontaminated site (Kocan et al., 1996a).

There are numerous sublethal effects of PAH contamination in adult fish that can affect reproduction. Decreased gonadal size is a parameter that may potentially affect the entire reproductive process and is found in fish exposed to PAHs (Munkittrick et al., 1995; Tuvikene, 1995). Circulating hormone levels (i.e. plasma-estradiol) have been shown to be decreased in female English sole from contaminated sites (Johnson et al., 1993). Vitellogenesis (yolk formation) can be affected by PAH contamination. As an example of the outcome of such changes, anthracene lowered the reproductive output (measured as the mean number of eggs laid) in fathead minnows and was also concentrated more in the ovaries of the minnows than in the testes (Hall and Oris, 1991).

Developmental anomalies in fish may occur after exposure to PAH contamination. Pacific herring larvae from adults exposed to oil from the Exxon Valdez spill showed an increase in morphological changes (Kocan et al., 1996a). Exposure to BaP can cause an increase in developmental anomalies, as demonstrated in sand sole, English sole and rainbow trout by Hose et al. (1982, 1984). Emergence success in coho salmon was reduced and altered by embryonic exposure to BaP (Ostrander et al., 1990).

6.3 Relationship Between Exposure and Effects

Understanding the quantitative relationship between exposure and effects is useful for purposes of predictive risk assessment and retrospective forensic investigation. Both the identification of a "safe" (no-effect) concentration and a dose-response relationship at higher concentrations are useful in this regard. Information documenting the relationship between exposure and effects in fish is limited and has not been compiled on a comprehensive basis. Additionally, much of the available information is based on field studies where the identification of specific agents causing the observed toxicity is challenging. The following subsections discuss the process of establishing causality in field studies and summarize available concentration-response data for PAHs in sediment and other measures of PAH exposure.

6.3.1 Establishing Causality

Where feasible, it is preferable to draw on controlled laboratory experiments capable of characterizing cause-effect concentration-response relationships. However, for PAHs in sediment and their effects on fish, much of the available data regarding the relationship between exposure and effects has been obtained from field studies rather than controlled laboratory exposures. This is due primarily to the long latent period between PAH exposures and effects such as lesions and tumors. In response to this type of difficulty, analytical epidemiologists have developed criteria for determining whether a field-based association is due to a cause-effect relationship. As described by Landahl et al. (1990), key criteria include:

- Strength, consistency, and specificity of the association;
- Biological plausibility;
- Time sequence where exposure occurs before effects; and
- Occurrence of a dose-response gradient.

Fox (1991) and USEPA (2000c) list additional lines of evidence that should be considered, such as predictive performance, factual coherence, evidence of a complete exposure pathway, concordance with experimental studies, and existence of analogous cases.

Investigations that have developed multiple lines of evidence supporting the identification of PAHs as the cause of adverse histopathological effects include those conducted on English sole in Puget Sound and elsewhere along the U.S. Pacific coast (Collier et al., 1997; Landahl et al., 1990; reviewed by Myers et al., 1998b) and on brown bullhead in the Great Lakes regions (Baumann and Harshbarger, 1995; 1998). Among the lines of evidence that have been developed to establish causality are:

- Documentation of a complete exposure pathway, based on analyses of bile metabolites, DNA adducts, and/or tissue residues of PAHs (Baumann and Harshbarger, 1995; Baumann et al., 1991; Myers et al., 1998b);
- Laboratory induction of lesions in fish exposed to extracts of contaminated sediments (Fabacher et al., 1991; Myers et al., 1990);
- Documentation of a plausible biological mechanism of toxicity and carcinogenesis, by analogy to controlled laboratory experiments using rodents and fish (Myers et al., 1998b);
- Documentation of a high degree of consistency of association between PAHs and adverse effects at multiple sites (Baumann, 1998; Landahl et al., 1990);
- Calculation of a dose-response relationship between PAH concentrations and sediment and prevalence of liver lesions in English sole (Horness et al., 1998); and
- Documentation of a temporal sequence of changes in liver lesion prevalence in brown bullhead in the Black River, OH, during coking plant operation, plant closure and subsequent natural attenuation of PAHs, resuspension of contaminated sediments during sediment removal, and recovery following the remedial action (Baumann and Harshbarger, 1995; 1998; Baumann et al., 2000).

The extensive effort and credibility of these studies lends credence by analogy to associations between PAHs and similar effects observed elsewhere, although the potential for observable adverse effects due to non-PAH stressors must be recognized.

6.3.2 Sediment PAH Concentrations versus Effects

Horness et al. (1998) used hockey stick regression methods to identify a threshold concentration of PAHs in sediment, above which the incidence of liver lesions (including cancer) in English sole increases along a dose-response gradient. The data set included 29 pairs of sediment PAH and liver lesion data, where lesion prevalence was calculated for at least 30 fish in each case. English sole were collected from coastal sites in California, Oregon, Washington, and Alaska. Total PAH concentrations in sediment were calculated from the measured concentrations of 18 PAH compounds. It was not possible to account for the effect of organic carbon on PAH bioavailability, because total organic carbon levels were not consistently measured. A threshold concentration of 0.62 mg/kg total PAHs was identified, with confidence limits of 0.3 to 1 mg/kg.

Several issues merit particular consideration in applying the threshold developed by Horness et al. (1998). First, English sole are more susceptible to liver lesions than most other fish species (Horness et al., 1998; Myers et al., 1998b). Thus, the threshold concentration of 0.62 mg/kg may not be applicable to ecosystems not inhabited by English sole (e.g., fresh water). Second, the threshold concentration does not account for factors affecting PAH bioavailability (e.g., organic carbon or soot content of sediment) or the relative toxicity of different PAH compounds. This will tend to reduce the predictive ability of the threshold value. Third, it is possible that an appropriate threshold concentration for English sole would be higher in the absence of contaminants other than PAHs. In addition to PAHs, concentrations of PCBs, DDTs, chlordanes, and metals were strongly associated with lesion prevalence in English sole (Horness et al., 1998; Myers et al., 1998b). It is worth noting, however, that Baumann et al. (1991) observed no liver tumors in brown bullhead from the Fox River, despite elevated PCB concentrations in sediment, and PCBs were not strongly associated with liver lesions in winter flounder from various coastal sites in New York and New England (Myers et al., 1998b). These comparisons do not account for differences in toxicity among individual PCB compounds.

In contrast to the field-based approach implemented by Horness et al. (1998), the U.S. Environmental Protection Agency (USEPA, 2000a) has estimated PAH sediment toxicity thresholds for fish based solely on laboratory data from controlled experiments. The purpose of the USEPA evaluation was to determine whether the guidelines for PAH mixtures developed to protect benthic invertebrates would also be protective of fish. Specifically, USEPA (2000a) identified concentrations of BaP and anthracene in fish eggs that caused developmental abnormalities and then estimated the sediment concentrations that would be associated with the measured egg concentrations, under equilibrium conditions. A data set of PAH concentrations in 539 sediment samples was evaluated to determine whether the "critical" concentrations of BaP or anthracene would be exceeded when the benthic invertebrate guidelines were not. Although the "critical" concentrations for fish were not exceeded in samples deemed safe for benthic invertebrates, the evaluation was limited by the consideration of only two individual PAHs. Assuming an organic carbon concentration in sediment of 1%, the "critical" concentrations for BaP and anthracene were estimated as 0.57 mg/kg and 2.19 mg/kg, respectively (USEPA, 2000a).

Linton et al. (2000) extended this approach by utilizing BaP equivalents (Nisbit and LaGoy, 1992), allowing the assessment of mixtures of eight teratogenic or carcinogenic PAHs. BaP equivalents were calculated by multiplying the concentration of another PAH by an adjustment factor (Table 6-2) based on the relative carcinogenic potency, to mammals, of the two compounds (e.g., an adjustment factor of 0.5 would be applied to a PAH that exhibits one-half the carcinogenic potency of BaP). The adjusted concentration of the eight PAHs were summed to calculate total BaP equivalents, which in turn were compared to the same "critical" concentration developed for BaP. In the data set of PAHs in 539 sediment samples, the "critical" concentration was exceeded in approximately 1% of the samples deemed safe for benthic invertebrates (Linton et al., 2000). While this approach is again limited by the small number of compounds evaluated, it suggests that appropriate threshold concentrations of PAHs in sediment are similar for benthic invertebrates and fish. Indeed, Swartz (1999) proposed a screening-level guideline of 1 mg/kg total PAHs in sediment for the protection of benthic invertebrates, based loosely on the assessment model described by USEPA (2000a); this value is similar to the threshold PAH concentration identified by Horness et al. (1998) for the protection of English sole.

Table 6-2
Toxicity Equivalency Factors for PAH Carcinogenicity (adapted from Nisbet and LaGoy, 1992)

Compound	Toxicity Equivalency Factor
Dibenzo(a,h)anthracene	5
Benzo(a)pyrene (BaP)	1
Benzo(a)anthracene	0.1
Benzo(b)fluoranthene	0.1
Benzo(k)fluoranthene	0.1
Indeno(1,2,3-c,d)pyrene	0.1
Anthracene	0.01
Chrysene	0.01

It is important to note that at some sites, much higher concentrations of PAHs in sediment have shown only limited or no effect on fish (see Table 6-3). For example, mummichog in the creosote-contaminated Elizabeth River (Virginia) exhibited no liver lesions at a total PAH concentration in sediment of 61 mg/kg, although fatty liver was observed at this concentration (Vogelbein et al., 1990). In a coal tar contaminated estuary in Nova Scotia, PAH concentrations up to 191 mg/kg resulted in no significant effects on liver or gonad weight or body weight as a function of length; lesions were not evaluated (Vandermeulen and Mossman, 1996; Vignier et al., 1994). Conversely, liver and skin lesions have been noted at apparently uncontaminated reference sites (e.g., Baumann et al., 1991; Smith et al., 1994).

Sved et al. (1997) demonstrated that high molecular weight PAHs produced different and more severe effects on spot (*Leostomus xanthurus*) than low molecular weight PAHs, in tests using suspended sediments that had been spiked with fractions of creosote. In addition to PAH composition, it is also logical to expect that the organic carbon and soot content of sediment would influence the toxicity of sediment-associated PAHs to fish, given their critical role in determining PAH bioavailability to invertebrates (USEPA, 2000a). Models that account for both PAH composition and bioavailability are likely to provide a more reliable prediction of adverse effects on fish than the exposure-effects relationships developed to date.

Table 6-3
Comparison of PAH Concentrations in Sediment and Adverse Effects on Fish in Selected Studies

Species	Exposure Description	PAH Concentration (dry weight)	Adverse Effects	Reference
Brown bullhead (*Ameiurus nebulosus*)	Black River, OH, adjacent/downstream from coking plant; Wild-caught fish	381.2 mg/kg (1982) 4.27 mg/kg (1987) 16.6 mg/kg (1992, 2 y post-dredging) 9.83 mg/kg (1994) (sum of 11 PAHs)	77% of age 3 fish exhibit liver lesions; 31% liver cancer (1982) 55% of age 3 fish exhibit liver lesions; 7% liver cancer (1987) 54% of age 3 fish exhibit liver lesions; 27% liver cancer (1992) 15% of age 3 fish exhibit liver lesions; no cancer (1994)	Baumann and Harshbarger (1998)
Brown bullhead (*Ameiurus nebulosus*)	Cuyahoga River, OH (metals contamination noted); Wild-caught fish	50 mg/kg (sum of 50 PAHs) 10.8 mg/kg (sum of 6 carcinogenic PAHs)	Approximately 67% exhibit liver lesions; 8.9% exhibit external neoplasms, primarily carcinomas	Baumann et al. (1991); Fabacher et al. (1988)
Brown bullhead (*Ameiurus nebulosus*)	Fox River, WI (PCB/dioxin contamination noted); Wild-caught fish	82 mg/kg (sum of 50 PAHs) 3.6 mg/kg (sum of 6 carcinogenic PAHs)	No liver neoplasms or preneoplastic lesions; 7.7% exhibit external neoplasms, primarily epidermal papillomas	Baumann et al. (1991); Fabacher et al. (1988)
Brown bullhead (*Ameiurus nebulosus*)	Menominee River, WI (arsenic contamination noted); Wild-caught fish	27 mg/kg (sum of 50 PAHs) 2.98 mg/kg (sum of 6 carcinogenic PAHs)	No liver neoplasms or preneoplastic lesions; no external neoplasms	Baumann et al. (1991); Fabacher et al. (1988)
Brown bullhead (*Ameiurus nebulosus*)	Munuscong Lake, MI (reference site); Wild-caught fish	<1 mg/kg (sum of 50 PAHs) 0.181 mg/kg (sum of 6 carcinogenic PAHs)	Approximately 33% exhibit liver lesions; no external neoplasms	Baumann et al. (1991); Fabacher et al. (1988)

Species	Exposure Description	PAH Concentration (dry weight)	Adverse Effects	Reference
Brown bullhead (*Ameiurus nebulosus*)	Black River, OH adjacent/downstream from coking plant; Wild-caught fish	3.57 - 4.74 mg/kg (sum of 13 PAHs)	51% exhibit liver lesions; 53% exhibit external abnormalities Also fish community less diverse, more tolerant species relative to reference site	Smith et al. (1994)
Brown bullhead (*Ameiurus nebulosus*)	Cuyahoga River, OH; Wild-caught fish	34.44 - 91.55 mg/kg (sum of 13 PAHs)	45% exhibit liver lesions; 74% exhibit external abnormalities Also fish community less diverse, more tolerant species relative to reference site	Smith et al. (1994)
Brown bullhead (*Ameiurus nebulosus*)	Huron River, OH (reference site); Wild-caught fish	0.62 - 0.78 mg/kg (sum of 13 PAHs)	4% exhibit liver lesions; 14% exhibit external abnormalities	Smith et al. (1994)
Brown bullhead (*Ameiurus nebulosus*)	Anacostia River, Washington DC (PCB, pesticide and metal contamination noted); Wild-caught fish, spring and fall collections	26.8 mg/kg (total resolved PAHs) 9.0 mg/kg (sum of 8 carcinogenic PAHs)	77-80% exhibit liver or skin lesions; 50-60% exhibit liver/bile tumors; 10-37% exhibit skin tumors; relative liver weight elevated	Pinkney et al. (2001)
Brown bullhead (*Ameiurus nebulosus*)	Quantico embayment, VA (PCB and DDT contamination noted); Wild-caught fish	5.1 mg/kg (total resolved PAHs) 1.2 mg/kg (sum of 8 carcinogenic PAHs)	10% exhibit liver or skin lesions; 7% exhibit liver/bile tumors; 3% exhibit skin tumors	Pinkney et al. (2001)
Brown bullhead (*Ameiurus nebulosus*)	Neabsco Creek, VA (marinas and urban runoff noted); Wild-caught fish	4.8 mg/kg (total resolved PAHs) 1.4 mg/kg (sum of 8 carcinogenic PAHs)	20% exhibit liver or skin lesions; 17% exhibit liver/bile tumors; 3% exhibit skin tumors	Pinkney et al. (2001)
Brown bullhead (*Ameiurus nebulosus*)	Tuckahoe River, MD (reference area); Wild-caught fish	1.8 mg/kg (total resolved PAHs) 0.5 mg/kg (sum of 8 carcinogenic PAHs)	13% exhibit liver or skin lesions; 10% exhibit liver/bile tumors; no skin tumors	Pinkney et al. (2001)

Species	Exposure Description	PAH Concentration (dry weight)	Adverse Effects	Reference
English sole (*Pleuronectes vetulus*)	29 Locations, U.S. Pacific coast; Wild-caught fish, hockeystick regression	0.62 mg/kg (sum of 18 PAHs)	Threshold concentration above which incidence of liver lesions increases in dose-response manner	Horness et al. (1998)
European eel (*Anguilla anguilla*)	6 freshwater sites, Amsterdam Wild-caught fish	1.3 - 45 mg/kg (19 - 141 µg/g organic carbon) (sum of 16 PAHs)	No liver tumors	Van der Oost et al. (1994)
European eel (*Anguilla anguilla*)	2 freshwater sites, Amsterdam Wild-caught fish	25 - 128 µg/g organic carbon (sum of multiple PAHs)	No effect on relative weight of liver	Van der Oost et al. (1991)
Mummichog (*Fundulus heteroclitus*)	Creosote-contaminated estuary (Elizabeth River, VA); Wild-caught fish	3 - 2,200 mg/kg (Total PAHs, individual compounds not quantified)	No effect on liver condition (3 mg/kg) Fatty liver observed; no neoplasms or preneoplastic lesions (61 mg/kg) 93% of fish exhibit liver lesions; 33% exhibit hepatocellular carcinomas (2,200 mg/kg)	Vogelbein et al. (1990)
Northern pike (*Esox lucius*)	2 freshwater sites, Amsterdam Wild-caught fish	25 - 128 µg/g organic carbon (sum of multiple PAHs)	No effect on relative weight of liver	Van der Oost et al. (1991)
Roach (*Rutilus rutilus*)	2 freshwater sites, Amsterdam Wild-caught fish	25 - 128 µg/g organic carbon (sum of multiple PAHs)	No effect on relative weight of liver	Van der Oost et al. (1991)
Shortnose Sturgeon (*Acipenser brevirostrum*)	Coal tar-contaminated sediment from Connecticut River, MA; 21-day laboratory test	28,790 mg/kg (sum of 17 PAHs)	95% mortality of embryos/larvae; surviving larvae appeared incapable of long-term survival	Kocan et al. (1996b)

Species	Exposure Description	PAH Concentration (dry weight)	Adverse Effects	Reference
Spot (*Leiostomus xanthurus*)	Creosote-contaminated sediment from Elizabeth River, VA; 28-day laboratory study	62.5 - 27,150 mg/kg (sum of 21 PAHs)	Weight loss (62.5 mg/kg) LT50 = 19.5 days (230.5 mg/kg) LT50 = 57 minutes (27,150 mg/kg)	Roberts et al. (1989)
Walleye (*Stizostedion vitreum*)	Fox River, WI (PCB/dioxin contamination noted); Wild-caught fish	82 mg/kg (sum of 50 PAHs) 3.6 mg/kg (sum of 6 carcinogenic PAHs)	2.5% exhibit liver neoplasms; no external lesions	Baumann et al. (1991); Fabacher et al. (1988)
Walleye (*Stizostedion vitreum*)	Menominee River, WI (arsenic contamination noted); Wild-caught fish	27 mg/kg (sum of 50 PAHs) 2.98 mg/kg (sum of 6 carcinogenic PAHs)	3.6% exhibit liver lesions; no external lesions	Baumann et al. (1991); Fabacher et al. (1988)
Walleye (*Stizostedion vitreum*)	Munuscong Lake, MI (no industrial sources); Wild-caught fish	<1 mg/kg (sum of 50 PAHs) 0.181 mg/kg (sum of 6 carcinogenic PAHs)	2.5% exhibit liver lesions; 10% exhibit non-neoplastic external lesions	Baumann et al. (1991); Fabacher et al. (1988)
Winter flounder (*Pleuronectes americanus*)	Coal tar-contaminated estuary (Sydney estuary, Nova Scotia); Wild-caught fish, multiple years	7.19 - 191 mg/kg (sum of 13 PAHs)	No effect on fish weight relative to length, no effect on relative weight of liver or gonads	Vignier et al. (1994); Vandermeulen and Mossman (1996)

Species	Exposure Description	PAH Concentration (dry weight)	Adverse Effects	Reference
Winter flounder (*Pleuronectes americanus*)	Crude oil-spiked sediment; 4 month laboratory exposure	0.41 - 589 mg/kg (Total PAHs by HPLC/fluorescence)	No significant effect (0.41 mg/kg) Increased relative weight of liver (27.3 - 44.3 mg/kg) Increased relative weight of liver, decreased relative weight of spleen (254 - 589 mg/kg) No effect on fish weight relative to length or relative weight of testis, kidney, or gut at any concentration; stepwise trend in fatty liver for all concentrations, not significant	Payne et al. (1988)

6.4 Other Measures of PAH Exposure versus Effects

One might expect that exposure measures in fish specimens, such as DNA adducts, bile metabolites, or possibly EROD, would provide a more accurate assessment of fish exposure to PAHs than the measurement of PAH concentrations in sediment, given the variety of factors affecting PAH uptake from sediment by fish (described previously). As such, one might expect these alternative exposure measures to be more reliably predictive of adverse effects on fish. However, little evidence exists to date to support this hypothesis, as described below.

Myers et al. (1998b) summarize the results of extensive Pacific and Northeast coast studies that measured sediment PAHs, bile metabolites, DNA adducts, CYP1A expression, and liver lesion prevalence in several fish species. Average levels of all exposure parameters were generally associated with lesion prevalence. However, sediment PAH concentrations always explained more of the variation in lesion prevalence than did average bile metabolite levels. Additionally, paired exposure and effects data were measured for individual fish in Eagle Harbor, Washington. These data indicated that DNA adducts, but not bile metabolites or CYP1A expression, were a significant risk factor for liver lesions. No attempt was made to identify a threshold DNA adduct level below which liver lesions would not be expected to occur, or to compare the reliability of sediment PAHs versus DNA adducts in predicting liver lesions.

Pinkney et al. (2001) measured sediment PAHs, bile metabolites, EROD, and lesion prevalence (both liver and skin) in brown bullheads collected from four locations in Chesapeake Bay. Sediment PAH concentrations were more predictive of lesion prevalence than either bile metabolites or EROD. The authors cite the short-term nature and variability of bile metabolites as an exposure metric, as well as the susceptibility of EROD to seasonal fluctuation and induction by PCBs, as possible explanations for the observed results.

Ericson et al. (1998b) evaluated DNA adducts, body and organ measurements, and liver lesions in perch collected from Sweden's Baltic Coast. The study included four sites along a gradient of sediment PAH contamination associated with an aluminum smelter, as well as a relatively uncontaminated reference site. Concentrations of PAHs in sediment were not measured, but a previous survey had indicated that PAH concentrations decreased with distance from the smelter, with a maximum concentration of 211 mg/kg (based on 15 PAH compounds). Concentrations of DNA adducts in perch liver ranged from approximately 2 to 29 nmol/mol normal nucleotides (values estimated from figure) in the study area and were not detected in fish from the reference area. The two intermediate locations in the study area exhibited the same levels of DNA adducts (approximately 5 nmol/mol normal nucleotides in liver), but not the same levels of liver lesions. At the location nearer the smelter, perch livers contained lesions, whereas no lesions were observed at the more distant of the two locations. Other observed effects included decreased fish size (three inner locations) and decreased spleen weight (most highly exposed location only). Fish condition as evaluated by body weight relative to length, gonad weight, and liver weight were similar at all locations.

Finally, Au et al. (1999) attempted to draw a link between EROD activity and liver histopathology in fish injected with BaP. Although changes were observed at the cellular level

(increased lipofuscin granules and peroxisomes in liver cells), it is not clear that the observed effects should be considered adverse from an ecological perspective.

6.5 Risks to Piscivorous Wildlife

In addition to experiencing adverse effects themselves, fish can serve as a vehicle for wildlife exposures to bioaccumulative chemicals. Because fish efficiently metabolize PAHs, consumption of fish by wildlife is not likely to be an important ecological exposure pathway. However, PAH metabolism is not instantaneous, and PAHs can therefore be present in fish at detectable (though low) concentrations. For this reason, an assessment of risks associated with wildlife fish consumption may sometimes be required in a regulatory context. This type of assessment is necessarily restricted to un-metabolized PAHs in prey fish, because the effects on wildlife of consuming PAH metabolites in prey are completely unknown.

It would be convenient for screening purposes to identify a generic, conservative biota-sediment accumulation factor (BSAF) for PAHs in fish. This BSAF could be used to estimate wildlife exposure concentrations, in lieu of directly measuring site-specific PAH concentrations in fish. Tracey and Hansen (1996) compiled published BSAFs for several chemicals in benthic invertebrates and bottom-dwelling fish, but they identified no BSAFs for PAHs in fish (although numerous BSAFs were identified for invertebrates). As part of the present review, a small number of studies were identified that provide an indication of the BSAFs that may be encountered for PAHs in fish (Baumann and Harshbarger, 1995; Baumard et al., 1998; Djomo et al., 1996; van der Oost et al., 1991;1994). Biota-sediment accumulation factors were calculated from the data presented in those studies as follows:

$$BSAF = \frac{PAH\ concentration\ in\ fish\ (wet\ weight)}{PAH\ concentration\ in\ sediment\ (dry\ weight)}$$

Wet weight tissue residues were used, because risks to wildlife are often assessed using ingestion rates of fresh diet rather than dry matter. The available data are insufficient in most cases to develop BSAFs that are normalized to account for the influence of sediment organic carbon content and fish lipid content on the extent of bioaccumulation. Normalization would be expected to reduce the variability among BSAFs.

Baumard et al. (1998) measured PAH bioaccumulation in red mullet and painted sea comber collected from multiple sites that exhibited very low PAH contamination. The average BSAF derived from these analyses is 0.31, calculated assuming the fish contained 75% moisture and excluding one outlier. However, BSAFs for fish are reduced when MFO systems are induced due to sediment contamination (van der Oost, 1991). Van der Oost (1991;1994) observed significant differences between multiple sampling events in BSAFs for European eel muscle tissue, but it is clear that the BSAFs were always less than or equal to 0.1 at multiple contaminated sites and an order of magnitude higher at reference sites. These trends were also observed for northern pike and roach (van der Oost, 1991). In a spiked sediment study with zebra fish, Djomo et al. (1996) obtained an average steady-state BSAF of approximately 0.085. Tissue analyses of brown bullhead from the Black River, OH, indicate BSAFs of approximately 0.001 to 0.006 (Baumann and Harshbarger, 1995). Based on these studies, it is unlikely that fish collected from PAH-contaminated sites will contain (on a wet weight basis) more than half the

PAH concentration observed in sediment (on a dry weight basis), i.e., a BSAF of 0.5 is conservative.

The conservative BSAF of 0.5 is used here to demonstrate the low susceptibility of wildlife to PAH exposure via fish consumption, by calculating a safe concentration of PAHs in sediment for piscivorous wildlife. As described by Cura et al. (this volume), wildlife exposures can be characterized using the following equation:

$$Dose = Normalized ingestion rate \times Concentration$$

where the normalized ingestion rate is the amount of material ingested divided by the species' body weight. For prey fish, the concentration term can be expressed as:

$$Concentration\ in\ fish = Concentration\ in\ sediment \times BSAF$$

In order to calculate a safe concentration of PAHs in sediment based on the fish consumption exposure pathway, (1) piscivorous wildlife species of interest are selected; (2) the dose is set to an appropriate safe (no-effect) level; (3) values are obtained for the normalized ingestion rate and BSAF parameters; and (4) the equation is solved for the concentration in sediment.

The belted kingfisher and mink are selected as representative piscivorous wildlife receptors of interest for this example. Most piscivorous wildlife species are birds. The belted kingfisher is often conservatively selected as a representative piscivorous bird species, because it forages within a relatively small area and exhibits a higher normalized ingestion rate than other, larger fish-eating birds (USEPA, 1993). The prey of belted kingfisher, consisting of smaller pelagic fish, may contain lower chemical concentrations than other fish species, but for the purposes of this example calculation all prey fish are assumed to exhibit the same conservative BSAF. Aside from marine mammals (e.g., whales and seals), few mammal species feed primarily on fish. Mink and river otters are frequently assessed; mink are better understood from a toxicological perspective. Although mink are opportunistic feeders and may consume a substantial fraction of their diet as benthic invertebrates (e.g., crayfish) and terrestrial organisms, they will consume fish and are often selected as a representative piscivorous mammal for risk assessment purposes.

To select no-effect doses for this example, toxicity studies are identified in which birds and mammals were exposed to PAHs over a significant duration (i.e., chronic studies). Relatively few PAH toxicity studies have been conducted using birds. A study by Patton and Dieter (1980) reported mortality in mallards exposed for 7 months to a PAH mixture in food; no effects were observed at a dose of 277 mg/kg/day. For mammals, naphthalene and BaP have been the most thoroughly studied PAHs. Data for these compounds are used here as a surrogate for other, less-studied PAHs. Based on the results of Mackenzie and Angevine (1981) and NTP (1991), and using a body-weight scaling factor as recommended by Sample et al. (1996), appropriate no-effect doses for mink exposed to naphthalene and BaP are identified as 40 and 0.43 mg/kg/day, respectively.

Based on data provided by USEPA (1993), normalized ingestion rates for belted kingfisher and mink are 0.49 and 0.22 kg/day, respectively. Safe concentrations of PAHs in sediment are calculated using these values, together with the no-effect doses identified above and a

conservative BSAF of 0.5. For the belted kingfisher, adverse effects would not be expected at a total PAH concentration in sediment of approximately 1,000 mg/kg. Significant adverse effects on aquatic receptors would be expected at this concentration, indicating that risks to piscivorous birds are not likely to influence environmental management decisions for PAHs in sediments. Similarly, a safe concentration of PAHs in sediment for mink consuming 100% fish lies between 4 and 360 mg/kg, assuming that the mink consumes fish containing a mixture of PAHs exhibiting toxicities intermediate between naphthalene and BaP. Again, adverse effects on aquatic organisms would be expected to occur at lower concentrations. These example calculations illustrate the low importance of PAH bioaccumulation in fish with respect to consumption by piscivorous wildlife.

6.6 Implications for MGP Sites

Many former MGP sites are located near waterways, because these sites provided an economical method of delivering raw materials, a plentiful supply of water to support the operations of the plant, and a receiving body for dissemination of wastewater. In the years since these plants were operational, significant regulatory attention was focused on the cleanup of the sites and the transition of these properties to other beneficial uses. As these land use issues are resolved, the regulatory focus is shifted to off-site issues, including assessing past and on-going impacts to the adjacent waterways. Although not specific to manufactured gas sites, surface water and sediment issues have gained the national spotlight and are commanding the attention of federal and state regulators and natural resource trustee agencies.

It is clear that PAHs in sediment can adversely affect fish, as well as benthic invertebrates (McGrath et al., this volume), although the concentrations at which adverse effects on fish occur are less clearly established. Source control measures to prevent or reduce PAH releases to water bodies are likely to be required or may be implemented proactively. Depending on site conditions, it may be appropriate to implement such controls before initiating a formal evaluation of in-place sediment contamination.

This chapter provides information on a variety of approaches to measuring PAH exposures and effects on fish. These approaches have been implemented by natural resource trustees, agencies charged with water quality monitoring, and other researchers at various PAH-contaminated sites. However, not all of the widely used assessment methods are equally relevant to environmental management decision-making. For example, measures of exposure are only useful for assessing risks if they can be linked to a measure of adverse effects. If a fish is exposed to PAHs but rapidly biotransforms and eliminates them, adverse effects will not necessarily occur. As described above, biomarkers such as MFO enzymes, bile metabolites, and DNA adducts have shown a weaker relationship with adverse effects than PAH concentrations in sediment.

The USEPA Office of Research and Development recently sponsored a study to determine whether the equilibrium partitioning sediment guidelines (ESGs) developed for the protection of benthic invertebrates (McGrath et al., this volume) are also protective of fish (Linton et al., 2000; USEPA, 2000a). The preliminary conclusion was that the ESGs do protect fish from carcinogenic and developmental effects related to PAHs in sediment. This work needs to be verified, especially in the context of long-term exposure to PAHs in sediment and the development of cancerous lesions and tumors in free-ranging fish populations. Environmental

management decision-making at former MGP sites would be facilitated by the development and refinement of methods for using PAH concentrations in sediment to assess risks to fish.

In the absence of PAH sediment guidelines for the protection of fish, direct observations of fish community quality and fish health will be a practical assessment option in many cases. As described earlier in this chapter, the IBI is a widely used tool for this purpose. Based on non-destructive fish survey results, the IBI integrates various measures of fish abundance, species richness, prevalence of tolerant and intolerant species, trophic and spawning guilds, and grossly observable fish condition. This approach has been effectively implemented throughout the U.S. in freshwater, estuarine, and coastal settings (Simon, 1998; USEPA, 2000d). As such, it is more readily implemented than specialized procedures such as liver histopathology studies. The IBI integrates effects on fish reproduction and survival (both a function of fish health), as well as external anomalies. In addition to a community-level assessment, individual components of the IBI can be related to specific natural resource services based on feeding guilds, spawning guilds, etc. This is especially important because bottom-feeding species are particularly susceptible to effects associated with PAHs but supply modest ecological, human use, and cultural services.

As discussed previously, the observation of an adverse effect on free-ranging fish populations and communities does not necessarily indicate that the effect is due to PAHs. Therefore, it is essential to account for factors such as physical habitat quality, PAHs from other sources, and chemical stressors other than PAHs. In practice, regulators and natural resource trustees frequently disregard the various factors that are independent of the chemical release but may impact a natural resource, such as the fishery. However, such factors can be evaluated through a variety of forensic tools, such as biological response signature analysis (Yoder and Rankin, 1995), multivariate statistics (e.g., Norton et al., 2000), and correlation analysis (USEPA, 2000c).

Regarding wildlife that may consume fish from PAH-contaminated sites, common sense suggests that risks to piscivorous wildlife will be minimal because fish (and wildlife) metabolize PAHs. This chapter provides data to support this assumption, which may prove useful in limiting further evaluation of piscivorous wildlife in a quantitative risk assessment context. Alternatively, it may be practical to simply analyze and evaluate PAH concentrations in fish tissue, particularly if tissue samples at the site are already being analyzed for other chemicals of interest.

6.7 Summary and Conclusions

Many former MGP sites are located near or adjacent to rivers, streams, impoundments, lakes, or coastal environments. The presence of PAHs in sediment adjacent to these former MGP sites can trigger the need to assess potential adverse effects on aquatic ecosystems. The assessment of sediment quality in terms of protecting sediment-dwelling organisms is well understood. The assessment of PAH-related risks to fish and aquatic feeding wildlife (birds and mammals) is less thoroughly understood.

Fish metabolize PAHs and excrete the more water-soluble metabolites through the digestive system. It is difficult to document exposure, much less an adverse effect based on chemical analyses of biological tissue. Biomarkers, such as MFO, bile metabolites, and DNA adducts,

have been developed as sensitive, low cost methods to measure exposure to PAHs. No cause and effect relationship between biomarker response and an ecologically relevant effect has been quantitatively documented, however. PAHs have been found to cause lesions, tumors, and reproductive effects in the laboratory, but there is currently no reliable analytical tool to predict the probability of these effects in free ranging fish populations.

The most promising approach to assess risks to fish is a model based on the bioavailability of PAHs in sediment. PAHs tend to accumulate in sediment, and bottom-dwelling fish species are the most susceptible to the effects of PAHs. Some research has been conducted to support the development of sediment benchmarks that are protective of fish. Although promising, more research is needed to ensure sediment concentrations are protective of long-term exposure to PAHs in sediment and the development of cancerous lesions and tumors in free-ranging fish populations. Fish and invertebrates are affected at lower PAH concentrations than piscivorous wildlife; therefore, the assessment of ecological health at former MGP sites will be greatly simplified when fish can be adequately addressed by evaluating PAH concentrations in sediment.

Allegations of injury to fish populations and communities associated with PAHs can be effectively and efficiently addressed by using direct measures of fish community quality and health, such as the IBI. This approach has the advantage of integrating any effects on fish reproduction and survival (both a function of fish health). However, it is essential to account for potential effects due to physical habitat quality, PAHs from other sites, and other chemical stressors.

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Attachment 3

Oregon DEQ's Comments on Revision 1 of the Johnson Lake Investigation Work Plan, dated August 20, 2003

Johnson Lake Remedial Investigation Work Plan
Issues for discussion – August 20, 2003
Jennifer Sutter, DEQ Project Manager

1. Response to comment 1 suggests that an upland investigation will only be necessary if sediment data in Johnson Lake indicate a problem. It should be clarified that upland investigation has already been determined to be warranted based on historical information and upland site data. A No Further Action determination cannot be provided for the site until these issues are addressed.

2. The 4th bullet on page 4 indicates that PCBs were detected upgradient but does not document that PCBs were also detected in sediment samples from Johnson Lake. Also, the sample identified as having the highest PCB concentration, SD-07, is more cross-gradient to the site than upgradient. Finally, the Figure that shows the locations of these samples as well as other historical samples from the lake and surrounding Slough water bodies (Figure 2-2 in previous draft) appears to have been removed from the work plan. This Figure should remain in the work plan.

3. Petroleum hydrocarbons need to be considered a CPOC based on historical evidence of sheens on the lake.

4. It is stated on page 6 that the specific assumptions used to derive the bioaccumulation screening values are not documented. These assumptions are documented in the references to the draft screening level table provided by DEQ.

5. Also on page 6, it is stated that baseline metal concentrations reflect natural background concentrations. This is not always the case. Some of the baseline metal values reflect urban run-off issues.

6. At the bottom of page 6 it is stated that the basis for the baseline values has not been provided. We thought we had provided a copy of the background report on this evaluation to Keely O'Brien. Another copy can be sent.

7. It is stated that DEQ has not identified a baseline value for butylbenzyl phthalate or thallium; however, we do have values of 151 ppb and .58 ppm respectively. These values were not, however, included on the screening table for the Slough.

8. The detections of PCE and toluene in SLRA sediment samples should be discussed in section 3.1. While they were not detected at concentrations that exceed sediment screening levels they may be indicative of a groundwater contamination issue at the site. This could be deferred to the upland investigation.

9. The reference to DMEF bioaccumulation trigger values for PAHs on page 14 should be removed. These levels are currently being re-evaluated and their technical basis is questionable. However, PAHs need not be considered a

bioaccumulative contaminant of concern based on the fact that they were generally not detected in fish tissue samples from the Slough and in the very few samples where they were detected, the concentrations were generally below acceptable tissue levels.

10. While surface water sampling may not be useful due to the laboratory MRLs generally being higher than concentrations of concern, exposures via surface water should still be addressed either by modeling expected concentrations considering detected levels in sediment or providing justification, in terms of other pathways being the risk drivers, for not quantitatively addressing this pathway.

11. Petroleum hydrocarbons should be analyzed for in sediment samples to help in interpretation of bioassay results. Analysis for petroleum constituents only does not consider potential risk from exposure to the majority of chemicals in the free-phase product that are not represented in VOC or PAH analysis. DEQ currently evaluates risk from the major toxic components of petroleum hydrocarbons but this evaluation procedure cannot be used to evaluate risks from the majority of the compounds in petroleum mixtures. Petroleum hydrocarbons consist primarily of a mixture of aliphatic and aromatic compounds, only a portion of which are represented in VOC and PAH analysis. TPH has been found to be a likely source of toxicity at other sediment sites (e.g., Port of Portland Terminal 4) and there is clearly a potential source of TPH at this site.

12. The first bullet in Section 3.2.2 indicates that PCB homologue analysis will be conducted. While we agree, as noted in your response to our first comment on this issue, that there is considerable uncertainty with risk evaluations based on PCB congeners, this is the current direction that risk assessment for this group of compounds in headed. A fallback may be to conduct Aroclor analysis with more limited congener analysis where Aroclors are not detected. We continue to recommend congener analysis of tissue samples.

13. Lead was elevated in one of the sediment samples collected from Johnson Lake and has also been detected in fish tissue in the Slough at levels of concern for human ingestion of fish. Consequently, it should be included in the list of analytes for fish tissue samples provided in Section 3.2.3.

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Attachment 4

ARCADIS's Response to the Oregon DEQ's August 2003 Comments on Revision 1 of the Johnson Lake Investigation Work Plan

Response to August 20, 2003 Issues for Discussion, Johnson Lake Remedial Investigation Work Plan, Revision 1

Comments are provided in italics, followed by ARCADIS' response. These responses are intended to serve as an addendum to the work plan.

1. *Response to comment 1 suggests that an upland investigation will only be necessary if sediment data in Johnson Lake indicate a problem. It should be clarified that upland investigation has already been determined to be warranted based on historical information and upland site data. A No Further Action determination cannot be provided for the site until these issues are addressed.*

> Owens Illinois has conducted a series of investigations to determine soil quality adjacent to the transformer yard and all 6 stormwater outfalls. The scope and necessity for any additional on-site investigations will be determined after the completion of the Johnson Lake investigation.

2. *The 4th bullet on page 4 indicates that PCBs were detected upgradient but does not document that PCBs were also detected in sediment samples from Johnson Lake. Also, the sample identified as having the highest PCB concentration, SD-07, is more cross-gradient to the site than upgradient. Finally, the Figure that shows the locations of these samples as well as other historical samples from the lake and surrounding Slough water bodies (Figure 2-2 in previous draft) appears to have been removed from the work plan. This Figure should remain in the work plan.*

> PCBs were detected at locations upgradient, cross-gradient, and downgradient from Johnson Lake, as well as within Johnson Lake adjacent to the Meyers outfall. Sediment was not collected adjacent to the Owens-Brockway facility during the sampling event in question. The sample location figure is attached as Figure 2-2 (from the original Johnson Lake Investigation Work Plan, dated April 10, 2003).

3. *Petroleum hydrocarbons need to be considered a CPOC based on historical evidence of sheens on the lake.*

> Total petroleum hydrocarbons (TPH) will be analyzed in sediment, but not in biota tissue. Revised Tables B-1 and B-2 are attached and provide relevant sampling and analytical information for the TPH analyses.

4. *It is stated on page 6 that the specific assumptions used to derive the bioaccumulation screening values are not documented. These assumptions are documented in the references to the draft screening level table provided by DEQ.*

The screening level table references the equations used to derive bioaccumulation screening values. However, these equations require chemical-specific values for several parameters, including a no observed adverse effect level (NOAEL) for each wildlife species (derivation of which may involve use of a body-weight extrapolation factor), a bioaccumulation factor (BAF), and an octanol-water partition coefficient (K_{ow}), as well as parameter values specific to the wildlife species (water and food ingestion rates). While values for each of these parameters are listed in ORNL TM-86/R3 (the source of the previously cited equations), we are not able to reproduce the Oregon DEQ screening values using these ORNL-listed parameter values. Alternative parameter values may have been used but are not documented. Thus, it would be accurate to state that the chemical-specific and species-specific information used to derive the bioaccumulation screening values is not documented.

5. *Also on page 6, it is stated that baseline metal concentrations reflect natural background concentrations. This is not always the case. Some of the baseline metal values reflect urban run-off issues.*

The statement is corrected as follows: "The Columbia Slough baseline values are intended to represent naturally occurring concentrations of metals and/or ubiquitous concentrations of anthropogenic metals and organic chemicals in sediment."

6. *At the bottom of page 6 it is stated that the basis for the baseline values has not been provided. We thought we had provided a copy of the background report on this evaluation to Keely O'Brien. Another copy can be sent.*

ARCADIS has now received the text portion of the "Columbia Slough – Baseline Concentrations, Documentation of Methodology," dated November 21, 2002.

7. *It is stated that DEQ has not identified a baseline value for butylbenzyl phthalate or thallium; however, we do have values of 151 ppb and .58 ppm respectively. These values were not, however, included on the screening table for the Slough.*

The statement that DEQ had not identified baseline values for butylbenzyl phthalate or thallium was incorrect. The work plan should have stated that the maximum detected concentrations of these constituents exceed DEQ's baseline values. The conclusions of the screening evaluation are not affected by this change.

8. *The detections of PCE and toluene in SLRA sediment samples should be discussed in section 3.1. While they were not detected at concentrations that exceed sediment screening levels they may be indicative of a groundwater contamination issue at the site. This could be deferred to the upland investigation.*

In addition to their detection in sediment, toluene and PCE were the only organic chemicals detected in recent analyses of surface water collected from Johnson Lake (see response to Comment 10). The presence of toluene and PCE is most likely due to one or more off-site sources, as described below.

Surface water sample locations and toluene concentrations in Johnson Lake are shown in the attached Figure 1. The spatial distribution of toluene concentrations suggests that toluene is entering the lake from the north, rather than from the Owens-Brockway site. Additionally, although toluene is a component of petroleum hydrocarbons, other characteristic petroleum hydrocarbon chemicals (e.g., benzene, ethylbenzene, xylenes) are not present at elevated concentrations in Johnson Lake sediment or surface water. This suggests an off-site source related specifically to toluene use rather than petroleum hydrocarbons.

Concentrations of PCE in surface water were similar throughout Johnson Lake. Given that Owens-Brockway did not use PCE in its operations, it is likely that the PCE in Johnson Lake also is related to off-site sources.

Based on the presence of toluene and PCE in surface water, it appears there may be one or more off-site sources of contaminated groundwater impacting Johnson Lake. A preliminary review of environmental databases indicates several potentially responsible parties including, but not limited to, Cadet Manufacturing, Oregon Fir Supply Company, United Medical Laboratory/Drum Recovery. Other sources may exist north of Johnson Lake along Colfax Avenue that are currently unidentified.

9. *The reference to DMEF bioaccumulation trigger values for PAHs on page 14 should be removed. These levels are currently being re-evaluated and their technical basis is questionable. However, PAHs need not be considered a bioaccumulative contaminant of concern based on the fact that they were generally not detected in fish tissue samples from the Slough and in the very few samples where they were detected, the concentrations were generally below acceptable tissue levels.*

As requested, we will not rely on the DMEF bioaccumulation trigger values. PAHs will be analyzed in sediment but not in biota tissue, for the reasons described in this comment.

10. *While surface water sampling may not be useful due to the laboratory MRLs generally being higher than concentrations of concern, exposures via surface water should still be addressed either by modeling expected concentrations considering detected levels in sediment or providing justification, in terms of other pathways being the risk drivers, for not quantitatively addressing this pathway.*

Detection limits that exceed screening values for some chemicals, as well as temporal variability, are sources of uncertainty in interpreting surface water chemistry data. However, modeling chemical concentrations in surface water would also entail a high degree of uncertainty. Exposure to chemicals via ambient surface water is typically negligible for humans and wildlife receptors, relative to other exposure pathways. However, surface water exposures are potentially important for some receptors and chemicals (e.g., aquatic organisms exposed to metals). Therefore, Owens Illinois collected and analyzed surface water from Johnson Lake for screening purposes. The surface water sampling was conducted according the methods described in the original Johnson Lake Investigation Work Plan, dated April 10, 2003. Appendix A (attached) shows the analytical results and reporting limits, as well as Oregon DEQ criteria and screening values for comparison purposes.

No chemical concentrations detected in Johnson Lake surface water exceed any of the ecological criteria or screening values. Detected concentrations of arsenic and PCE exceed conservative human health criteria; however, these concentrations are well below federal drinking water standards and therefore are unlikely to pose a risk to human health. Further, the detected arsenic concentrations are consistent with naturally occurring background levels,[1] and PCE is likely unrelated to the Owens-Brockway site (see response to Comment 8). As such, no chemicals of interest are identified in surface water. Arsenic will be evaluated as a chemical of interest in sediment, however. Based on the surface water analytical results, chemical exposures via surface water will be considered negligible relative to other exposure pathways.

11. *Petroleum hydrocarbons should be analyzed for in sediment samples to help in interpretation of bioassay results. Analysis for petroleum constituents only does not consider potential risk from exposure to the majority of chemicals in the free-phase product that are not represented in VOC or PAH analysis. DEQ currently evaluates risk from the major toxic components of petroleum hydrocarbons but this evaluation procedure cannot be used to evaluate risks from the majority of the compounds in petroleum mixtures. Petroleum hydrocarbons consist primarily of a mixture of aliphatic and aromatic compounds, only a portion of which are represented in VOC and PAH analysis. TPH has been found to be a likely source of toxicity at other sediment sites (e.g., Port of Portland Terminal 4) and there is clearly a potential source of TPH at this site.*

Petroleum hydrocarbons will be analyzed (see response to Comment 3).

[1] Smedley, P.L. and D.G. Kinniburgh, 2002. A review of the source, behavior and distribution of arsenic in natural waters. Appl. Geochem. 17, 517-568.

12. *The first bullet in Section 3.2.2 indicates that PCB homologue analysis will be conducted. While we agree, as noted in your response to our first comment on this issue, that there is considerable uncertainty with risk evaluations based on PCB congeners, this is the current direction that risk assessment for this group of compounds in headed. A fallback may be to conduct Aroclor analysis with more limited congener analysis where Aroclors are not detected. We continue to recommend congener analysis of tissue samples.*

As agreed during discussions with the DEQ, limited sampling and analyses will be conducted for PCB congeners for comparison purposes. Specifically, three samples of each of the following types will be analyzed for PCB congeners: game fish fillets, medium whole fish, benthic invertebrates, and sediment. Sample locations for PCB congener analysis will be randomly selected from among the eight zones to be sampled for other analytical parameters.

The analysis of PCB congeners will be conducted using USEPA Method 8082. Target analytes will include the 12 congeners for which toxicity equivalence factors are available, namely PCBs 77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, and 189. Sampling and analytical information is included in the revised Tables B-1 and B-2, attached.

As detailed in the Johnson Lake Work Plan, Revision 1, Owens Illinois will collect and analyze sediment and biota tissue for PCB homologues using USEPA Method 680. A PCB homologue approach is more certain, more accurate, and preferable to a PCB congener-based approach. However, Owens Illinois will collect a limited PCB congener data set to quantitatively evaluate the uncertainty associated with the presence of certain PCB congeners that exhibit “dioxin-like” molecular structure, as requested by DEQ.

13. *Lead was elevated in one of the sediment samples collected from Johnson Lake and has also been detected in fish tissue in the Slough at levels of concern for human ingestion of fish. Consequently, it should be included in the list of analytes for fish tissue samples provided in Section 3.2.3.*

Biota tissue will be analyzed for lead. Sampling and analytical information is included in the revised Tables B-1 and B-2, attached.

ARCADIS

Table B-1 (Revised). Target Compounds, Analytical Methods, and Sampling Information, Owens-Brockway, Portland, Oregon

Target Compounds	Analytical Method	Containers, Preservatives, Holding Times Sediment	Tissue	Laboratory
PCB-Homologues	USEPA 680	250 mL glass jar 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	12 g wrapped in foil 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	STL Savannah Laboratories
PCB-Congeners	USEPA 8082	250 mL glass jar 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	12 g wrapped in foil 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	Columbia Analytical Services
Alkylated PAHs	USEPA 8270c	500 mL glass jar 4 degrees Celcius HT 14 days/ 40 days	NA	Columbia Analytical Services
Organochlorine Pesticides	USEPA 8081 (low level reporting limits)	250 mL glass jar 4 degrees Celcius HT 7 days/ 40 days	NA	Columbia Analytical Services
Organochlorine Pesticides	USEPA 8081 (low level reporting limits)	NA	12 g wrapped in foil (same sample as PCB-homologues) 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	Columbia Analytical Services
Total Metals	USEPA 6010	250 mL glass jar 4 degrees Celcius HT 6 months	NA	Columbia Analytical Services
Lead	USEPA 6010/200.8	NA	12 g wrapped in foil (same sample as PCB-homologues) 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	Columbia Analytical Services
Selenium	USEPA 7742	NA	12 g wrapped in foil (same sample as PCB-homologues) 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	Columbia Analytical Services
SEM/AVS[a]	Allen, Fu et al. 1992	500 mL widemouth glass jar, no headspace 4 degrees Celcius HT 14 days	NA	Columbia Analytical Services
Total Organic Carbon (TOC)	ASTM D4129-82M (sediment)	500 mL glass jar (same jar as alkylated PAHs) 4 degrees Celcius HT 14 days	NA	Columbia Analytical Services

Table B-1 (Revised). Target Compounds, Analytical Methods, and Sampling Information, Owens-Brockway, Portland, Oregon

Target Compounds	Analytical Method	Containers, Preservatives, Holding Times Sediment	Tissue	Laboratory
Total Petroleum Hydrocarbons (TPH)	TPH D (C-10 to C-34)	500 mL glass jar (same jar as alkylated PAHs) 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	NA	Columbia Analytical Services
Grain Size	ASTM D422	500 mL glass jar NA NA	NA	Columbia Analytical Services
Lipids	USEPA OB1090	NA	12 g wrapped in foil (same sample as PCB-homologues) 4 degrees Celcius HT 7 days until extraction, 40 days to analysis	Columbia Analytical Services
Moisture Content	ASTM D2216	500 mL plastic NA HT 7 days	NA	Columbia Analytical Services
Porewater Extraction[b]	Hoffman 1998	2 Liters plastic 4 degrees Celcius HT 14 days	NA	Columbia Analyical Services
10-Day Toxicity Test w/ *Hyalella azteca*	USEPA 100.1	3.5 Liters 4 degrees Celcius HT 14 days	NA	Springborn Smithers Laboratories
10-Day Toxicity Test w/ *Chironomus tentans*	USEPA 100.2	3.5 Liters 4 degrees Celcius HT 14 days	NA	Springborn Smithers Laboratories

a. SEM metals = cadmium, copper, lead, nickel, silver, and zinc
b. The following analyses will be performed on sediment porewater: dissolved arsenic (USEPA 200.8), dissolved selenium (USEPA 200.8), ammonia (USEPA 350.2), and porewater pH (USEPA 150.1).

ASTM - American Society for Testing and Materials
AVS - acid volatile sulfide
g - grams
HT - holding time to extraction/holding time for the extract preparation
M - modified
mL - milliliters
NA - not applicable/available
PAHs - polycyclic aromatic hydrocarbons
PCBs - polychlorinated biphenyls
SEM - simultaneously extracted metals
USEPA - United States Environmental Protection Agency

References:

Allen, H.E., G. Fu, and B. Deng. 1992. Analysis of acid-volatile sulfide (AVS) and simultaneously extracted metals (SEM) for the estimation of potential toxicity in aquatic sediments. Environmental Toxicology and Chemistry 12:1441-1453.

Hoffman, E. 1998. Tributyltin analysis: Clarification of interstitial water extraction and analysis methods – interim. DMMP clarification paper/SMS Technical information memorandum. U.S. Army Corps of Engineers.

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Table B-2 (Revised). Laboratory- Specific Precision and Accuracy Control Limits for Analytical Parameters, Owens-Brockway, Portland, Oregon

Parameters	Analytical Methods	MDL	RL	Units	Accuracy % Recovery	Precision % RPD
Sediment						
PCB-Homologues						
Monochlorobiphenyl	USEPA 680	0.68	3.3	µg/kg	30-130	50
Dichlorobiphenyl	USEPA 680	0.76	3.3	µg/kg	30-130	50
Trichlorobiphenyl	USEPA 680	0.68	3.3	µg/kg	30-130	50
Tetrachlorobiphenyl	USEPA 680	1.3	6.7	µg/kg	40-140	50
Pentachlorobiphenyl	USEPA 680	0.83	6.7	µg/kg	40-140	50
Hexachlorobiphenyl	USEPA 680	0.89	6.7	µg/kg	40-140	50
Heptachlorobiphenyl	USEPA 680	1.6	10	µg/kg	40-140	50
Octachlorobiphenyl	USEPA 680	0.95	10	µg/kg	40-140	50
Nonachlorobiphenyl	USEPA 680	1.9	17	µg/kg	30-130	50
Decachlorobiphenyl	USEPA 680	1.9	17	µg/kg	30-130	50
PCB Congeners						
PCB 77	USEPA 8082	0.05	0.5	µg/kg, dry wt.	60-130	40
PCB 81	USEPA 8082	0.1	0.5	µg/kg, dry wt.	60-130	40
PCB 105	USEPA 8082	0.09	0.5	µg/kg, dry wt.	60-130	40
PCB 114	USEPA 8082	0.3	0.7	µg/kg, dry wt.	60-130	40
PCB 118	USEPA 8082	0.04	0.5	µg/kg, dry wt.	60-130	40
PCB 123	USEPA 8082	0.08	0.5	µg/kg, dry wt.	60-130	40
PCB 126	USEPA 8082	0.1	0.5	µg/kg, dry wt.	60-130	40
PCB 156	USEPA 8082	0.09	0.5	µg/kg, dry wt.	60-130	40
PCB 157	USEPA 8082	0.2	0.5	µg/kg, dry wt.	60-130	40
PCB 167	USEPA 8082	0.3	0.7	µg/kg, dry wt.	60-130	40
PCB 169	USEPA 8082	0.1	0.5	µg/kg, dry wt.	60-130	40
PCB 189	USEPA 8082	0.09	0.5	µg/kg, dry wt.	60-130	40
Alkylated PAHs	USEPA 8270c	0.08-0.6	5	µg/kg, dry wt.	45-128	40
Organochlorine Pesticides	USEPA 8081 (LL)	0.3-2	5	µg/kg, dry wt.	42-150	40
Total Metals	USEPA 6010	0.004-0.2	0.02-1	mg/kg, dry wt.	70-130	30
SEM/AVS	Allen, Fu et al. 1992	Method	Method	mg/kg, dry wt.	50-150	20
Total Petroleum Hydrocarbons	TPH D (C-10 to C-34)	4	25	mg/kg, dry wt.	63-133	40
Total Organic Carbon (TOC)	ASTM D4129-82M	0.02	0.05	Percent	85-115	20
Grain Size	ASTM D422	NA	NA	Percent	NA	20
Moisture Content	ASTM D2216	NA	NA	Percent	NA	20
Sediment Porewater						
Porewater Extraction	Hoffman 1998	NA	NA	NA	NA	NA
Selenium-dissolved	USEPA 200.8	0.80	1	µg/L	85-115	20
Arsenic-dissolved	USEPA 200.8	0.20	0.5	µg/L	85-115	20
Ammonia	USEPA 350.3	0.03	0.05	mg/L	85-115	20
pH	USEPA 150.1	NA	NA	standard units	NA	NA

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Table B-2 (Revised). Laboratory- Specific Precision and Accuracy Control Limits for Analytical Parameters, Owens-Brockway, Portland, Oregon

Parameters	Analytical Methods	MDL	RL	Units	Accuracy % Recovery	Precision % RPD
Biota						
PCB-Homologues						
Monochlorobiphenyl	USEPA 680	0.68	3.3	µg/kg	30-130	50
Dichlorobiphenyl	USEPA 680	0.76	3.3	µg/kg	30-130	50
Trichlorobiphenyl	USEPA 680	0.68	3.3	µg/kg	30-130	50
Tetrachlorobiphenyl	USEPA 680	1.3	6.7	µg/kg	40-140	50
Pentachlorobiphenyl	USEPA 680	0.83	6.7	µg/kg	40-140	50
Hexachlorobiphenyl	USEPA 680	0.89	6.7	µg/kg	40-140	50
Heptachlorobiphenyl	USEPA 680	1.6	10	µg/kg	40-140	50
Octachlorobiphenyl	USEPA 680	0.95	10	µg/kg	40-140	50
Nonachlorobiphenyl	USEPA 680	1.9	17	µg/kg	30-130	50
Decachlorobiphenyl	USEPA 680	1.9	17	µg/kg	30-130	50
PCB Congeners						
PCB 77	USEPA 8082	0.3	0.5	µg/kg, wet wt.	60-130	40
PCB 81	USEPA 8082			µg/kg, wet wt.	60-130	40
PCB 105	USEPA 8082	0.2	0.5	µg/kg, wet wt.	60-130	40
PCB 114	USEPA 8082	0.2	0.7	µg/kg, wet wt.	60-130	40
PCB 118	USEPA 8082	0.2	0.5	µg/kg, wet wt.	60-130	40
PCB 123	USEPA 8082	0.2	0.5	µg/kg, wet wt.	60-130	40
PCB 126	USEPA 8082	0.2	0.5	µg/kg, wet wt.	60-130	40
PCB 156	USEPA 8082	0.2	0.5	µg/kg, wet wt.	60-130	40
PCB 157	USEPA 8082	0.2	0.5	µg/kg, wet wt.	60-130	40
PCB 167	USEPA 8082	0.2	0.7	µg/kg, wet wt.	60-130	40
PCB 169	USEPA 8082	0.2	0.5	µg/kg, wet wt.	60-130	40
PCB 189	USEPA 8082	0.1	0.5	µg/kg, wet wt.	60-130	40
Organochlorine Pesticides	USEPA 8081 (LL)	0.1-0.4	1	µg/kg, wet wt.	70-130	40
Lead	USEPA 200.8	0.004	0.02	mg/kg, dry wt.	70-130	30
Selenium	USEPA 7742	0.05	0.1	mg/kg, dry wt.	70-130	30
Lipid Content	USEPA OB1090	NA	NA	percent	NA	NA

ASTM - American Society for Testing and Materials
AVS - acid volatile sulfide
LL - low level reporting limits
M - modified
MDL - method detection limit
mg/kg - milligrams per kilogram
mg/L - milligrams per liter
NA - not applicable/available
PAHs - polycyclic aromatic hydrocarbons
PCBs - polychlorinated biphenyls
RL - reporting limit
RPD - relative percent difference
SEM - simultaneously extracted metals
µg/kg - micrograms per kilogram
µg/L - micrograms per liter
USEPA - United States Environmental Protection Agency

References:

Allen, H.E., G. Fu, and B. Deng. 1992. Analysis of acid-volatile sulfide (AVS) and simultaneously extracted metals (SEM) for the estimation of potential toxicity in aquatic sediments. Environmental Toxicology and Chemistry 12:1441-1453.

Hoffman, E. 1998. Tributyltin analysis: Clarification of interstitial water extraction and analysis methods – interim. DMMP clarification paper/SMS Technical information memorandum. U.S. Army Corps of Engineers.

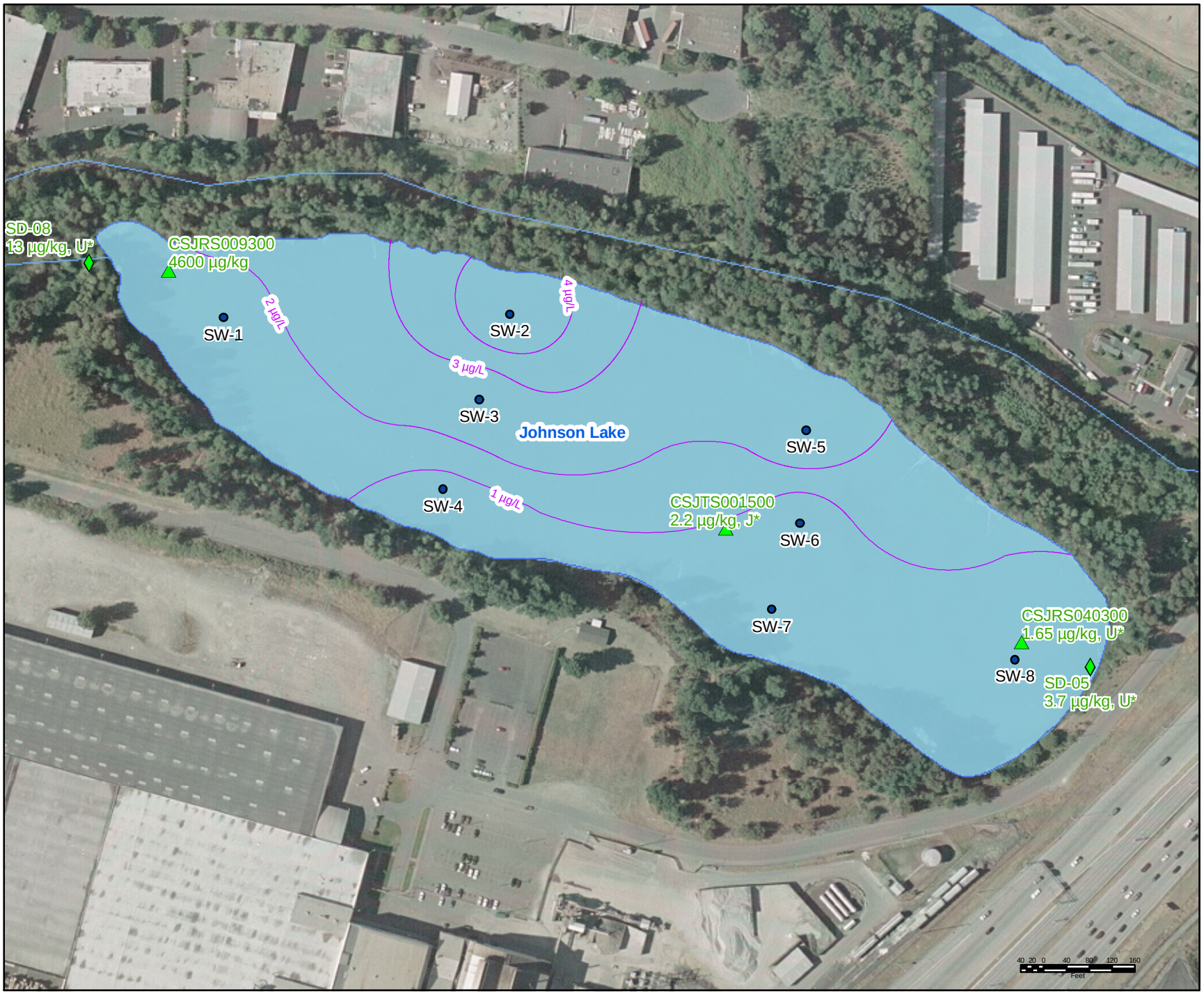


Figure 1.

Surface Water Sampling Locations and Toluene Concentrations Johnson Lake Portland, Oregon

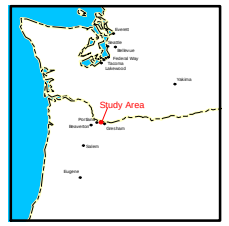
Legend

- Sediment Samples (2003)
- Sample Locations
- Sediment Samples (1/1/1994)
- Toluene Concentration
- River Line
- River Fill

Basedata from METRO DRC 1"=100' orthophotos, City of Portland, and US Census Bureau 2000 Tiger Files. Positions may vary in accuracy due to scale differences between the data sets. Spatial information is meant for general reference only.

Lambert Conformal Conic projection, North American Datum 1983 HARN, Local coordinate grid State Plane Oregon (North) FIPS 3601.

Cartography by ARCADIS - Greenville, SC



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Appendix A

Surface Water Data

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Appendix A. Summary of Polychlorinated Biphenyls Aroclors in Surface Water, Johnson Lake, Portland, Oregon

Constituent	Surface Water Criteria or Screening Values				Units	Reporting Limit	Sample Location							
	Human Health[a]		Ecological											
	Water and Fish Ingestion	Fish Consumption Only	Aquatic Life[b]	Wildlife[c]			SW-1	SW-2	SW-3	SW-4	SW-5	SW-6	SW-7	SW-8
PCB (total)	0.000079	0.000079	0.014	-										
Aroclor 1016	-	-	-	13000	µg/L	0.02	ND	ND	ND	ND	ND	ND	ND	ND
Aroclor 1221	-	-	0.28	-	µg/L	0.04	ND	ND	ND	ND	ND	ND	ND	ND
Aroclor 1232	-	-	0.58	-	µg/L	0.02	ND	ND	ND	ND	ND	ND	ND	ND
Aroclor 1242	-	-	0.053	700	µg/L	0.02	ND	ND	ND	ND	ND	ND	ND	ND
Aroclor 1248	-	-	0.081	-	µg/L	0.02	ND	ND	ND	ND	ND	ND	ND	ND
Aroclor 1254	-	-	0.033	300	µg/L	0.02	ND	ND	ND	ND	ND	ND	ND	ND
Aroclor 1260	-	-	94	-	µg/L	0.02	ND	ND	ND	ND	ND	ND	ND	ND

µg/L - micrograms per liter

- - criterion not available

ND - not detected at or above reporting limit

a. values represent Oregon DEQ water quality criteria if available, or USEPA (2002) National Recommended water Quality Criteria

b. values represent Oregon DEQ water qualtiy criteria if available, or aquatic life screening values from Oregon DEQ (1998) Ecological Risk Guidance

c. values represent the lower of avian and mammalian wildlife screening values from Oregon DEQ (1998) Ecological Risk Guidance

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Appendix A. Summary of Pesticides in Surface Water, Johnson Lake, Portland, Oregon

Constituent	Surface Water Criteria or Screening Values				Units	Reporting Limit	Sample Location							
	Human Health[a]		Ecological											
	Water and Fish Ingestion	Fish Consumption Only	Aquatic Life[b]	Wildlife[c]			SW-1	SW-2	SW-3	SW-4	SW-5	SW-6	SW-7	SW-8
4,4'-DDD	0.00031	0.00031	0.001	20	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
4,4'-DDE	0.00022	0.00022	-	20	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
4,4'-DDT	0.000024	0.000024	0.001	20	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
Aldrin	0.000074	0.000079	0.06	1,500	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
alpha-BHC	0.0026	0.0049	2.2	-	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
alpha-Chlordane[d]	0.00046	0.00048	0.0043	15500	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
beta-BHC	0.0091	0.017	2.2	-	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
delta-BHC	-	-	-	-	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
Dieldrin	0.000071	0.000076	0.0019	150	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
Endosulfan I[e]	74	159	0.056	1000	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
Endosulfan II[e]	74	159	0.056	1000	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
Endosulfan Sulfate	62	89	-	-	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
Endrin	1.0	0.81	0.0023	70	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
Endrin Aldehyde	0.29	0.3	-	-	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
Endrin Ketone	-	-	-	-	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
gamma-BHC (Lindane)	0.019	0.063	0.08	14500	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
gamma-Chlordane[d]	0.00046	0.00048	0.0043	15500	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
Heptachlor	0.00028	0.00029	0.0038	2000	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
Heptachlor Epoxide	0.000039	0.000039	0.0038	-	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
Methoxychlor	100	-	0.0300	30000	µg/L	0.01	ND	ND	ND	ND	ND	ND	ND	ND
Toxaphene	0.00071	0.00073	0.0002	60000	µg/L	0.05	ND	ND	ND	ND	ND	ND	ND	ND

µg/L - micrograms per liter

- - criterion not available

ND - not detected at or above reporting limit

a. values represent Oregon DEQ water quality criteria if available, or USEPA (2002) National Recommended water Quality Criteria

b. values represent Oregon DEQ water qualtiy criteria if available, or aquatic life screening values from Oregon DEQ (1998) Ecological Risk Guidance

c. values represent the lower of avian and mammalian wildlife screening values from Oregon DEQ (1998) Ecological Risk Guidance

d - criteria are for chlordane

e - criteria are for endosulfan

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Appendix A. Summary of Dissolved Metals in Surface Water, Johnson Lake, Portland, Oregon

Constituent	Surface Water Criteria or Screening Values				Units	Reporting Limit	Sample Location							
	Human Health[a]		Ecological											
	Water and Fish Ingestion	Fish Consumption Only	Aquatic Life[b]	Wildlife[c]			SW-1	SW-2	SW-3	SW-4	SW-5	SW-6	SW-7	SW-8
Antimony	146	45,000	1,600	1,000	µg/L	0.05	0.44	0.33	0.27	0.18	0.1	0.34	0.37	0.09
Arsenic (III/V)	0.0022	0.018	150/150	6,000/ -	µg/L	0.5	1.1	1.2	1.1	1.1	1.1	1.1	1.1	1
Beryllium	0.0068	0.12	5.3	5,000	µg/L	0.02	0.03	ND	ND	ND	ND	ND	ND	ND
Cadmium	10	-	1.1	8,000	µg/L	0.02	0.05	0.03	0.03	ND	ND	ND	ND	0.04
Chromium (III/VI)	170,000/50	3,433,000/100	210/11	7,200 (Cr III)	µg/L	0.2	0.6	0.6	0.4	0.3	0.5	0.4	0.3	0.4
Copper	1,300	-	12	53,000	µg/L	0.1	1.2	1.1	0.6	0.5	0.6	0.9	0.9	0.7
Lead	50	-	3.2	28,000	µg/L	0.02	0.12	0.68	0.12	0.1	0.12	0.04	0.06	0.1
Mecury	0.14	0.15	0.012	3,300	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Nickel	13	100	160	38,000	µg/L	0.2	0.6	0.7	0.4	0.4	0.7	0.5	0.6	0.9
Selenium	10	4,200	35	1,500	µg/L	1	ND	ND	ND	ND	ND	ND	ND	ND
Silver	50	-	0.12	-	µg/L	0.02	0.03	ND	ND	ND	ND	ND	ND	ND
Thallium	13	48	40	60	µg/L	0.02	0.03	ND	ND	ND	ND	ND	ND	ND
Zinc	7,400	26,000	110	105,000	µg/L	0.50	2.2	3.4	ND	0.8	0.7	0.8	0.8	0.7
Hardness as CaCO3	-	-	-	-	µg/L	10000	86000	86000	87000	85000	84000	86000	86000	86000

µg/L - micrograms per liter

- - criterion not available

ND - not detected at or above reporting limit

CaCO3 - calcium carbonate

a. values represent Oregon DEQ water quality criteria if available, or USEPA (2002) National Recommended water Quality Criteria

b. values represent Oregon DEQ water qualtiy criteria if available, or aquatic life screening values from Oregon DEQ (1998) Ecological Risk Guidance

c. values represent the lower of avian and mammalian wildlife screening values from Oregon DEQ (1998) Ecological Risk Guidance

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Appendix A. Summary of Volatile Organic Compounds (VOC) in Surface Water, Johnson Lake, Oregon

Constituent	Surface Water Criteria or Screening Values				Units	Reporting Limit	Sample Location							
	Human Health[a]		Ecological											
	Water and Fish Ingestion	Fish Consumption Only	Aquatic Life[b]	Wildlife[c]			SW-1	SW-2	SW-3	SW-4	SW-5	SW-6	SW-7	SW-8
1,1,1,2-Tetrachloroethane	-	-	186	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
1,1,1-Trichloroethane (TCA)	18,400	103,000	11	4,000,000	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
1,1,2,2-Tetrachloroethane	0.17	11	2,400	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
1,1,2-Trichloroethane	0.6	42	9,400	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
1,1-Dichloroethane (1,1-DCA)	-	-	47	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
1,1-Dichloroethene (1,1-DCE)	0.057	3.2	25	230,000	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
1,1-Dichloropropene	-	-	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
1,2,3-Trichlorobenzene	-	-	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
1,2,3-Trichloropropane	-	-	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
1,2,4-Trichlorobenzene	260	940	110	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
1,2,4-Trimethylbenzene	-	-	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
1,2-Dibromo-3-chloropropane (DBCP)	-	-	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
1,2-Dibromoethane (EDB)	-	-	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
1,2-Dichlorobenzene	2700	17000	14	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
1,2-Dichloroethane (EDC)	0.94	243	20,000	125,000	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
1,2-Dichloropropane	0.5	15	5700	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
1,3,5-Trimethylbenzene	-	-	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
1,3-Dichlorobenzene	320	960	71	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
1,3-Dichloropropane	-	-	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
1,4-Dichlorobenzene	400	2600	15	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
2,2-Dichloropropane	-	-	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
2-Butanone (MEK)	-	-	-	14,000,000	µg/L	20	ND	ND	ND	ND	ND	ND	ND	ND
2-Chlorotoluene	-	-	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
2-Hexanone	-	-	99	-	µg/L	20	ND	ND	ND	ND	ND	ND	ND	ND
4-Chlorotoluene	-	-	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
4-Isopropyltoluene	-	-	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
4-Methyl-2-pentanone (MIBK)	-	-	170	-	µg/L	20	ND	ND	ND	ND	ND	ND	ND	ND
Acetone	-	-	1500	76,000	µg/L	20	ND	ND	ND	ND	ND	ND	ND	ND
Benzene	0.66	40	130	200,000	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Bromobenzene	-	-	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
Bromochloromethane	-	-	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Bromodichloromethane	0.55	17	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Bromoform	4.3	140	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Bromomethane (methyl bromide)	47	1500	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Carbon Disulfide	-	-	0.92	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Carbon Tetrachloride	0.40	6.94	74	123,000	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND

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Appendix A. Summary of Volatile Organic Compounds (VOC) in Surface Water, Johnson Lake, Oregon

Constituent	Surface Water Criteria or Screening Values				Units	Reporting Limit	Sample Location							
	Human Health[a]		Ecological											
	Water and Fish Ingestion	Fish Consumption Only	Aquatic Life[b]	Wildlife[c]			SW-1	SW-2	SW-3	SW-4	SW-5	SW-6	SW-7	SW-8
Chlorobenzene	488	21000	50	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Chloroethane (methyl chloride)	-	-	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Chloroform	0.19	16	1240	115,000	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Chloromethane	-	-	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
cis-1,2-Dichloroethene	-	-	590	180,000	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
cis-1,3-Dichloropropene[c]	10	1700	244	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Dibromochloromethane	0.4	13	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Dibromomethane	-	-	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Dichlorodifluoromethane (CFC 12)	-	-	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Dichloromethane (Methylene Chloride)	4.6	590	2200	45,000	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
Ethylbenzene	1,400	3,280	7.3	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Hexachlorobutadiene	0.45	50	9.3	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
Isopropylbenzene	-	-	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
m,p-Xylenes	-	-	13	8,000	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Naphthalene	-	-	620	284,000	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
n-Butylbenzene	-	-	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
n-Propylbenzene	-	-	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
o-Xylene	-	-	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
sec-Butylbenzene	-	-	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
Styrene	-	-	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
tert-Butylbenzene	-	-	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
Tetrachloroethene (PCE)	0.8	8.85	840	6,000	µg/L	0.5	0.88	0.87	0.87	0.82	0.77	0.78	0.83	0.73
Toluene	14,300	424,000	9.8	104,000	µg/L	0.5	1.9	5	2.6	0.86	2.8	0.54	0.53	0.64
trans-1,2-Dichloroethene	700	140,000	590	180	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
trans-1,3-Dichloropropene[d]	10	1,700	244	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Trichloroethene (TCE)	2.7	80.7	21900	3,000	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Trichlorofluoromethane (CFC 11)	-	-	-	-	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND
Vinyl Chloride	2	525	-	1300	µg/L	0.5	ND	ND	ND	ND	ND	ND	ND	ND

µg/L - micrograms per liter

- - criterion not available

ND - not detected at or above reporting limit

a. values represent Oregon DEQ water quality criteria if available, or USEPA (2002) National Recommended water Quality Criteria

b. values represent Oregon DEQ water qualtiy criteria if available, or aquatic life screening values from Oregon DEQ (1998) Ecological Risk Guidance

c. values represent the lower of avian and mammalian wildlife screening values from Oregon DEQ (1998) Ecological Risk Guidance

d. criteria are for 1,3-Dichloropropene

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Appendix A. Summary of Semi-Volatile Organic Compounds in Surface Water, Johnson Lake,Portland, Oregon

Constituents	Surface Water Criteria or Screening Values				Units	Reporting Limit	Sample locations							
	Human Health[a]		Ecological											
	Water and Fish Ingestion	Fish Consumption Only	Aquatic Life[b]	Wildlife[c]			SW-1	SW-2	SW-3	SW-4	SW-5	SW-6	SW-7	SW-8
1,2,4-Trichlorobenzene	260	940	110	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
1,2-Dichlorobenzene	2700	17000	14	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
1,3-Dichlorobenzene	320	960	71	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
1,4-Dichlorobenzene	400	2600	15	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
2,4,5-Trichlorophenol	2,600	3600	-	-	µg/L	0.49	ND	ND	ND	ND	ND	ND	ND	ND
2,4,6-Trichlorophenol	1.2	3.6	970	-	µg/L	0.49	ND	ND	ND	ND	ND	ND	ND	ND
2,4-Dichlorophenol	3,090	290	365	-	µg/L	0.49	ND	ND	ND	ND	ND	ND	ND	ND
2,4-Dimethylphenol	380	850	42	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
2,4-Dinitrophenol	69	5300	-	-	µg/L	3.9	ND	ND	ND	ND	ND	ND	ND	ND
2,4-Dinitrotoluene	0	9	230	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
2,6-Dinitrotoluene	-	-	230	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
2-Chloronaphthalene (beta)	1000	1600	32	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
2-Chlorophenol	81	150	2000	-	µg/L	0.49	ND	ND	ND	ND	ND	ND	ND	ND
2-Methylnaphthalene	-	-	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
2-Methylphenol	-	-	13	2,200,000	µg/L	0.49	ND	ND	ND	ND	ND	ND	ND	ND
2-Nitroaniline	-	-	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
2-Nitrophenol	-	-	-	-	µg/L	0.49	ND	ND	ND	ND	ND	ND	ND	ND
3,3'-Dichlorobenzidine	0.021	0.028	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
3-Nitroaniline	-	-	-	-	µg/L	0.98	ND	ND	ND	ND	ND	ND	ND	ND
4,6-Dinitro-2-methylphenol	13	280	-	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
4-Bromophenyl Phenyl Ether	-	-	1.5	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
4-Chloro-3-methylphenol	-	-	-	-	µg/L	0.49	ND	ND	ND	ND	ND	ND	ND	ND
4-Chloroaniline	-	-	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
4-Chlorophenyl Phenyl Ether	-	-	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
4-Methylphenol	-	-	-	-	µg/L	0.49	ND	ND	ND	ND	ND	ND	ND	ND
4-Nitroaniline	-	-	-	-	µg/L	0.98	ND	ND	ND	ND	ND	ND	ND	ND
4-Nitrophenol	-	-	150	-	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
Acenaphthene	670	990	520	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Acenaphthylene	-	-	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Anthracene	8300	40000	13	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Benz(a)anthracene	0.0038	0.018	0.027	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Benzo(a)pyrene	0.0038	0.018	0.014	8,000	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Benzo(b)fluoranthene	0.0038	0.018	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Benzo(g,h,i)perylene	-	-	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Benzo(k)fluoranthene	0.0038	0.018	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Benzoic Acid	-	-	42	-	µg/L	4.9	ND	ND	ND	ND	ND	ND	ND	ND
Benzyl Alcohol	-	-	8.6	-	µg/L	4.9	ND	ND	ND	ND	ND	ND	ND	ND

ARCADIS

Appendix A. Summary of Semi-Volatile Organic Compounds in Surface Water, Johnson Lake,Portland, Oregon

Constituents	Surface Water Criteria or Screening Values				Units	Reporting Limit	Sample locations							
	Human Health[a]		Ecological											
	Water and Fish Ingestion	Fish Consumption Only	Aquatic Life[b]	Wildlife[c]			SW-1	SW-2	SW-3	SW-4	SW-5	SW-6	SW-7	SW-8
Bis(2-chloroethoxy)methane	-	-	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Bis(2-chloroethyl) Ether	0.03	1.36	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Bis(2-chloroisopropyl) Ether	35	4,360	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Bis(2-ethylhexyl) Phthalate	1.2	2.2	3	8,000	µg/L	2	ND	ND	ND	ND	ND	ND	ND	ND
Butyl Benzyl Phthalate	1500	1900	19	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Chrysene	0.0038	0.018	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Dibenz(a,h)anthracene	0.0038	0.018	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Dibenzofuran	-	-	3.7	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Diethyl Phthalate	350,000	180,000	210	18,000,000	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Dimethyl Phthalate	313,000	290,000	3	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Di-n-butyl Phthalate	2000	4500	35	800	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Di-n-octyl Phthalate	-	-	708	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Fluoranthene	42	54	6.16	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Fluorene	1100	5300	3.9	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Hexachlorobenzene	0.00072	0.00074	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Hexachlorobutadiene	0.45	50	9.3	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Hexachlorocyclopentadiene	206	17,000	5.2	-	µg/L	0.98	ND	ND	ND	ND	ND	ND	ND	ND
Hexachloroethane	1.9	8.7	540	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Indeno(1,2,3-cd)pyrene	0.0038	0.018	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Isophorone	5,200	520,000	2,340	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Naphthalene	-	-	620	284,000	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Nitrobenzene	19,800	690	540	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
N-Nitrosodi-n-propylamine	0.005	0.51	117	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
N-Nitrosodiphenylamine	4.9	16.1	210	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Pentachlorophenol (PCP)	1,010	3	13	1,800	µg/L	0.98	ND	ND	ND	ND	ND	ND	ND	ND
Phenanthrene	-	-	6	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Phenol	3,500	1,700,000	2560	-	µg/L	0.49	ND	ND	ND	ND	ND	ND	ND	ND
Pyrene	830	4,000	-	-	µg/L	0.2	ND	ND	ND	ND	ND	ND	ND	ND

µg/L - micrograms per liter

- - criterion not available

ND - not detected at or above reporting limit

a. values represent Oregon DEQ water quality criteria if available, or USEPA (2002) National Recommended water Quality Criteria

b. values represent Oregon DEQ water qualtiy criteria if available, or aquatic life screening values from Oregon DEQ (1998) Ecological Risk Guidance

c. values represent the lower of avian and mammalian wildlife screening values from Oregon DEQ (1998) Ecological Risk Guidance

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Appendix A. Summary of Dissolved Organic Carbon & Total Organic Carbon in Surface Water, Johnson Lake, Portland, Oregon

Constituent	Units	Reporting Limit	Sample Location							
			SW-1	SW-2	SW-3	SW-4	SW-5	SW-6	SW-7	SW-8
Dissolved Organic Carbon	µg/L	500	2100	2300	2100	2100	2200	2000	1900	2000
Total Organic Carbon	µg/L	500	5700	4000	3300	3100	3200	2700	2800	2700

µg/L - micrograms per liter

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Attachment 5

Oregon DEQ's Comments on Revision 1 of the Johnson Lake Investigation Work Plan, dated December 1, 2003

Oregon
Theodore Kulongoski, Governor

Department of Environmental Quality
Northwest Region Portland Office
2020 SW 4th Avenue, Suite 400
Portland, OR 97201-4987
(503) 229-5263
FAX (503) 229-6945
TTY (503) 229-5471

December 1, 2003

Ms. Keely O'Bryan
Thompson Hine
3900 Key Center
127 Public Square
Cleveland, OH 44114

Re: Owens-Brockway Glass Containers, ECSI No. 1311

Dear Keely:

Thank you for providing the response (dated November 11, 2003) to the issues we raised in an email (dated August 20, 2003) and discussed with you on October 22, 2003 relating to the Johnson Lake Remedial Investigation Work Plan. We approve of the work plan as modified by this document, the previous response to DEQ comments which we received July 17, 2003, and the comments provided below. These documents will be considered addendums to the draft work plan submitted April 30, 2003.

Comments

1. PCB congeners should be analyzed in predator fish samples because of the greater potential for biomagnification. Fish are generally thought of as predators when they reach a size of greater than 250 mm. Fish this size fall into either the medium or large whole fish categories identified for analysis.

2. PCB congeners should also be analyzed in Carp since they are bottom feeders and therefore in greater contact with the sediment and associated prey. They also have high lipid content and are important prey for wildlife as well as sometimes being consumed by humans. A minimum size of 250 grams would be preferable; however, we recognize that the sample set may be limited. We encourage frequent communication with DEQ during work plan implementation so that we can participate in decisions such as which fish samples will be analyzed.

3. Please consider substituting congener analysis for homologue analysis for all tissue samples analyzed. It is our understanding that the analytical costs are similar and the data generated can be used to develop homologue and total PCB concentrations.

Please provide a schedule for work plan implementation no later than 30 days ahead of when you expect to be in the field so that we can plan to provide oversight and have necessary resources available when critical field decisions will be made.

If you have any questions, please call me at (503) 229-6148.

Sincerely,

Jennifer Sutter, Project Manager
Lower Willamette Section

Cc: Mark Tussing, Owens-Brockway
Susan Smith, Owens-Brockway
Alicia Voss, LWS/NWR
Jennifer Peterson, LWS/NWR

DEQ-DC1

ARCADIS

Attachment 6

ARCADIS' Response to the Oregon DEQ's December 2003 Comments on Revision 1 of the Johnson Lake Investigation Work Plan

ARCADIS

Response to December 1, 2003 Comments, Johnson Lake Remedial Investigation Work Plan, Revision 1, as Revised November 11, 2003

Comments are provided in italics, followed by ARCADIS' response. These responses are intended to serve as an addendum to the work plan.

1. PCB congeners should be analyzed in predator fish samples because of the greater potential for biomagnification. Fish are generally thought of as predators when they reach a size of greater than 250 mm. Fish this size fall into either the medium or large whole fish categories identified for analysis.

As agreed during previous discussions and written correspondence with the DEQ, limited sampling and analyses will be conducted for PCB congeners for comparison purposes only. Specifically, three samples of each of the following types will be analyzed for PCB congeners: fish fillets, medium whole fish, benthic invertebrates, and sediment. Sample locations for PCB congener analysis will be randomly selected from among the eight zones to be sampled for other analytical parameters.

The analysis of PCB congeners will be conducted using USEPA Method 8082. Target analytes will include the 12 congeners for which toxicity equivalence factors are available, namely PCBs 77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, and 189.

Consistent with State and Federal risk assessment guidance, the purpose of the sampling and analysis effort to collect data of known quality that permits a quantitative evaluation of potential human health and ecological risks. Our proposed collection protocol includes the sampling and analysis of medium and large predatory fish. It also includes sediment and benthic macroinvertebrate samples to evaluate the uncertainty associated with environmental weathering and preferential uptake processes.

2. PCB congeners should also be analyzed in Carp since they are bottom feeders and therefore in greater contact with the sediment and associated prey. They also have high lipid content and are important prey for wildlife as well as sometimes being consumed by humans. A minimum size of 250 grams would be preferable; however, we recognize that the sample set may be limited. We encourage frequent communication with DEQ during work plan implementation so that we can participate in decisions such as which fish samples will be analyzed.

As stated in our Response to Comment No. 1, the purpose of the PCB congener sampling is to evaluate the uncertainty associated with environmental weathering and preferential uptake processes. We agreed to analyze three samples of each of the following types for PCB

congeners: fish fillets, medium whole fish, benthic invertebrates, and sediment. Our work plan states three fillet samples will be collected for homologue analysis for each of these three types of fish: pan, rough (e.g., carp, suckers), and game. Since game fish are top predators and the preferred catch for recreational fishers, we proposed to analyze three game fish fillet samples for PCB congeners. To accommodate DEQ's request, we will instead analyze three fillet samples, one pan, one rough, and one game fish fillet for PCB congeners. We do not have any information on what is the dominant medium-size fish species in Johnson Lake. Our goal is to collect three representative medium, whole-body fish samples for PCB congener analysis.

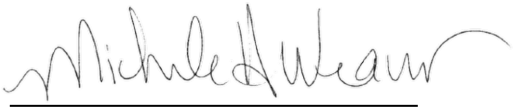
As requested by DEQ, we will provide at least 30 days notice prior to implementing the Johnson Lake Investigation Work Plan. The work plan will be implemented in a manner consistent with State and Federal guidance; field decisions will be made in consultation with DEQ with the understanding that the ARCADIS Field Manager will make the final decisions in the field.

3. Please consider substituting congener analysis for homologue analysis for all tissue samples analyzed. It is our understanding that the analytical costs are similar and the data generated can be used to develop homologue and total PCB concentrations.

We have presented in writing a partial list of scientifically sounds reasons why PCB congener analyses and the TEF assessment approach is not necessary for assessing human health and ecological risks. The PCB homologue approach is more certain, more accurate, and preferable to a PCB congener-based approach. If DEQ has a response to our technical concerns, we would like to discuss them prior to implementing our investigation of Johnson Lake. We agreed to analyze 12 samples for PCB congeners to evaluate the potential uncertainty associated with environmental weathering and preferential uptake processes. We are prepared to implement this scope of work as soon as we receive an unequivocal approval of the Johnson Lake Investigation Work Plan.

(541-776-6111) non-emergency dispatch after hours.

7. An annual activity/collection report associated with this permit must be submitted to ODFW by December 31, 2022 via **https://apps.nmfs.noaa.gov/.** Renewal of this permit will not be issued until ODFW reviews and approves the annual activity/collection report. All fish, crayfish, and mussel take must be recorded in the annual report. Take is defined as harass, harm, pursue, hunt, shoot, wound, kill, trap, control, capture, or collect, or to attempt to engage in any such conduct.
8. May only conduct research and monitoring activities as described in Project Description, Supplemental Information, and Location/Take Information sections of the attached APPS permit. Actual distribution of take among sites is authorized as described in the APPS permit application. All numbers of fish authorized in this permit and the attached APPS permit take table are annual totals.
9. Unless an exception has been granted for your project, indirect mortality may not exceed 3% (electrofishing) or 1% (other methods). Mortality rates are calculated as a percentage of total catch, not to exceed the number as described in the take tables in the permit application. In the event that mortality for any species exceeds this rate, the permittee should contact ODFW ESA staff, Fish.Research@odfw.oregon.gov (503-947-6254 or 541-464-2185) prior to any further activity.
10. If electrofishing, protocols must follow the guidelines established by the National Marine Fisheries Service (NMFS) in June 2000, including no shocking if water temperatures exceed 64.4°F. http://www.westcoast.fisheries.noaa.gov/publications/reference_documents/esa_refs/section4d/electro2000.pdf
11. If the water temperature exceeds 70°F at the capture site, all capturing and handling must stop in stream habitats that contain any salmon, steelhead, trout, or lamprey species.
12. Follow FDA approved protocols and use only FDA approved substances for anesthetizing fish.
    a) **MS-222** may not be used where fish may be subject to sport harvest fisheries within 21 days;
    b) **Clove oil** may not be used at all;
    c) **AQUI-S®** may be used as an alternative to MS-222. To use AQUI-S® 20E as an immediate release sedative in freshwater fish for field-based activities, permit holder must sign up to participate in USFWS-AADAP INAD 11-741 and must comply with the requirements as set forth in the INAD Study Protocol for AQUI-S® 20E https://www.fws.gov/fisheries/aadap/inads/AQUI-S20E-INAD-11-741.html
    d) **Carbon dioxide** can be used as a fish anesthetic as per FDA rules and requires no withdrawal time;
    e) **Electro-anesthesia** can be used as a fish anesthetic as an alternative to chemicals, and requires no withdrawal time.
13. If sampling in multiple sub-basins (4th field HUCs), we recommend that after each sampling event concludes all gear is cleaned and/or allowed to completely dry to prevent the spread of invasive species.
14. If you approach your permitted take or percent indirect mortality at a location and still have sampling to conduct, or would like to revise your sampling methods or procedures; please contact the ODFW ESA staff as soon as possible to request a permit modification so that you can continue your project.

ISSUED BY:  ISSUED DATE:March 31, 2022

Endangered Species Act Specialists
Holly Huchko (541)464-2185
Michele Weaver (503)947-6254

Distribution: Michael Hayworth, Ben Walczak-ODFW

OREGON FISH BIOLOGISTS CONTACT DETAILS updated 01.05.22

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Vacant*	(541) 388-6363 x233	Vacant	Deschutes-Upper	Bend
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ODFW District Biologist	Phone	E-mail	Watershed District	Office
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Cristen Don	(541) 867-7701 x228	Cristen.N.Don@odfw.oregon.gov	Marine Reserves	Newport
Art Martin	(971) 673-6061	Art.C.Martin@odfw.oregon.gov	Columbia River	Clackamas
Kelly Reis	(541) 726-3515 x29	Kelly.E.Reis@odfw.oregon.gov	Willamette BiOp	Springfield[i]

* = Assistant District Biologist

File Number: 26458

Applicant/Holder

Name: Scott Maharry
Title: Senior Biologist
Affiliation: Grette Associates, LLC
Address: 2102 North 30th Street
City,State,Zip: Tacoma, WA 98403
Phone Number: (253)573-9300
Fax Number: (253)573-9321
Email: scottm@gretteassociates.com

Project Information

File Number:	26458
Application Status:	**Application Complete - Issued**
Project Title:	Renew: Johnson Lake Sediment Remedial Action: Fish Tissue Monitoring Study
Project Status:	Renewal
Previous Federal or State Permit/Authorization:	21341
Permit/Authorization Requested:	• Oregon Scientific Taking Permit for Fish and Marine and Freshwater Invertebrates - Issued
Where will activities occur?	Oregon (including Columbia River and offshore waters)
State department of fish and game/wildlife:	N/A

Research Timeframe:	**Start:** 04/05/2022 **End:** 10/05/2022
Sampling Season/Project Duration:	The field sampling component of the project is anticipated to occur over four consecutive days in April of 2022 and if adequate species are not collected another 4 day field effort will be completed before October 2022
Project Type:	Monitoring

Project Description

Purpose: The following is a summarization of the ongoing study at Johnson Lake and includes a project description and purpose in support of the wildlife scientific taking permit. The permit is required to collect specimens of freshwater fish in Oregon for the completion of the scientific fish study on Johnson Lake in Portland, Oregon, Multnomah County.

Purpose:
Owens-Brockway Glass Container, Inc. (Owens) owns and operates a glass manufacturing plant located at 5850 NE 92nd Drive, Portland, Oregon. Johnson Lake lies to the north of the Owens' plant site, with a buffer area of trees and shrubs lying between the plant and Johnson Lake. Owens' Property includes the majority of the lakeshore and much of the lake itself. The site is comprised of the Owens' Property and the remainder of Johnson Lake that is not owned by Owens.
Sediments in Johnson Lake were found to contain polychlorinated biphenyls (PCBs), as described in the Johnson Lake Investigation Work Plan (ARCADIS 2004a), Site Investigation Report (ARCADIS 2004b), and Johnson Lake Risk Assessment (Environ and ARCADIS 2004). Based on the previous investigations and a Feasibility study prepared in 2006, the Oregon Department of Environmental Quality (DEQ) selected a final remedy for the site in the Record of Decision (October 2007) and Record Decision Amendment (July 2009).
The final remedy for cleanup of the PCBs and other contaminants at the Site, as described in the ROD, consisted of the following tasks.
1. Surface soils from the low-lying area will be excavated.
2. Excavated soils will be consolidated in a containment area or disposed of at an off-site landfill.
3. The excavated area will be covered with clean soil and revegetated.
4. A stormwater bio-swale (swale) will be constructed.
5. A thin layer cap will be placed over Johnson Lake sediments.
All tasks, including the off-site landfill disposal for excavated PCB-containing soil, were completed in 2012.

The objective of this study is to evaluate the level of PCBs in fish tissue sampled from Johnson Lake, consistent with the Remediation Operations & Maintenance Plan, Sediment Remedial Action (DOF 2012b). The ROD included the following objective with respect to PCBs in fish tissue, "Prevent human consumption of fish tissue with tissue concentrations of greater than 0.003 ug/kg PCB congener 126". Fish tissue results will be compared with this concentration.
In 2017, a collection and sampling field effort was completed, and all field and lab data were included in a monitoring report submitted to DEQ in January of 2018. Based on the results of this study, PCBs were found to still be present in fish tissue at levels exceeding the ROD standard. Therefore, further efforts to collect fish tissue in 2022 (year 10) will be conducted to further assess levels of PCBs in fish tissue.
The 2022 Johnson Lake fish tissue monitoring study is a requirement of the O&M Plan in response to the results of the 2017 monitoring effort showing contaminant levels in fish samples exceeding the levels specified in the 2007 Record of Decision for the site cleanup. The purpose and objective of this study are to build upon the previous efforts in continuing to evaluate the level of PCBs in fish tissue sampled from Johnson Lake, consistent with the O&M Plan. (DOF, 2012b)

Description: Description:

The fish collection will be conducted on foot or from a small vessel(s) using a waveform electro-shocker, traditional hook and line methods, and nets or seining. Fish will be collected from a variety of locations around the lake, as practicable, and where fish can be collected to represent general fish tissue concentrations lake-wide.

The fish collection will target the following categories of fish. The goal for each category is to collect enough fish for the lab to composite 2 to 5 samples of the same fish type (fillets) and size range (whole fish) into a single composite sample for each category.

• Game fish fillets: One composite sample will target fish typically consumed by humans (e.g., largemouth bass).
• Panfish fillets: One composite sample will target panfish (e.g., bluegills); panfish tend to be smaller than gamefish and may be consumed by humans.
• Rough fish fillets: One composite sample will target rough fish that are not typically targeted by humans but may be consumed by humans (e.g., suckers, carp).
• Large whole fish: One composite sample will target fish between 125 and 300 mm in length that may be consumed by large wildlife species.
• Small whole fish: One composite sample will target fish <125 mm in length that may be consumed by smaller wildlife species.

Due to habitat limitations and low oxygen conditions previously documented in Johnson Lake, collecting and analyzing the numbers and types of fish samples described above may not be practicable during the initial four-day field effort. A good faith effort (four days moving about the lake on foot or by boat, attempting to electroshock or otherwise collect fish that are present) will be made to characterize fish PCB concentrations that are representative of those existing in Johnson Lake. In the event that adequate fish are not collected from each category, a second 4-day collection and sampling effort will be made over the same period at a later date during 2022. This approach is consistent with the O&M Plan (DOF 2012b).

During the 2017 field effort, Grette biologists encountered an approximately six-foot length, White sturgeon. All efforts were made during the 2017 study to avoid catching or otherwise harassing the fish. It is possible that this sturgeon may still be present in Johnson Lake during the 2022 field effort. While efforts will be made to avoid catching this particular fish, placement of gill nets and other passive sampling equipment may result in the capture of the sturgeon. The sturgeon has been included in the table of species anticipated to be collected in anticipation of this.

Supplemental Information

Methods:

The fish collection will be conducted on foot or from a small vessel(s) using a waveform electro-shocker, traditional hook and line methods, and nets or seining. Fish will be collected from a variety of locations around and within the lake, as practicable, and where fish can be collected to represent general fish tissue concentrations lake-wide. Composite samples of each fish collected shall adequately represent the sampling category and be of the same type (species) and size (age) class. The collection will be consistent with EPA Guidelines (Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume 1 Fish Sampling and Analysis) (EPA, 2015), in a manner such that the differences between lengths of individuals in a composite sample and the average of the lengths shall not exceed 10 percent, and at least one replicate shall also be analyzed. (DEQ 2018)

Fish collected at each sample location will be enumerated by species, categorized (game fish, panfish, rough fish), and measured. Fish collected during sampling from a specific location within the lake will be temporarily held in a decontaminated cooler filled with water from Johnson Lake. Once sampling activities from that area are complete, fish targeted for tissue analysis will be euthanized via CO_2 immersion (dry ice added to holding water), wrapped in aluminum foil, and stored on ice in collection bags for transport to ALS Global laboratories in Kelso, Washington for analysis. In an effort to implement humane practices, larger fish will be dispatched by stunning the fish with an appropriately sized blunt tool just above the eyes on the back of the skull. Any fish not required for tissue analysis will be returned to the location in the lake from which they were collected prior to CO_2 immersion.

Based on previous sampling in Johnson Lake (ARCADIS 2004b), it is unlikely that more than five fish of any fillet category or >125 mm will be collected during the course of sampling. If fish in any category continue to be collected after the goal of five individuals has been met, they may be used to replace previously collected

	fish in order to get a more spatially-representative sample of fish tissue in each category. This will be noted on field forms. Each sampling location will be recorded using a dGPS. This will include locations where fish collection was not successful. Water temperature and dissolved oxygen levels at will be collected at each sample location in the middle water column. Weather conditions (precipitation) also will be recorded.
Intentional Lethal Take:	Fish targeted for tissue analysis will be euthanized via CO2 immersion (dry ice added to holding water), or dispatched in the most humane method. Fish not targeted for analysis will be returned to the lake in the location where they were captured prior to CO2 immersion.
Anticipated Effects on Animals:	Fish targeted for tissue analysis will be humanely euthanized. While upland wildlife may be temporarily disturbed during sampling efforts, no permanent wildlife impacts are anticipated.
Measures to Minimize Effects:	Only fish needed to fulfill the O&M sample requirements will be euthanized. All other fish will be returned to the lake in the location where they were captured. Care will be taken to avoid disturbance of native vegetation surrounding the lake while accessing the lake for sampling activities.
Disposition of Tissues:	Whole fish will be bagged, labeled and placed on ice in a cooler. The cooler will be transported to the laboratory where the samples will be processed for analysis. After analysis, the laboratory will dispose of the tissue samples in accordance with their Standard Operating Procedures.
Public Availability of Product/Publications:	

District Biologist Comments

Date	From	Comments
03/23/2022	Michael Hayworth	The North Willamette District does not have any concerns regarding the proposed sampling. Please inform us of any novel or unexpected species discovered in your efforts. Good Luck!

Federal Information

No Federal comments or authorizations.

Location/Take Information

Freshwater Location

Research Area: Pacific Ocean **State:** OR **Sub Basin (4th Field HUC):** Lower Willamette **Stream Name:** Johnson Lake **Latitude:** 45.565416 **Longitude:** -122.563001
Sale in Oregon of species taken: None
Location Description: Owens-Brockway Glass Container, Inc. 5850 NE 92nd Drive Portland, OR

Take Information

Line	Ver	Species	Listing Unit/Stock	Production /Origin	Life Stage	Sex	Expected Take	Indirect Mort	Take Action	Observe /Collect Method	Procedure	Transport Record	Begin Date	End Date

1		Bass, Largemouth	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Electrofishing, Backpack	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
2		Bluegill	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Electrofishing, Backpack	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
3		Carp, Common	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Electrofishing, Backpack	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
4		Sucker (unknown)	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Electrofishing, Backpack	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
5		Stickleback, Threespine	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Electrofishing, Backpack	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
6		Sculpin (unknown)	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Electrofishing, Backpack	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
7		Pumpkinseed	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Electrofishing, Backpack	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
8		Dace (unknown)	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Electrofishing, Backpack	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
9		Shiner (unknown)	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Electrofishing, Backpack	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022

10		Bass, Smallmouth	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Electrofishing, Backpack	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
11		Bass, Largemouth	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Angling (Rod and Reel)	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
12		Bluegill	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Angling (Rod and Reel)	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
13		Carp, Common	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Angling (Rod and Reel)	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
14		Sucker (unknown)	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Angling (Rod and Reel)	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
15		Stickleback, Threespine	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Angling (Rod and Reel)	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
16		Sculpin (unknown)	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Angling (Rod and Reel)	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
17		Pumpkinseed	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Angling (Rod and Reel)	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
18		Dace (unknown)	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Angling (Rod and Reel)	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022

19		Shiner (unknown)	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Angling (Rod and Reel)	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
20		Bass, Smallmouth	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Angling (Rod and Reel)	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
21		Bass, Largemouth	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Seine, Beach	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
22		Bluegill	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Seine, Beach	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
23		Carp, Common	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Seine, Beach	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
24		Sucker (unknown)	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Seine, Beach	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
25		Stickleback, Threespine	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Seine, Beach	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
26		Sculpin (unknown)	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Seine, Beach	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
27		Pumpkinseed	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Seine, Beach	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022

28		Dace (unknown)	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Seine, Beach	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
29		Shiner (unknown)	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Seine, Beach	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
30		Bass, Smallmouth	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Seine, Beach	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
31		Bass, Largemouth	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Net, Gill/Trammel	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
32		Bluegill	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Net, Gill/Trammel	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
33		Carp, Common	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Net, Gill/Trammel	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
34		Sucker (unknown)	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Net, Gill/Trammel	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
35		Stickleback, Threespine	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Angling (Rod and Reel)	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
36		Sculpin (unknown)	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Net, Gill/Trammel	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022

37		Pumpkinseed	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Net, Gill/Trammel	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
38		Dace (unknown)	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Net, Gill/Trammel	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
39		Shiner (unknown)	NA	Natural	All	Male and Female	5	0	Intentional (Directed) Mortality	Net, Gill/Trammel	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
40		Bass, Smallmouth	NA	Natural	All	Male and Female	10	0	Intentional (Directed) Mortality	Net, Gill/Trammel	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
41		Sturgeon, white	NA	Natural	Adult	Male and Female	1	0	Capture/Handle/Release Animal	Electrofishing, Backpack	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
42		Sturgeon, white	NA	Natural	Adult	Male and Female	1	0	Capture/Handle/Release Animal	Hook and line/angler/rod and reel	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
43		Sturgeon, white	NA	Natural	Adult	Male and Female	1	0	Capture/Handle/Release Animal	Seine, Beach	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022
44		Sturgeon, white	NA	Natural	Adult	Male and Female	1	0	Capture/Handle/Release Animal	Net, Gill	Anesthetize; Tissue sample (other internal tissues)	N/A	4/5/2022	10/5/2022

Project Contacts

Primary Contact: Scott J Maharry

Principal Investigator: Scott J Maharry

Other Personnel

Name	Role(s)
Janae Dinkins	Co-Investigator
Donny Neel	Co-Investigator
Chad A Wallin	Co-Investigator

Status

Application Status: Application Complete
Date Submitted: February 10, 2022
Last Date Archived: March 31, 2022

• Oregon Scientific Taking Permit for Fish and Marine and Freshwater Invertebrates

Current Status: Issued **Status Date:** March 31, 2022
Date State Approved: March 31, 2022
Expire Date: December 31, 2022

Attachments

Application Archive - P26458T14Issued.pdf (Added Mar 31, 2022)

Modification Requests

This section is currently empty.

Reports

Report Required

Nbr	Report Type	Report Period		Date Due	Status	Date Received
		Start Date	End Date			
1	Annual-Year End	04/05/2022	12/31/2022	12/31/2022	N/A	