LWG
LOWER WILLAMETTE GROUP

PORTLAND HARBOR RI/FS

ROUND 2 QUALITY ASSURANCE PROJECT PLAN ADDENDUM 9: FISH AND INVERTEBRATE TISSUE AND COLLOCATED SEDIMENT SAMPLING FOR ROUND 3B

DRAFT

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The Lower Willamette Group

Prepared by
Integral Consulting Inc.

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SECTION A: PROJECT MANAGEMENT

A1 TITLE AND APPROVAL SHEET

PORTLAND HARBOR RI/FS
FISH AND INVERTEBRATE TISSUE AND COLLOCATED SEDIMENT SAMPLING FOR ROUND 3B

Quality Assurance Plan Approvals

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CAS Laboratory QA Manager:	Lee Wolf	_____	Date: _____
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A2 TABLE OF CONTENTS

A2.1 List of Tables

A2.2 LIST OF ACRONYMS

ACG	analytical concentration goal
Axys	Axys Analytical Services
CAS	Columbia Analytical Services
COI	chemicals of interest
CVAAS	cold vapor atomic absorption spectrometry
DQO	data quality objective
EPA	U.S. Environmental Protection Agency
FSP	field sampling plan
GPC	gel permeation chromatography
GC/ECD	gas chromatography/electron capture detector
GC/FPD	gas chromatography/flame photometric detection
GC/MS	gas chromatography/mass spectrometry
GFAAS	graphite furnace atomic absorption spectrometry
HRGC/HRMS	high-resolution gas chromatography/high-resolution mass spectrometry
ICP/AES	Inductively coupled plasma/atomic emission spectrometry
ICP/MS	inductively coupled plasma/mass spectrometry
LWG	Lower Willamette Group
LWR	Lower Willamette River
MDL	method detection limit
MRL	method reporting limit
PAH	polycyclic aromatic hydrocarbon
PARCC	precision, accuracy, representativeness, completeness, and comparability
PCB	polychlorinated biphenyl
PSEP	Puget Sound Estuary Program
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
RI/FS	remedial investigation and feasibility study
RM	river mile
SIM	selected ion monitoring
SOP	standard operating procedure
SWCA	SWCA Environmental Consultants
SVOC	semivolatile organic compound
Vista	Vista Analytical Laboratory

A3 DISTRIBUTION LIST

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A4 INTRODUCTION AND PROJECT ORGANIZATION

A4.1 Introduction

This quality assurance project plan (QAPP) addendum describes procedures that will be used to determine concentrations of selected chemicals of interest (COI) in fish and invertebrate tissue and collocated sediment samples collected for Round 3B of the Portland Harbor Remedial Investigation/Feasibility Study (RI/FS). The U.S. Environmental Protection Agency (EPA) has specified the target sample areas and numbers of samples of each species to be collected in Round 3B. Five species of fish and shellfish will be collected, including carp, smallmouth bass, and the small-home-range species sculpin, crayfish, and clams. The area to be fished includes river mile (RM) 2 to RM 11 of the lower Willamette River (LWR), as well as additional locations outside of the Study Area at the direction of EPA, as specified in EPA's comments (EPA 2007a) to the draft Round 3B Fish and Invertebrate Tissue Field Sampling Plan (FSP; Integral 2007).

The Round 3B tissue study is part of the RI/FS for the Portland Harbor Superfund Site (Site) in Portland, Oregon. Study objectives, station locations, and sample collection and shipping procedures are described in *Portland Harbor RI/FS Round 3B Field Sampling Plan: Fish and Invertebrate Tissue and Collocated Surface Sediment Sampling* (Integral 2007). The technical approach to the RI/FS is described in the RI/FS Work Plan (Integral et al. 2004).

This QAPP addendum supplements the Round 2 QAPP (Integral and Windward 2004). The Round 2 QAPP describes procedures and requirements for the generation of data of documented and acceptable quality that will be used for the RI/FS, including the ecological and human health risk assessments. This QAPP addendum addresses procedures that will be used for the Round 3B fish and invertebrate tissue and collocated sediment study that are not described in the Round 2 QAPP.

Supplemental information to Sections A and B of the Round 2 QAPP is provided in this QAPP addendum. Project management, special training, and certification requirements are described in Section A8 of the Round 2 QAPP, and specifications for documents and records are described in Section A9 of the Round 2 QAPP; these items are not addressed further in this QAPP addendum. Supplemental information provided in Section B and referred to in the Round 2 QAPP is summarized in the introduction to Section B.

Procedures for project assessment and oversight (Section C of the Round 2 QAPP) will be completed as described in the Round 2 QAPP. Laboratories will only be audited by LWG if serious problems are encountered, as described in the Round 2 QAPP. Procedures for data validation (Section D of the Round 2 QAPP) will be

completed as described in the Round 2 QAPP. No supplemental information is required for QAPP Sections C and D.

A4.2 PROJECT AND TASK ORGANIZATION

The organizational structure for activities associated with the Round 3B investigation is provided in Section 3.0 and Figure 3-1 of the Round 3B Fish and Invertebrate Tissue FSP (Integral 2007). Key project personnel are identified in Section 3.0 of the FSP and in Table A4-1 of this QAPP addendum. Task descriptions are provided in the FSP and the Round 2 QAPP.

Chemical analyses for tissue samples will be completed by Columbia Analytical Services (CAS) and Axys Analytical Services (Axys). Chemical analyses for collocated sediments will be completed by CAS and Vista Analytical Laboratory (Vista; formerly known as Alta Analytical Laboratory).

Contact information for key project personnel for the fish and invertebrate tissue and collocated sediment study is provided in Table A4-1 of this QAPP addendum.

A5 PROBLEM DEFINITION AND BACKGROUND

The *Portland Harbor RI/FS Comprehensive Round 2 Site Characterization Summary and Data Gaps Analysis Report* (Integral et al. 2007) summarizes the site data collected to date, and presents an evaluation of the data needed to complete the RI/FS. However, in its June 8, 2007 letter to the LWG (EPA 2007b), EPA Region 10 indicated additional composite tissue samples from various species were needed to meet two primary data quality objectives (DQOs). Excerpts from these DQOs are listed below:

- **Contaminant of Interest Uncertainty** – "for refining uncertainties in COI tissue concentrations in order to ensure that the full range of contaminant sources are captured in the tissue sampling," and "to ensure a representative data set both to identify sources and to characterize ranges in risk for both ecological receptors and humans"
- **Food Web Model Calibration and Validation** – "to confirm the utility, ability, and accuracy of the Food Web Model to meet its objectives."

The Round 3B FSP and this QAPP are designed to implement the Round 3B biota sampling program outlined in EPA's Round 3 Data Gap Summary table provided to the LWG on June 26, 2007 and EPA's comments to the draft FSP (EPA 2007a,b).

A6 TASK DESCRIPTION

The tasks to be completed for the Round 3B tissue study include the following:

- Collection of smallmouth bass and carp

- Collection of small-home-range species (clams, crayfish, and sculpin) and collocated sediment samples
- Preparation of fillets, sample homogenization, and laboratory analysis of tissue and collocated sediment samples
- Data validation and data quality evaluation
- Data management
- Report preparation.

Summaries of field and laboratory tasks and references to detailed descriptions are provided in this section. Procedures for data quality evaluation, data management, and report preparation are described in the Round 2 QAPP.

A6.1 Sample Collection and Processing

Overall, a total of 62 composite samples of fish and invertebrate tissue and 35 composite samples of collocated surface sediment are targeted for collection in Round 3B. In addition, if the targeted fish are collected, 27 composite samples of carp and smallmouth bass fillets will be prepared. The following samples will be targeted:

- **Clams**. Ten composite samples will be targeted. A minimum of 140 g of soft tissue will be targeted for each station. A single collocated composite surface (0-10 cm) sediment sample will also be collected from each area where clams are successfully sampled following the sampling protocol used in Round 2 (Windward and Integral 2005).
- **Crayfish**. Nine composite samples will be targeted. A minimum of 300 g will be targeted for each sample (approximately 12 crayfish) with a minimum size of 100 mm for each individual. The crayfish will be identified to species in the field. Following successful collection of a crayfish composite from a target location, a single collocated composite surface sediment sample will be collected.
- **Sculpin**. Sixteen composite samples will be targeted. Each composite will contain a minimum of 195 g (approximately 22 fish) with a minimum fish size of 90 mm. The sculpin will be identified to species in the field. Following successful collection of a sculpin composite from a target location, a single collocated composite surface sediment sample will be collected.
- **Smallmouth bass**. Eighteen composites each consisting of five individual fish will be targeted. Target samples will include one composite for each 1-mile river section from each river bank (east and west) from RM 2.5 to 11.5, excluding Swan Island Lagoon and an east composite from RM 4.5 to 5.5. An east composite from RM 1.0 to 2.5 and east and west composites from RM 10.5 to 12 will also be collected. The target total length range for smallmouth

bass is 225 to 355 mm, as described in the FSP. Smallmouth bass smaller than 225 mm will not be included in the composite samples. In addition, the smallest fish in any composite will be no smaller than 75% of the largest fish (EPA 2000). Samples will be filleted and separate composites will be prepared of the fillets and the remaining fish bodies.

- **Carp**. Nine composites each consisting of five individual fish will be targeted. Samples will include three composites from three LWR reaches (RM 0-4, RM 4-8, and RM 8-12). The target total length range for carp is 508 to 677 mm. Samples will be filleted and separate composites will be prepared of the fillets and the remaining fish bodies.

Sample and analysis summaries for all tissue and collocated sediment samples are provided in Tables A6-1 and A6-2, respectively. Sampling areas are shown in Figures 2-1a-d of the FSP. Sampling procedures are described in Section 4.0 of the FSP and summarized in Section B2 of this QAPP addendum.

Biota tissue composites and collocated sediment samples will be analyzed for the target analytes listed in Table 2-2 of the FSP. Table 2-3 of the FSP shows the mass of tissue needed to meet analytical goals for each of the species being sampled, the target weight of individuals, and the number of individuals required in a composite to meet the target mass. For clams, crayfish, and sculpin, mass rather than number of individuals will be prioritized. Thus, the number of individuals in a sample may be more or less than indicated in Table 2-3 of the FSP. However, a minimum of five organisms will be targeted for smallmouth bass and carp regardless of the mass of the individuals. One post-homogenization split will be prepared for each species provided that sufficient material is collected.

SWCA Environmental Consultants (SWCA) will be responsible for determining final weights and lengths and completing health assessments for the fish and crayfish, in addition to filleting smallmouth bass and carp. CAS will be responsible for shucking clams and homogenizing all of the tissue samples. Once the homogenized composite samples have been prepared, subsamples for chemical analysis will be placed in glass jars and stored frozen (below -20°C) until chemical analysis (EPA 2000). CAS will supply subsamples of the tissue samples to Axys for analysis of polychlorinated biphenyl (PCB) congeners, pesticides, dioxins, and lipids. In addition, 20 g of sample will be provided to EPA for bass and carp samples and, when available, for clam samples.

A6.2 Laboratory Analyses and Deliverables

Chemical analysis of fish and invertebrate tissue samples will be completed by two laboratories, as follows:

- CAS (Kelso, WA) will homogenize the tissue samples and will complete analyses for phthalates and selected semivolatile organic compounds

(SVOCs), polycyclic aromatic hydrocarbons (PAHs), alkylated PAHs, metals (including mercury), butyltin compounds, and percent moisture.

- Axys (Sidney, BC, Canada) will complete analyses for lipids, organochlorine pesticides, PCB congeners, and dioxins/furans. The same extract will be used for all four analyses.

LWG and EPA will make final decisions regarding the priority of analyses and the sample mass to be used for each analysis when sample weights are available.

Chemical analysis of collocated surface sediment samples will be completed by two laboratories, as follows:

- CAS will complete analyses for conventional constituents, metals (including mercury), butyltin compounds, petroleum hydrocarbons, SVOCs, alkylated PAHs, organochlorine pesticides, PCB Aroclors, and dioxins and furans.
- Vista will complete PCB congener analyses.

Laboratories and analysis methods for the tissue and collocated sediment samples are provided in Tables A6-3 and A6-4, respectively. Complete analyte lists with analytical concentration goals (ACGs), method detection limits, and method reporting limits (MRLs) are provided in Tables A6-5 and A6-6 for tissue and collocated sediment samples, respectively. The ACGs provided in the Round 1 QAPP (SEA 2002) are provided in Tables A6-5 and A6-6.

Analyses will be completed using EPA methods and other established methods as indicated in Tables A6-3 and A6-4. Axys will use internal standard operating procedures (SOPs) for analysis of pesticides and for coextraction and fractionation of pesticides, PCBs, and dioxins. Laboratory data deliverables are described in the Round 2 QAPP.

A6.3 Project Schedule

The proposed Round 3B target sampling areas for smallmouth bass, carp, sculpin, crayfish, and clams will be extensively sampled in the proposed period between mid-August and October 31, 2007. Clam collection and collocated sediment collection is expected to take place in early November 2007. Allowing 3 weeks for EPA and LWG to determine which fish to include in composites for the various fishing areas and to resolve any issues related to small sample mass, the following tasks are expected to be completed within the approximate timeframes indicated:

- Fish compositing and homogenization, 3 weeks; fish tissue preparation complete and samples delivered to Axys in mid-December 2007
- Sample analysis, 4 weeks at CAS and Vista and 12 weeks at Axys; laboratory data delivery mid-January 2008 for CAS and Vista, and mid-March 2008 for Axys

- Data validation, 4 weeks; final validated data delivered in mid-April 2008
- Data reduction and reporting, 4 weeks; final database complete in mid-May 2008.

A field sampling report will be submitted to EPA within 60 days of completing the final field sample collection effort described in the FSP. LWG-validated analytical laboratory data will be provided to EPA in an electronic format within 6 weeks of receipt of the final validated data for the Round 3B tissue study. A data summary report will be submitted to EPA no later than 90 days after validation of analytical laboratory data is complete.

A7 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

A7.1 Data Quality Objectives

DQOs for the Portland Harbor RI/FS are described in Section 7 of the Work Plan (Integral et al. 2004). ACGs have been established to identify analytical sensitivity levels that will be sufficient to determine ecological and human health risks for the Portland Harbor RI/FS. Although ACGs can be met for many analytes, methods and method modifications that were designed to optimize laboratory MRLs will not be sufficient to meet the ACGs in all cases. MRLs and ACGs for the Round 3B fish and invertebrate tissue and collocated sediment study are provided in Tables A6-5 and A6-6.

A7.2 Data Quality Indicators

The overall quality objective for Round 3B is to develop and implement procedures that will ensure the collection of representative data of known and acceptable quality. The QA procedures and measurements that will be used for this project are based on EPA and Puget Sound Estuary Program (PSEP) guidance (EPA 1994, 1999, 2005a,b; Plumb 1981; PSEP 1986, 1997a,b) and on established laboratory methods from other sources (Krone et al. 1988).

Quality control (QC) samples and procedures are specified in each method protocol that will be used for this project. Methods are summarized in Tables A6-3 and A6-4. All QC requirements will be completed by each laboratory as described in the protocols and in the Round 2 QAPP. Laboratory control limits for QC samples and procedures are provided in Tables A7-1 through A7-4 and in the laboratory QA manuals (Appendix C of the Round 2 QAPP, and Appendices A of QAPP Addenda 1 and 2 [Integral 2004a,b]). Data validation criteria and procedures are described in Sections D1 and D2 of the Round 2 QAPP. During data validation, low matrix spike or surrogate recoveries will be evaluated for analytes with wide laboratory control limits to evaluate potential bias of the data. Data will be qualified on this basis if a

bias is identified. PARCC parameters (i.e., precision, accuracy or bias, representativeness, completeness, comparability) are described in the Round 2 QAPP.

Target MRLs for this study are summarized in Tables A6-5 and A6-6. The MRLs for the equipment blanks are summarized in Table A6-7. Laboratory methods are described below in Section B4. Method detection limits (MDLs) have been determined by each laboratory for each analyte, as described in the Round 2 QAPP. MDLs are provided in Tables A6-5 and A6-6.

Analyte concentrations for this investigation will be reported to the MDL, as described in the Round 2 QAPP. For analyses completed by Axys and Vista (i.e., dioxins/furans, chlorinated pesticides and PCB congeners by HRGC/HRMS), sample-specific detection limits will be reported as described in EPA methods 1613B and 1668A and Axys' standard operating procedures.

SECTION B: DATA GENERATION AND ACQUISITION

Section B of this Round 2 QAPP addendum includes the following supplemental sections:

- B1 Sampling Process Design
- B2 Sampling Methods
- B3 Sample Handling and Custody
- B4 Field and Laboratory Methods
- B5 Quality Control.

The following information is provided in the Round 2 QAPP and is not addressed further in this QAPP addendum:

- B6 Instrument/Equipment Testing, Inspection, and Maintenance
- B7 Instrument/Equipment Calibration and Frequency
- B8 Inspection/Acceptance of Supplies and Consumables
- B9 Non-direct Measurements
- B10 Data Management.

Details regarding field documentation for fish and shellfish tissue collection are provided in the FSP (Integral 2007).

B1 SAMPLING PROCESS DESIGN

Fish and shellfish tissue will be targeted from 62 sampling areas. For clams, crayfish, and sculpin, the three species with small home ranges, composite collocated surface sediment samples (0-10 cm) will also be collected from each of the 35 sampling locations if biota composite samples are successfully collected. The composite collocated sediment samples will reflect the locations from which the biota were collected, as described in Section 4.2.4 of the FSP.

For smallmouth bass and carp, each individual will be filleted, and the fillets and remaining bodies will be composited and analyzed separately for each sample. All samples for this study will be composites of five or more individual fish or shellfish, or three or more collocated sediment grab samples. Details regarding the sampling design for the Round 3B tissue study are provided in the FSP (Integral 2007).

B2 SAMPLING METHODS

This section outlines sample collection methods, equipment, and sample requirements for fish, shellfish, and collocated sediment. Procedural details and SOPs for field methods are provided in the FSP (Integral 2007). Sample containers, sample size requirements, preservation, and holding times are summarized in Table B2-1. Corrective actions are addressed in Sections B2.3 and C1 of the Round 2 QAPP.

B2.1 Sample Collection and Processing Procedures

To meet the sampling objectives of the Round 3B fish and invertebrate tissue study, several fishing methods will be utilized. The sampling methods for each of the target species will be as follows:

- Clams will be collected by benthic sledge trawling.
- Crayfish will be collected using standard baited crayfish traps.
- Sculpin will be collected with set lines and by backpack electrofishing.
- Smallmouth bass and carp will be collected by boat electrofishing and may additionally be collected by beach seine, backpack electrofishing, angling, or set line.

Details regarding these sampling methods and the sampling areas are provided in Section 4 of the FSP.

Collocated sediment samples will be collected for the small-home-range species (i.e., clams, crayfish, and sculpin). Collocated surface sediment samples will be collected with a stainless-steel, modified, 0.1-m^2, van Veen grab sampler. The top 10 cm of sediment will be collected. Composite collocated sediment samples will be prepared

to reflect the locations from which fish or shellfish were collected for each sample, as described in Section 4.6.2 of the FSP.

All samples will be stored in a cooler with ice and transported to the field laboratory at the end of the day. If the samples cannot be delivered to CAS within 24 hours of sampling, they will be frozen at the field lab. Frozen samples will be transferred to CAS in coolers with ice and stored frozen at CAS.

Carp and bass samples will be filleted at the Portland field lab. A portion of the clams will be depurated at the field lab for samples with sufficient clam tissue mass, as described in Section 4.8.2 of the FSP. Fish whole-body and fillet samples will be composited and homogenized at CAS. Invertebrate samples will be shucked (clams only), composited, and homogenized at CAS. Procedures for equipment decontamination and sample preparation will be completed as described in CAS's Standard Operating Procedure for Tissue Sample Preparation (SOP MET-TISP, Revision 5). The samples will then be frozen at -20°C until analysis.

B3 SAMPLE HANDLING AND CUSTODY

Sample custody procedures are described in Section B3 of the Round 2 QAPP. Sample preservation and storage requirements and holding times for the tissue samples and collocated sediments are provided in Table B2-1 of this QAPP addendum.

B4 ANALYTICAL METHODS

Laboratory methods to be used for Round 3B are consistent with requirements provided in EPA methods and other widely accepted protocols (EPA 1994, 1999, 2005a,b; Krone et al. 1988; Plumb 1981; PSEP 1986, 1997a,b). Modifications will be made to these methods, as necessary and technically feasible, to improve MRLs. Analytes, MRLs, and ACGs for tissue and collocated sediment samples are provided in Tables A6-5 and A6-6, respectively. Method modifications will not be sufficient to reduce MRLs to the level of the ACGs for several analytes, and ACGs will not be attained in these cases when the analyte is not detected.

B4.1 Analysis of Tissue Samples

Tissue samples from the Study Area will be analyzed for the following constituents as shown in Table 2-2 of the FSP:

- 209 PCB congeners
- Organochlorine pesticides
- PAHs

- Alkylated PAHs (clam samples only)
- Butyltin compounds
- Phthalates, phenols, and additional SVOCs
- Metals
- Dioxins and furans
- Percent lipids
- Percent moisture.

Table 2-3 in the FSP shows the target mass of tissue to be collected for each species, the target weight of individuals, and the approximate number of individuals required in a composite to meet the target mass. For clams, crayfish, and sculpin, mass rather than number of individuals will be prioritized. Thus, the number of individuals in a sample may be more or less than indicated in Table 2-3 of the FSP. However, a minimum of five organisms will be targeted for smallmouth bass and carp regardless of the mass of the individuals. One post-homogenization split will be prepared for each species provided that sufficient material is collected.

The laboratory methods for tissue sample preparation and analysis are summarized in Table A6-3. CAS will homogenize the samples and will provide subsamples of homogenized tissue for analysis at Axys. [The SOP for sample homogenization will be provided to the EPA QA Manager under separate cover.] Axys will complete analyses for lipids, PCB congeners, dioxins/furans, and organochlorine pesticides for tissue samples. CAS will complete the remaining analyses. All tissue data will be reported on a wet-weight basis.

Tissue samples will be analyzed using the procedures previously described in QAPP Addendum 4 and its supplement, for subyearling chinook samples, and Addendum 5, for multiplate tissue samples (Integral 2005b,c,d). These procedures are repeated below for convenience. The simultaneous extraction and subsequent fractionation by Axys for pesticides, PCB congeners, and dioxins/furans is also described below. This procedure was used for the benthic tissue study as described in Round 2 QAPP Addendum 6 (Integral and Windward 2005) and will be used for clam samples for the Round 3B study as available tissue mass is likely to be limited at many locations.

LWG and EPA will make final decisions regarding the analyses to be completed if sample mass is not sufficient to complete all intended analyses. Consideration will be given to the order of priority described in Section B4.1 of Round 2 QAPP Addendum 6 (Integral and Windward 2005).

B4.1.1 Lipids Content in Tissue Samples

Lipid analyses will be completed by Axys. A subsample of the extract for pesticides (tissue samples except clams) or the combined extract for dioxins/furans, PCB congeners, and organochlorine pesticides (clam samples) will be used for lipids

determination. The solvent will be evaporated from the subsample, and the residual lipids will be weighed.

B4.1.2 Metals and Butyltin Compounds in Tissue Samples

Four different methods will be used to analyze the tissue samples for total metals (Table A6-3). Sample digestion with nitric acid and hydrogen peroxide will be used to prepare samples for analysis of metals other than mercury. Analysis for target metals, except chromium, selenium, and mercury, will be completed by inductively coupled plasma/mass spectrometry (ICP/MS). Selenium will be analyzed by graphite furnace atomic absorption spectrometry (GFAAS) to avoid isobaric interferences that occur when selenium analyses are conducted by ICP/MS. Chromium analyses will be completed by inductively coupled plasma/atomic emission spectrometry (ICP/AES).

Mercury samples will be extracted with aqua regia and oxidized using potassium permanganate. Analysis will be completed by cold vapor atomic absorption spectrometry (CVAAS).

The procedure for butyltins will include extraction, derivitization, and analysis of mono-, di-, tri-, and tetrabutyltin. Tropolone in methylene chloride will be used to extract the butyltins, followed by a Grignard reaction with hexylmagnesium bromide to form hexyl derivatives. The extract will be cleaned up using silica and alumina cartridge columns and analyzed by gas chromatography/flame photometric detection (GC/FPD).

B4.1.3 Organic Compounds in Tissue Samples Analyzed at CAS

PAHs will be analyzed by gas chromatography/mass spectrometry (GC/MS) with selected ion monitoring (SIM) to improve detection limits. Automated Soxhlet extraction will be used for PAHs in tissue samples. Ten grams of homogenized tissue will be extracted. The extracts will be cleaned up using gel permeation chromatography (GPC) and silica gel. The concentrations of alkylated PAH homologs will be based on the instrument response of their parent compounds.

SVOCs (including phthalate esters and phenols) will be analyzed by GC/MS. Automated Soxhlet extraction will be used for phthalates in tissue samples. Ten grams of homogenized tissue will be extracted. The extracts will be cleaned up using GPC.

B4.1.4 Organic Compounds in Tissue Samples Analyzed at Axys

PCB congeners, dioxins/furans, and organochlorine pesticides in tissue samples will be analyzed using HRGC/HRMS. A sample aliquot of 10 g will be used for clam samples if sufficient tissue mass is available. For the remaining sample types, 75 g of sample will be extracted for dioxin analysis and 10 g will be extracted for pesticides and PCB congeners. Fifteen percent of the initial pesticide extract (or the combined extract for clams) will be removed for the lipids analysis. The remaining extract will be used for the PCB congener, dioxin/furan, and pesticide analyses.

Tissue samples will be extracted for these analyses with a mixture of dichloromethane and hexane by Soxhlet extraction. For clams, a combination of procedures will be used to separate the various analyte groupings and clean up the extracts, including Florisil®, carbon celite, silver nitrate/acid base silica, and alumina. Four final extracts will be generated for the separate analysis of polar pesticides, less polar pesticides, PCB congeners, and dioxins and furans. Because the various analyte groups will be separated, each final extract will represent the whole of the original sample mass extracted for the analysis, minus the portion used for lipids.

Dioxins and furans will be extracted separately from pesticides and PCBs for the remaining samples. A sample mass of 75 g will be used for the dioxin/furan extractions to attain the reporting limits provided in Table A6-5. Sample extracts will be subjected to the following automated chromatographic column clean-ups prior to instrumental analysis: acid silica, layered acid/base silica, alumina, and carbon celite. Additional cleanup procedures will be used, as necessary, to remove analytical interferences.

Detection limits for PCB congeners and dioxins and furans will be calculated on an individual compound and sample basis, dependent upon the signal-to-background ratio for the specific labeled isomer. The detection limits listed in Table A6-5 are based on MDL studies completed by Axys for each method and are expected to approximate the sample-specific detection limits for typical samples. Sample-specific detection limits will be reported in the database for coplanar PCB congeners, dioxins, and furans that are not detected.

B4.2 Analysis of Collocated Sediment Samples

Three surface sediment grabs will be sampled at each successfully collected invertebrate sampling station and combined into one composite sample. FSP Tables 2-2 and 2-4 list the total number of collocated surface sediment composites. Collocated sediment samples will be analyzed for the following constituents as shown in Table 2-2 of the FSP:

- 209 PCB congeners
- Organochlorine pesticides
- PCB Aroclors
- PAHs
- Alkylated PAHs (collocated sediment for clam samples only)
- Butyltin compounds
- Phthalates, phenols, and additional SVOCs
- Metals, including mercury
- Dioxins and furans
- Total organic carbon

- Grain-size distribution
- Percent moisture.

The laboratory methods for sample preparation and analysis are summarized in Table A6-4.

Collocated sediment samples will be analyzed as described in the Round 2 QAPP (Integral and Windward 2004), and in the Round 2 QAPP Addendum 2 for PCB congener analysis and its supplement (Integral 2004b, 2005a). PCB congener procedures and modifications or refinements to QAPP procedures, as described in the Round 2 Site Characterization Report (Integral 2005e), are included below. The remaining procedures for sediment analyses will be completed as described in the Round 2 QAPP (Integral and Windward 2004).

B4.2.1 Metals in Collocated Sediment Samples

Collocated sediment samples will be analyzed for metals as described in the Round 2 QAPP, with the following modifications. Analyses for aluminum, chromium, copper, nickel, and zinc will be completed by EPA Method 6010B rather than EPA Method 6020. The elevated aluminum concentrations present in Study Area sediments are not appropriate for analysis by ICP/MS. Copper, nickel, and zinc required multiple dilutions for analysis by ICP/MS due to matrix interferences in previously analyzed Round 2 samples (Integral 2005e). Since the concentrations of these analytes are expected to be sufficiently high, they will be reported from the ICP/AES analysis for all samples.

Two options for analysis of arsenic and selenium were provided in the Round 2 QAPP, depending on their concentrations in the samples. Based on previous Round 2 sediment data (Integral 2005e), arsenic levels are expected to be detected by EPA Method 6020 (ICP/MS) for all of the samples (the alternate method, EPA Method 7062, will be used should arsenic be undetected in a sediment sample). Selenium will be analyzed by EPA Method 7742 due to expected isobaric interference on both the primary selenium isotope (^{82}Se) and the secondary isotope (^{77}Se) when EPA Method 6020 (ICP/MS) is used.

B4.2.2 Organic Compounds in Collocated Sediment Samples

SVOCs will be analyzed according to methods provided in the corrective action plan for SVOC analysis (Integral 2004c). These are described below.

SVOC analyses will be completed by CAS using a series of three analyses. These analyses include the following components:

1. Prescreening the samples to determine the approximate levels of analytes and matrix interferences
2. Analysis of SVOCs by full scan GC/MS at an appropriate dilution, as determined by the screening

3. Analysis of PAHs by GC/MS with SIM; the concentrations of alkylated PAH homologs will be based on the instrument response of their parent compounds.

4. Analysis of tri-, tetra-, and pentachlorophenols by gas chromatography/electron capture detection (GC/ECD).

Automated Soxhlet extraction will be used for the SVOC and PAH analyses, and sonication extraction will be used for the chlorinated phenols. GPC cleanup will be used for SVOCs and PAHs, and silica gel cleanup will additionally be used for PAH analyses. The tri-, tetra-, and pentachlorophenols will be esterified and analyzed by GC/ECD, as described in EPA Method 8151M.

For pesticide analyses (i.e., organochlorine pesticides, hexachlorobenzene, hexachlorobutadiene, and hexachloroethane), an equivalent dry-weight mass of 20 g will be extracted and taken to a final volume of 1.0 mL after appropriate cleanup(s). Florisil® and sulfur cleanups will be performed, if necessary. The extracts will be run against a low-level calibration curve to produce the MRLs listed in Table A6-6. The method will be modified by adding analysis of PCB interference check standards to the pesticide analysis to evaluate the pesticide chromatograms for PCB interference, as requested in recent EPA Region 10 guidance for organochlorine pesticide analysis (EPA 2005a). PCB interference check standards will be analyzed within 72 hours of the sample if interference is noted during the laboratory's review of the pesticide chromatograms. The PCB interference check standard will be used to determine which column to use to quantify the pesticides, in the case when PCBs only interfere on one column. The PCB interference check standards will be reviewed during validation to evaluate the laboratory's quantification of pesticide results and to qualify data when interference is present.

For PCB Aroclor analyses, an equivalent dry-weight mass of 20 g will be extracted and taken to a final volume of 1.0 mL after appropriate acid cleanup. Multiple acid cleanups are not necessarily expected, but will be employed if necessary. The extracts will be run against a low-level calibration curve to produce the MRLs provided in Table A6-6.

B4.2.3 PCB Congeners and Chlorinated Dioxins and Furans in Collocated Sediment Samples

PCB congeners and dioxins/furans in sediment samples will be analyzed using HRGC/HRMS. Dioxins/furans will be analyzed by CAS as described in the Round 2 QAPP. PCB congeners will be analyzed by Vista as described below and in Integral (2004b, 2005a).

For PCB congener analysis, the sediment samples will be extracted with toluene by Soxhlet extraction. The cleanup procedures that will be used by the laboratory include back-extraction with sulfuric acid, acidic and basic silica gel column chromatography, and acidic alumina column chromatography. These procedures are

expected to provide sufficient cleanup even for samples that contain high levels of interferents such as petroleum hydrocarbons. Vista will use a DB-1 column rather than the more commonly used SPB-octyl column. This will allow the resolution of PCB 156 and PCB 157, which coelute on the SPB-octyl column. Although PCB 118, a dioxin-like congener, coelutes with PCB 106 on the DB-1 column, PCB 106 is not a significant constituent of any of the Aroclors. Therefore, the concentration of this coelution can be attributed wholly to PCB 118.

Vista will analyze 10 g of sample initially. If PCB 126 is not detected in a sample and if the sample matrix allows, the sample will be re-extracted and reanalyzed using a sample mass of up to 50 g. However, if other coplanar PCB congeners are detected and PCB 126 will not contribute significantly to the sediment's toxicity even if it is present at a level below the MDL, the sample will not be reanalyzed using a larger sample mass. A sample size of 10 g is expected to be sufficient to meet the ACGs for all of the coplanar PCB congeners except PCB 126. The collocated sediment samples will be analyzed for all 209 PCB congeners.

Detection limits for PCB congeners and dioxins and furans are calculated on an individual compound and sample basis and depend on the signal-to-background ratio for the specific labeled isomer. The detection limits listed in Table A6-6 are based on MDL studies completed by CAS and Vista for each method and are expected to approximate the sample-specific detection limits for typical samples. Sample-specific detection limits will be reported in the database for PCB congeners, dioxins, and furans that are not detected.

B5 QUALITY CONTROL

Quality control samples will be prepared in the field and at the laboratories to monitor the bias and precision of the sample collection and analysis procedures.

Field QC samples for this study will include a field splits (i.e., a subsample of a single homogenized sample) for collocated sediment samples. Post-homogenization splits per tissue species will be collected if sufficient tissue mass is collected. An equipment rinse blank for collocated sediment samples will be collected in the field, as described in the Round 2 QAPP. Homogenization equipment rinsate blanks for tissue samples will be collected in the lab for equipment used for sample preparation and homogenization. One rinse blank will be collected for each type of tissue.

Laboratory QC samples and procedures will be completed as described in the Round 2 QAPP. Sufficient sample quantity is unlikely to be available for the preparation of matrix spike samples and duplicates for some of the tissue samples, particularly for clams. If the tissue sample quantity is too small to allow for matrix spike samples and duplicate samples, laboratory control samples and laboratory control sample duplicates will be used to evaluate the accuracy and precision of the analytical procedures and data. All other laboratory QC procedures will be completed as described in the Round 2 QAPP, the referenced method descriptions, and the laboratories' SOPs.

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Tables

Table A4-1. Project Team Contact Information.

Name	Project Role	Phone	Fax	Email
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Table A6-1. Sample and Analysis Summary for Tissue Samples.

Sample Type	Number of Composite Samples [a]	Analyses
Samples for Human Health and Ecological Risk Assessment		
Bass [b]		Lipids
Fillets [c]	18	Percent moisture
Bodies	18	Metals [h]
Carp [b]		Butyltins
Fillets [c]	9	Organochlorine pesticides
Bodies	9	PAHs
Crayfish (whole-body)	9	Alkylated PAHs (clam tissue only)
Clams (whole-body) [d]	10	Phthalate esters
		SVOCs
Bait samples [e]	3	Phenols
Split samples [f]	6	Dioxin/Furans
Homogenization blanks [g]	4	PCB congeners (209 congeners)
Samples for Ecological Risk Assessment		
Sculpin (whole-body)	16	Lipids
		Percent moisture
Bait samples [e]	1	Metals [h]
Split samples [f]	1	Butyltins
Homogenization blanks [g]	1	Organochlorine pesticides
		Alkylated PAHs
		Phthalate esters
		Dioxin/Furans
		PCB congeners (209 congeners)

Notes:

TBD - to be determined.

[a] Fewer samples may be collected if species are not found at all sampling sites.

[b] Target of 5 individual samples per composite sample.

[c] Fillets will be removed from the fish and submitted for compositing and analysis separately from the fish bodies.

[d] A minimum of 35 g of clam tissue is needed for the full suite of analyses; 20 g will be provided to EPA when available.

[e] Assume one type of bait per tissue species except clams.

[f] Post-homogenization splits will be collected at a frequency of 5% of the total number of samples per species.

[g] Homogenization equipment rinsate blanks will be collected at a frequency of approximately 5% of the total number of samples per species.

[h] Metals for tissue analyses include Al, Sb, As, Cd, Cr, Cu, Pb, Mn, Hg, Ni, Se, Ag, Tl, Zn.

Table A6-2. Sample and Analysis Summary for Collocated Sediment Samples.

Sample Type	Number of Composite Samples	Analyses
Samples for Human Health and Ecological Risk Assessment [a]		
Sediment	19	Conventionals [e] Metals [f]
Split samples [b]	1	Butyltins
Equipment blanks [c]	1	Organochlorine pesticides PAHs Alkylated PAHs (collocated sediment for clam samples only) SVOCs Phenols Dioxin/Furans PCB congeners (209 congeners) Phthalate esters
Samples for Ecological Risk Assessment [d]		
Sediment	16	Conventionals [e] Metals [f]
Split samples [b]	1	Butyltins
Equipment blanks [c]	1	Organochlorine pesticides PAHs Dioxin/Furans PCB congeners (209 congeners) Phthalate esters

Notes:

[a] Includes collocated sediments associated with crayfish and clam tissues.

[b] Field split samples will be collected for 5% of the total number of sediment samples.

[c] Field sampling equipment rinsate blanks will be collected for 5% of the total number of samples.

[d] Includes collocated sediments associated with sculpin tissues.

[e] Includes grainsize, TOC, and percent solids.

[f] Metals include Al, Sb, As, Cd, Cr, Cu, Pb, Mn, Hg, Ni, Se, Ag, Tl, Zn.

Table A6-3. Laboratory Methods for Tissue Samples.

Analysis	Laboratory	Sample Preparation		Quantitative Analysis	
		Protocol	Procedure	Protocol	Procedure
Lipids	Axys	EPA 3540C[a]	Soxhlet extraction	Axys SOP	Gravimetric
Percent moisture	CAS	--	--	CAS SOP	Gravimetric
Metals	CAS				
Aluminum, antimony, arsenic, cadmium, copper, lead, manganese, nickel, silver, thallium, zinc		EPA 3050B/PSEP	Acid digestion	EPA 6020	ICP/MS
Chromium		EPA 3050B/PSEP	Acid digestion	EPA 6010B	ICP/AES
Selenium		EPA 3050B/PSEP	Acid digestion	EPA 7740	GFAAS
Mercury		EPA 7471A	Acid digestion/oxidation	EPA 7471A	CVAAS
Butyltin compounds	CAS	Krone el al. 1998	Solvent extraction Derivitization	Krone et al. 1998	GC/FPD
Organochlorine pesticides	Axys	Axys SOP	Soxhlet Extraction Gel permeation chromatography Acid/base silica column Florisil® chromatography 1% deactivated basic Alumina	Axys SOP	HRGC/HRMS
Polycyclic aromatic hydrocarbons[b]	CAS	EPA 3541 EPA 3640A EPA 3630C	Automated Soxhlet extraction Gel permeation chromatography Silica gel cleanup	EPA 8270C-SIM	GC/MS-SIM

Table A6-3. Laboratory Methods for Tissue Samples.

Analysis	Laboratory	Sample Preparation		Quantitative Analysis	
		Protocol	Procedure	Protocol	Procedure
Semivolatile organic compounds [c]	CAS	EPA 3541 EPA 3640A	Automated Soxhlet extraction Gel permeation chromatography	EPA 8270C	GC/MS
Chlorinated dioxins and furans	Axys	EPA 1613B	Soxhlet extraction Gel permeation chromatography Acid/base silica column Florisil® chromatography Carbon celite Layered silver nitrate/acid/base silica 1% deactivated basic Alumina	EPA 1613B	HRGC/HRMS
PCB congeners [d]	Axys	EPA 1668A	Soxhlet extraction Gel permeation chromatography Acid/base silica column Florisil® chromatography 1% deactivated basic Alumina	EPA 1668A	HRGC/HRMS

Notes:

[a] A portion of the PCB congener, pesticide, dioxin/furan extract will be used for lipids determination.

[b] PAH analyses for clam tissue samples include alkylated PAHs.

[c] Includes phenols and phthalate esters.

[d] Analysis will be completed for all 209 PCB congeners.

CAS - Columbia Analytical Services
CVAAS - cold vapor atomic absorption spectrometry
EPA - U.S. Environmental Protection Agency
GC/FPD - gas chromatography/flame photometric detection
GC/MS - gas chromatography/mass spectrometry
GFAAS - graphite furnace atomic absorption spectrometry
HRGC/HRMS - high-resolution gas chromatography/high-resolution mass spectrometry
ICP/MS - inductively coupled plasma - mass spectrometry
PCB - polychlorinated biphenyl
PSEP - Puget Sound Estuary Program
SIM - selected ion monitoring
SOP - Standard operating procedure

Table A6-4. Laboratory Methods for Collocated Sediment Samples.

Analysis	Laboratory	Sample Preparation		Quantitative Analysis	
		Protocol	Procedure	Protocol	Procedure
Conventional Analyses	CAS				
Total solids		--	--	PSEP 1986	Gravimetric
Grain size		--	--	PSEP 1986	Sieve and pipette method
Total organic carbon		Plumb 1981	Acid pretreatment	Plumb 1981	Combustion; coulometric titration
Metals	CAS				
Antimony, arsenic [a], cadmium, lead, manganese, silver, thallium		EPA 3050	Strong acid digestion	EPA 6020	ICP/MS
Aluminum, chromium, copper, nickel, zinc		EPA 3050	Strong acid digestion	EPA 6010B	ICP/AES
Arsenic[a]		EPA 3050	Strong acid digestion	EPA 7062	AAS
Mercury		EPA 7471A	Acid digestion/oxidation	EPA 7471A	CVAAS
Selenium		EPA 3050 EPA 7742	Strong acid digestion Hydride generation	EPA 7742	AAS
Butyltins	CAS	Krone et al. 1988	Solvent extraction Derivatization	Krone et al. 1988	GC/FPD
Organochlorine pesticides	CAS	EPA 3541 EPA 3620B EPA 3660B	Soxhlet extraction Florisil® cleanup Sulfur cleanup	EPA 8081A	GC/ECD
Polycyclic aromatic hydrocarbons [b]	CAS	EPA 3541 EPA 3640A EPA 3630C	Automated Soxhlet Extraction Gel permeation chromatography Silica Gel cleanup	EPA 8270C-SIM	GC/MS-SIM
Tri-, Tetra-, and Pentachlorophenols	CAS	EPA 8151	Sonication extraction Esterification	EPA 8151M	GC/ECD
Semivolatile organic compounds [c]	CAS	EPA 3541 EPA 3640A	Automated Soxhlet Extraction Gel permeatioin chromatography	EPA 8270C	GC/MS

Table A6-4. Laboratory Methods for Collocated Sediment Samples.

Analysis	Laboratory	Sample Preparation		Quantitative Analysis	
		Protocol	Procedure	Protocol	Procedure
Chlorinated dioxins and furans	CAS	EPA 1613B	Soxhlet/Dean Stark extraction Sulfuric acid cleanup Silica/carbon column cleanup	EPA 1613B	HRGC/HRMS
PCB congeners [d]	Vista	EPA 1668A	Soxhlet/Dean Stark extraction Sulfuric acid cleanup Silica column cleanup	EPA 1668A	HRGC/HRMS

Notes:

[a] Arsenic will be analyzed by EPA Method 7062 if it is not detected at the MRL by EPA Method 6020.

[b] PAH analyses for sediment samples collocated with clam tissue include alkylated PAHs.

[c] Includes phthalate esters and mono-, dichlorophenols.

[d] Analysis will be completed for all 209 PCB congeners.

AAS - atomic absorption spectrometry
CAS - Columbia Analytical Services
CVAAS - cold vapor atomic absorption spectrometry
EPA - U.S. Environmental Protection Agency
GC/ECD - gas chromatography/electron capture detection
GC/FID - gas chromatography/flame ionization detection
GC/FPD - gas chromatography/flame photometric detection
GC/MS - gas chromatography/mass spectrometry
HRGC/HRMS - high-resolution gas chromatography/high-resolution mass spectrometry
ICP/AES - inductively coupled plasma/atomic emission spectrometry
NWTPH - Northwest total petroleum hydrocarbons
ICP/MS - inductively coupled plasma - mass spectrometry
PCB - polychlorinated biphenyl
PSEP - Puget Sound Estuary Program
SIM - selected ion monitoring

Table A6-5. Analytes, Analytical Concentration Goals, Method Detection Limits, and Method Reporting Limits for Tissue Samples.

Analytes	ACG[a]	MDL	MRL[b]
Conventional Analyses (percent of whole weight)			
Lipids	*	0.1	0.1
Moisture	*	0.01	0.01
Metals, mg/kg wet weight			
Aluminum	*	0.08	0.4
Antimony	*	0.0016	0.01
Arsenic	0.00027	0.006	0.1
Cadmium	0.01	0.0012	0.004
Chromium	0.054	0.1	0.1
Copper	0.67	0.018	0.02
Lead	*	0.0014	0.004
Manganese	0.431	0.02	0.05
Mercury	0.005	0.0004	0.004
Nickel	0.36	0.006	0.04
Selenium	*	0.2	0.2
Silver	0.089	0.0008	0.004
Thallium	0.001	0.001	0.004
Zinc	5.4	0.08	0.1
Butyltins, µg/kg wet weight			
Monobutyltin	*	0.11	1.0
Dibutyltin	*	0.17	1.0
Tributyltin	5.4	0.35	1.0
Tetrabutyltin	*	0.43	1.0
Organochlorine pesticides, µg/kg wet weight			
2,4'-DDD	*	0.016	0.02
2,4'-DDE	*	0.007	0.02
2,4'-DDT	*	0.009	0.02
4,4'-DDD	5.4	0.018	0.02
4,4'-DDE	3.8	0.008	0.02
4,4'-DDT	3.8	0.013	0.02
Aldrin	0.03	0.017	0.02
alpha-BHC	0.067	0.022	0.02
beta-BHC	0.233	0.010	0.02
delta-BHC	*	0.139	0.05
gamma-BHC (Lindane)	0.322	0.014	0.02
alpha-Chlordane	*	0.017	0.02
gamma-Chlordane	*	0.011	0.02
Oxychlordane	*	0.030	0.02
cis -Nonachlor	*	0.023	0.02
trans -Nonachlor	*	0.012	0.02
Dieldrin	0.026	0.018	0.05
Endosulfan I	108	0.035	0.05

Table A6-5. Analytes, Analytical Concentration Goals, Method Detection Limits, and Method Reporting Limits for Tissue Samples.

Analytes	ACG[a]	MDL	MRL[b]
Endosulfan II	*	0.050	0.05
Endosulfan sulfate	*	0.015	0.05
Endrin	5.4	0.009	0.05
Endrin aldehyde	*	0.017	0.05
Endrin ketone	*	0.017	0.05
Heptachlor	0.0933	0.013	0.02
Heptachlor epoxide	0.046	0.009	0.05
Methoxychlor	90	0.010	0.05
Toxaphene	0.38	--	0.05
Hexachlorobenzene	0.26	--	0.01
Hexachlorobutadiene	5.4	--	--
Polycyclic Aromatic Hydrocarbons, µg/kg wet weight			
2-Methylnaphthalene	*	0.44	1
Acenaphthene	1080	0.11	0.5
Acenaphthylene	*	0.069	0.5
Anthracene	5400	0.065	0.5
Benz(a)anthracene	0.575	0.066	0.5
Benzo(a)pyrene	0.0575	0.081	0.5
Benzo(b)fluoranthene	0.575	0.07	0.5
Benzo(g,h,i)perylene	*	0.073	0.5
Benzo(k)fluoranthene	5.75	0.056	0.5
Chrysene	58	0.076	0.5
Dibenzofuran	72	0.13	0.5
Dibenz(a,h)anthracene	0.0575	0.059	0.5
Fluoranthene	720	0.09	0.5
Fluorene	720	0.15	0.5
Indeno(1,2,3-cd)pyrene	0.575	0.064	0.5
Naphthalene	16	0.4	1
Phenanthrene	*	0.36	0.5
Pyrene	540	0.098	0.5
Alkylated PAHs, µg/kg wet weight			
1-Methylnaphthalene	*	0.3	0.5
C1-Chrysenes	*	NA	5
C1-Dibenzothiophenes	*	NA	5
C1-Fluoranthenes/Pyrenes	*	NA	5
C1-Fluorenes	*	NA	5
C1-Phenanthrenes/Anthracenes	*	NA	5
C2-Chrysenes	*	NA	5
C2-Dibenzothiophenes	*	NA	5
C2-Fluoranthenes/Pyrenes	*	NA	5
C2-Fluorenes	*	NA	5
C2-Naphthalenes	*	NA	5
C2-Phenanthrenes/Anthracenes	*	NA	5
C3-Chrysenes	*	NA	5

Table A6-5. Analytes, Analytical Concentration Goals, Method Detection Limits, and Method Reporting Limits for Tissue Samples.

Analytes	ACG[a]	MDL	MRL[b]
C3-Dibenzothiophenes	*	NA	5
C3-Fluoranthenes/Pyrenes	*	NA	5
C3-Fluorenes	*	NA	5
C3-Naphthalenes	*	NA	5
C3-Phenanthrenes/Anthracenes	*	NA	5
C4-Chrysenes	*	NA	5
C4-Naphthalenes	*	NA	5
C4-Phenanthrenes/Anthracenes	*	NA	5
Phthalate Esters, µg/kg wet weight			
Dibutyl phthalate	1800	2.6	10
Dimethyl phthalate	180000	1.8	10
Diethyl phthalate	*	3.5	10
Butyl benzyl phthalate	3600	1.5	10
Bis(2-ethylhexyl) phthalate	30	1.7	200
Di-n-octyl phthalate	360	1.2	10
Semivolatile Organic Compounds, µg/kg wet weight			
1,2,4-Trichlorobenzene	*	9.8	40
1,2-Dichlorobenzene	1620	11	40
1,2-Diphenylhydrazine	0.16	40	40
1,3-Dichlorobenzene	*	11	40
1,4-Dichlorobenzene	*	11	40
2,4-Dinitrotoluene	*	15	80
2,6-Dinitrotoluene	*	14	40
2-Chloronaphthalene	*	6.7	40
2-Nitroaniline	*	31	200
3,3'-Dichlorbenzidine	*	1000	1000
3-Nitroaniline	*	400	400
4-Bromophenyl phenyl ether	*	6.9	40
4-Chloroaniline	*	400	400
4-Chlorophenyl phenyl ether	*	6.4	40
4-Methylphenol	90	7.7	40
4-Nitroaniline	*	400	400
Aniline	*	800	800
Benzoic Acid	*	400	400
Benzyl alcohol	5400	22	40
Bis-(2-chloroethoxy) methane	*	8.3	40
Bis-(2-chloroethyl) ether	*	13	40
Bis(2-chloroisopropyl) ether	*	13	40
Hexachlorobenzene	0.26	7.3	40
Hexachlorobutadiene	5.4	10	40
Hexachlorocyclopentadiene	*	1000	1000
Hexachloroethane	18	20	40
Isophorone	*	9.2	40
Nitrobenzene	*	12	40

Table A6-5. Analytes, Analytical Concentration Goals, Method Detection Limits, and Method Reporting Limits for Tissue Samples.

Analytes	ACG[a]	MDL	MRL[b]
n-Nitrosodimethylamine	0.025	40	40
n-Nitroso-di-n-propylamine	0.18	22	40
n-Nitrosodiphenylamine	*	6.6	40
Phenols, ug/kg wet wt.			
Phenol	10800	45	100
2-Chlorophenol	*	7.5	40
2,4-Dichlorophenol	*	9.5	40
2,4,5-Trichlorphenol	1800	14	40
2,4,6-Trichlorophenol	*	9.1	40
2,3,4,6-Tetrachlorophenol	*	20	40
Tetrachlorophenol (2,3,4,5 and 2,3,5,6)	540	40	40
Pentachlorophenol	3.5	30	100
2-Methylphenol	*	8.2	40
2,4-Dimethylphenol	*	19	40
2-Nitrophenol	*	12	40
4-Nitrophenol	*	9.9	40
2,4-Dinitrophenol	*	800	800
4-Chloro-3-methylphenol	*	6.8	40
4,6-Dinitro-2-methylphenol	*	400	400
Chlorinated Dioxins and Furans, ng/kg wet weight [c,d]			
2,3,7,8-TCDD	0.0028	0.005	0.03
2,3,7,8-TCDF	0.028	0.010	0.03
1,2,3,7,8-PeCDD	0.0028	0.008	0.17
1,2,3,7,8-PeCDF	0.056	0.009	0.15
2,3,4,7,8-PeCDF	0.0056	0.008	0.15
1,2,3,4,7,8-HxCDD	0.028	0.007	0.17
1,2,3,6,7,8-HxCDD	0.028	0.013	0.15
1,2,3,7,8,9-HxCDD	0.028	0.015	0.17
1,2,3,4,7,8-HxCDF	0.028	0.009	0.16
1,2,3,6,7,8-HxCDF	0.028	0.007	0.15
1,2,3,7,8,9-HxCDF	0.028	0.011	0.15
2,3,4,6,7,8-HxCDF	0.028	0.008	0.16
1,2,3,4,6,7,8-HpCDD	0.028	0.017	0.15
1,2,3,4,6,7,8-HpCDF	0.28	0.016	0.15
1,2,3,4,7,8,9-HpCDF	0.28	0.012	0.15
OCDD	2.8	0.017	0.32
OCDF	2.8	0.046	0.32
Total tetrachlorinated dioxins	*	--	--
Total pentachlorinated dioxins	*	--	--
Total hexachlorinated dioxins	*	--	--
Total heptachlorinated dioxins	*	--	--
Total tetrachlorinated furans	*	--	--
Total pentachlorinated furans	*	--	--

Table A6-5. Analytes, Analytical Concentration Goals, Method Detection Limits, and Method Reporting Limits for Tissue Samples.

Analytes	ACG[a]	MDL	MRL[b]
Total hexachlorinated furans	*	--	--
Total heptachlorinated furans	*	--	--
PCB Congeners, ng/kg wet weight [d]			
Dioxin-like PCB congeners (WHO list)			
PCB-77	*	0.38	0.4
PCB-81	*	0.34	0.4
PCB-105	*	0.36	0.4
PCB-114	*	0.33	0.4
PCB-118	*	0.40	0.4
PCB-123	*	0.68	0.4
PCB-126	*	0.45	0.4
PCB-156/157	*	0.42	0.8
PCB-167	*	0.29	0.4
PCB-169	*	0.37	0.4
PCB-170	*	--	0.4
PCB-180	*	--	0.4
PCB-189	*	0.33	0.4
187 non-planar PCB congeners	*	0.24 - 6.68	--

Notes:

All data are provided on a wet-weight basis.

* A risk-based ACG has not been established.

[a] ACGs are as established for tissue analysis.

[b] The MRL represents the level of lowest calibration standard (i.e., the practical quantitation limit).

[c] MRLs and MDLs for dioxins are provided for 75-g samples. 10-g samples will be analyzed for clam tissue.

[d] Results for the WHO PCB congeners and for dioxins and furans will be reported to the sample-specific MDL.

ACG = analytical concentration goal; ACGs were established by EPA during *ad hoc* meeting with LWG on May 10, 2002.

CAS laboratory - Columbia Analytical Services

CAS number - Chemical Abstract Services number

MDL - method detection limit

MRL - method reporting limit

PCB - polychlorinated biphenyl

WHO - World Health Organization

Table A6-6. Analytes, Analytical Concentration Goals, Method Detection Limits, and Method Reporting Limits for Collocated Sediment Samples.

Analytes	ACG	MDL	MRL [a]
Conventional Analyses			
Total solids (percent of whole weight)	*	0.01	0.01
Grain size (percent) [b]	*	0.1	0.1
Total organic carbon (percent)	*	0.02	0.05
Metals, mg/kg dry wt			
Aluminum	*	0.5	2
Antimony	*	0.03	0.05
Arsenic	*	0.03	0.1
Cadmium	*	0.008	0.02
Chromium	*	0.04	0.2
Copper	*	0.1	0.1
Lead	*	0.02	0.05
Manganese	*	0.04	0.05
Mercury	*	0.006	0.02
Nickel	*	0.05	0.2
Selenium	*	0.02	0.1
Silver	*	0.02	0.02
Thallium	*	0.003	0.02
Zinc	*	0.2	0.5
Butyltins, µg/kg dry wt			
Monobutyltin	*	0.03	1.0
Dibutyltin	*	0.028	1.0
Tributyltin	0.08	0.056	1.0
Tetrabutyltin	*	0.07	1.0
Organochlorine pesticides, µg/kg dry wt			
2,4'-DDD	*	0.11	0.2
2,4'-DDE	*	0.12	0.2
2,4'-DDT	*	0.070	0.2
4,4'-DDD	0.083	0.060	0.2
4,4'-DDE	0.0588	0.050	0.2
4,4'-DDT	0.0588	0.032	0.2
Aldrin	0.00038	0.075	0.2
alpha-BHC	0.001	0.13	0.2
beta-BHC	0.0036	0.15	0.2
delta-BHC	*	0.028	0.2
gamma-BHC (Lindane)	0.005	0.075	0.2
alpha-Chlordane	*	0.12	0.2
gamma-Chlordane	*	0.032	0.2
Oxychlordane	*	0.029	0.2
cis-Nonachlor	*	0.036	0.2
trans-Nonachlor	*	0.033	0.2
Dieldrin	0.0004	0.15	0.2
Endosulfan I	1.7	0.085	0.2

Table A6-6. Analytes, Analytical Concentration Goals, Method Detection Limits, and Method Reporting Limits for Collocated Sediment Samples.

Analytes	ACG	MDL	MRL [a]
Endosulfan II	*	0.10	0.2
Endosulfan sulfate	*	0.040	0.2
Endrin	0.084	0.10	0.2
Endrin aldehyde	*	0.027	0.2
Endrin ketone	*	0.041	0.2
Heptachlor	0.0014	0.040	0.2
Heptachlor epoxide	0.0007	0.065	0.2
Methoxychlor	1.4	0.050	0.2
Toxaphene	0.0059	4.5	20
Hexachlorobenzene	0.33	0.040	0.2
Hexachlorobutadiene	0.6	0.065	0.2
Hexachloroethane	2.0	0.075	0.2
Mirex	0.056	0.032	0.2
Polycyclic aromatic hydrocarbons, µg/kg dry wt			
2-Methylnaphthalene	*	0.39	5
Acenaphthene	72	0.23	5
Acenaphthylene	*	0.24	5
Anthracene	360	0.47	5
Benz(a)anthracene	0.038	0.48	5
Benzo(a)pyrene	0.0038	0.14	5
Benzo(b)fluoranthene	0.038	0.25	5
Benzo(g,h,i)perylene	*	0.64	5
Benzo(k)fluoranthene	0.38	0.15	5
Chrysene	3.8	0.25	5
Dibenz(a,h)anthracene	0.0038	0.59	5
Dibenzofuran	8.2	0.28	5
Fluoranthene	48	0.61	5
Fluorene	48	0.5	5
Indeno(1,2,3-cd)pyrene	0.038	0.16	5
Naphthalene	24	0.37	5
Phenanthrene	*	0.75	5
Pyrene	36	0.37	5
Alkylated PAHs, µg/kg dry weight			
1-Methylnaphthalene	*	0.11	0.5
C1-Chrysenes	*	NA	5
C1-Dibenzothiophenes	*	NA	5
C1-Fluoranthenes/Pyrenes	*	NA	5
C1-Fluorenes	*	NA	5
C1-Phenanthrenes/Anthracenes	*	NA	5
C2-Chrysenes	*	NA	5
C2-Dibenzothiophenes	*	NA	5
C2-Fluoranthenes/Pyrenes	*	NA	5
C2-Fluorenes	*	NA	5
C2-Naphthalenes	*	NA	5
C2-Phenanthrenes/Anthracenes	*	NA	5

Table A6-6. Analytes, Analytical Concentration Goals, Method Detection Limits, and Method Reporting Limits for Collocated Sediment Samples.

Analytes	ACG	MDL	MRL [a]
C3-Chrysenes	*	NA	5
C3-Dibenzothiophenes	*	NA	5
C3-Fluoranthenes/Pyrenes	*	NA	5
C3-Fluorenes	*	NA	5
C3-Naphthalenes	*	NA	5
C3-Phenanthrenes/Anthracenes	*	NA	5
C4-Chrysenes	*	NA	5
C4-Naphthalenes	*	NA	5
C4-Phenanthrenes/Anthracenes	*	NA	5
Phthalate esters, µg/kg dry wt.			
Bis(2-ethylhexyl) phthalate	3.4	7.9	20
Butylbenzyl phthalate	400	1	10
Dibutyl phthalate	204	1.3	10
Diethyl phthalate	*	3.2	10
Dimethyl phthalate	20000	7	100
Di-n-octyl phthalate	40.9	1.7	10
Semivolatile Organic Compounds, µg/kg			
1,2,4-Trichlorobenzene	*	2.6	10
1,2-Dichlorobenzene	184	2.9	10
1,2-Diphenylhydrazine	184	2.4	10
1,3-Dichlorobenzene	*	3	10
1,4-Dichlorobenzene	2.0	2.9	10
2,4-Dinitrotoluene	*	1.5	10
2,6-Dinitrotoluene	*	2	10
2-Chloronaphthalene	*	1.6	10
2-Nitroaniline	*	3.2	20
3,3'-Dichlorbenzidine	*	3.7	100
3-Nitroaniline	*	2.5	20
4-Bromophenyl phenyl ether	*	1.6	10
4-Chloroaniline	*	1.9	10
4-Chlorophenyl-phenyl ether	*	1.4	10
4-Methylphenol	26	1.5	10
4-Nitroaniline	*	1.8	20
Aniline	*	1.5	20
Benzoic Acid	*	96	200
Benzyl alcohol	*	2.1	20
Bis-(2-chloroethoxy) methane	*	1.5	10
Bis-(2-chloroethyl) ether	*	1.9	10
Bis(2-chloroisopropyl) ether	*	2.6	10
Hexachlorobenzene	0.33	1.2	10
Hexachlorobutadiene	0.6	2.5	10
Hexachlorocyclopentadiene	*	29	50
Hexachloroethane	2	3.1	10
Isophorone	*	1	10
Nitrobenzene	*	2.2	10
n-Nitrosodimethylamine	0.0073	6.1	50

Table A6-6. Analytes, Analytical Concentration Goals, Method Detection Limits, and Method Reporting Limits for Collocated Sediment Samples.

Analytes	ACG	MDL	MRL [a]
n-Nitroso-di-n-propylamine	0.053	2.4	10
n-Nitrosodiphenylamine	*	1.6	10
Phenols, µg/kg dry wt			
Phenol	3146	2	30
2-Chlorophenol	26	2	10
2,4-Dichlorophenol	16	1	10
2,4,5-Trichlorophenol	524	0.55	5
2,4,6-Trichlorophenol	1.8	0.39	5
2,3,4,6-Tetrachlorophenol	*	1.7	10
Tetrachlorophenol (2,3,4,5 and 2,3,5,6)	*	0.62	5
Pentachlorophenol	0.58	0.14	5
2-Methylphenol	26	1.5	10
2,4-Dimethylphenol	*	5.5	50
2-Nitrophenol	*	1.5	10
4-Nitrophenol	*	18	100
2,4-Dinitrophenol	*	17	200
4-Chloro-3-methylphenol	*	1.4	10
4,6-Dinitro-2-Methylphenol	*	1.4	100
Chlorinated Dioxins and Furans, pg/g dry wt [c]			
2,3,7,8-TCDD	0.0001	0.08	0.2
2,3,7,8-TCDF	0.001	0.09	0.2
1,2,3,7,8-PeCDD	0.01	0.36	0.5
1,2,3,7,8-PeCDF	0.01	0.25	0.5
2,3,4,7,8-PeCDF	0.01	0.16	0.5
1,2,3,4,7,8-HxCDD	0.09	0.27	0.5
1,2,3,6,7,8-HxCDD	9.4	0.62	0.5
1,2,3,7,8,9-HxCDD	0.001	0.50	0.5
1,2,3,4,7,8-HxCDF	0.001	0.30	0.5
1,2,3,6,7,8-HxCDF	0.0002	0.37	0.5
1,2,3,7,8,9-HxCDF	0.01	0.28	0.5
2,3,4,6,7,8-HxCDF	0.01	0.29	0.5
1,2,3,4,6,7,8-HpCDD	0.01	0.27	0.5
1,2,3,4,6,7,8-HpCDF	0.01	0.39	0.5
1,2,3,4,7,8,9-HpCDF	0.09	0.42	0.5
OCDD	0.09	1.55	1.0
OCDF	9.4	4.04	1.0
Total tetrachlorinated dioxins	*	--	--
Total pentachlorinated dioxins	*	--	--
Total hexachlorinated dioxins	*	--	--
Total heptachlorinated dioxins	*	--	--
Total tetrachlorinated furans	*	--	--
Total pentachlorinated furans	*	--	--
Total hexachlorinated furans	*	--	--
Total heptachlorinated furans	*	--	--

Table A6-6. Analytes, Analytical Concentration Goals, Method Detection Limits, and Method Reporting Limits for Collocated Sediment Samples.

Analytes	ACG	MDL	MRL [a]
PCB congeners, pg/g dry wt [c, d]			
Dioxin-like PCB congeners (WHO list)			
PCB-77	10	0.94	50
PCB-81	10	0.65	50
PCB-105	10	0.68	50
PCB-114	2	0.64	50
PCB-118 (coelution with PCB 106)	10	2.40	50
PCB-123	10	1.00	50
PCB-126	0.01	0.33	50
PCB-156	2	0.88	50
PCB-157	2	0.47	50
PCB-167	100	0.25	50
PCB-169	0.1	0.36	50
PCB-170	*	--	50
PCB-180	*	--	50
PCB-189	10	0.31	50
187 non-planar PCB congeners	*	0.2 - 29	25 - 75

Notes:

* A risk-based ACG has not been established.

[a] The MRL is provided on a dry-weight basis and assumes 50% moisture in the samples.
The MRL for project samples will vary with moisture content in the samples.
The MRL represents the level of lowest calibration standard (i.e., the practical quantitation limit).

[b] Grain-size intervals will include the following:

Gravel	Fine silt
Very coarse sand	Very fine silt
Coarse sand	Clay, phi size >8
Medium sand	

[c] Expected MDLs are shown. MDLs for PCB congeners and dioxins and furans are sample-dependent and will vary from the indicated values.

[d] Results for the WHO PCB congeners will be reported to sample-specific MDLs. Method modifications are described in Section B4.2.3 to improve detection limits if PCBs 126 or 169 are not detected in sediment samples.

ACG = analytical concentration goal; ACGs were established by EPA during *ad hoc* meeting with LWG on May 10, 2002.
CAS laboratory - Columbia Analytical Services
CAS number - Chemical Abstract Services number
MDL = method detection limit
MRL = method reporting limit
PCB = polychlorinated biphenyl
WHO = World Health Organization

Table A6-7. Analytes, Method Detection Limits, and Method Reporting Limits for Equipment Blanks.

Analyte	Lab	CAS number	Equipment Blank Samples MDL	Equipment Blank Samples MRL[a]
Conventional Analyses				
Lipids	Axys	--	NA	NA
Moisture (percent)	Axys	--	NA	NA
Grain size (percent)	CAS	--	NA	NA
Total organic carbon (percent) , mg/L	CAS	--	0.07	0.50
Metals, ug/L	CAS			
Aluminum		7429-90-5	0.3	1
Antimony		7440-36-0	0.005	0.05
Arsenic		7440-38-2	0.05	0.5
Cadmium		7440-43-9	0.002	0.02
Chromium		7440-47-3	0.02	0.2
Copper		7440-50-8	0.01	0.1
Lead		7439-92-1	0.002	0.02
Manganese		7439-96-5	0.02	0.05
Mercury		7439-97-6	0.02	0.2
Nickel		7440-02-0	0.03	0.2
Selenium		7782-49-2	0.1	1
Silver		7440-22-4	0.002	0.02
Thallium		7440-28-0	0.003	0.02
Zinc		7440-66-6	0.05	0.5
Butyltins, µg/L	CAS			
Monobutyltin		78763-54-9	0.0017	0.05
Dibutyltin		14488-53-0	0.00055	0.05
Tributyltin		36643-28-4	0.0006	0.02
Tetrabutyltin		1461-25-2	0.0015	0.05
Organochlorine Pesticides and Selected SVOCs, µg/L	Axys			
2,4'-DDD		53-19-0	0.000121	0.0019
2,4'-DDE		3424-82-6	0.0000485	0.0019
2,4'-DDT		789-02-6	0.000121	0.0019
4,4'-DDD		72-54-8	0.000194	0.00241
4,4'-DDE		72-55-9	0.0000243	0.0019
4,4'-DDT		50-29-3	0.000097	0.00205
Aldrin		309-00-2	0.000299	0.0038
alpha-BHC		319-84-6	0.000233	0.00152
beta-BHC		319-85-7	0.000146	0.0038
delta-BHC		319-86-8	0.0000777	0.00187
gamma-BHC (Lindane)		58-89-9	0.000121	0.0038
alpha-Chlordane		5103-71-9	0.000146	0.0038
gamma-Chlordane		5103-74-2	0.000097	0.0038
Oxychlordane		27304-13-8	0.00034	0.0038
cis -Nonachlor		5103-73-1	0.000146	0.0038
trans -Nonachlor		39765-80-5	0.000194	0.0038
Dieldrin		60-57-1	0.000117	0.00152
Endosulfan I		959-98-8	0.000233	0.00152
Endosulfan II		33213-65-9	0.000272	0.00152
Endosulfan sulfate		1031-07-8	0.000311	0.00152
Endrin		72-20-8	0.0000777	0.00152

Table A6-7. Analytes, Method Detection Limits, and Method Reporting Limits for Equipment Blanks.

Analyte	Lab	CAS number	Equipment Blank Samples MDL	Equipment Blank Samples MRL[a]
Endrin aldehyde		7421-93-4	0.0000972	0.000759
Endrin ketone		53494-70-5	0.0000972	0.00152
Heptachlor		76-44-8	0.00017	0.0019
Heptachlor epoxide		1024-57-3	0.0000583	0.00152
Methoxychlor		72-43-5	0.0000194	0.00152
Toxaphene		8001-35-2	0.6-3.0	0.025
Hexachlorobenzene		118-74-1	0.6-3.0	0.0005
Hexachlorobutadiene		87-68-3	0.6-3.0	0.001
Hexachloroethane		67-72-1	NA	NA
Mirex		2385-85-5	NA	NA
Organochlorine Pesticides and Selected SVOCs, µg/L	CAS			
2,4'-DDD		53-19-0	0.00089	0.012
2,4'-DDE		3424-82-6	0.0019	0.012
2,4'-DDT		789-02-6	0.0011	0.012
4,4'-DDD		72-54-8	0.00094	0.012
4,4'-DDE		72-55-9	0.00079	0.012
4,4'-DDT		50-29-3	0.0023	0.012
Aldrin		309-00-2	0.0021	0.012
alpha-BHC		319-84-6	0.0056	0.012
beta-BHC		319-85-7	0.002	0.012
delta-BHC		319-86-8	0.0018	0.012
gamma-BHC (Lindane)		58-89-9	0.0025	0.012
alpha-Chlordane		5103-71-9	0.0015	0.012
gamma-Chlordane		5103-74-2	0.00057	0.012
Oxychlordane		27304-13-8	0.0014	0.012
cis -Nonachlor		5103-73-1	0.0014	0.012
trans -Nonachlor		39765-80-5	0.00068	0.012
Dieldrin		60-57-1	0.00077	0.012
Endosulfan I		959-98-8	0.00075	0.012
Endosulfan II		33213-65-9	0.0022	0.012
Endosulfan sulfate		1031-07-8	0.0016	0.012
Endrin		72-20-8	0.0018	0.012
Endrin aldehyde		7421-93-4	0.0014	0.012
Endrin ketone		53494-70-5	0.0021	0.012
Heptachlor		76-44-8	0.0045	0.012
Heptachlor epoxide		1024-57-3	0.0014	0.012
Methoxychlor		72-43-5	0.0018	0.012
Toxaphene		8001-35-2	0.081	0.57
Hexachlorobenzene		118-74-1	NA	NA
Hexachlorobutadiene		87-68-3	NA	NA
Hexachloroethane		67-72-1	NA	NA
Mirex		2385-85-5	NA	NA
Polycyclic Aromatic Hydrocarbons, µg/L	CAS			
2-Methylnaphthalene		91-57-6	0.0029	0.022
Acenaphthene		83-32-9	0.0022	0.0022
Acenaphthylene		208-96-8	0.002	0.022
Anthracene		120-12-7	0.0012	0.022
Benz(a)anthracene		56-55-3	0.0023	0.022
Benzo(a)pyrene		50-32-8	0.0018	0.022

Table A6-7. Analytes, Method Detection Limits, and Method Reporting Limits for Equipment Blanks.

			Equipment Blank Samples	
Analyte	**Lab**	**CAS number**	**MDL**	**MRL[a]**
Benzo(b)fluoranthene		205-99-2	0.0021	0.022
Benzo(g,h,i)perylene		191-24-2	0.004	0.022
Benzo(k)fluoranthene		207-08-9	0.0015	0.022
Chrysene		218-01-9	0.0014	0.022
Dibenzofuran		132-64-9	0.013	0.2
Dibenz(a,h)anthracene		53-70-3	0.0018	0.022
Fluoranthene		206-44-0	0.0026	0.022
Fluorene		86-73-7	0.0028	0.022
Indeno(1,2,3-cd)pyrene		193-39-5	0.0023	0.022
Naphthalene		91-20-3	0.0035	0.022
Phenanthrene		85-01-8	0.0035	0.022
Pyrene		129-00-0	0.0024	0.022
Alkylated PAHs, µg/kg	CAS			
1-Methylnaphthalene		90-12-0	NA	NA
C1-Chrysenes		--	NA	NA
C1-Dibenzothiophenes		--	NA	NA
C1-Fluoranthenes/Pyrenes		--	NA	NA
C1-Fluorenes		--	NA	NA
C1-Phenanthrenes/Anthracenes		--	NA	NA
C2-Chrysenes		--	NA	NA
C2-Dibenzothiophenes		--	NA	NA
C2-Fluoranthenes/Pyrenes		--	NA	NA
C2-Fluorenes		--	NA	NA
C2-Naphthalenes		--	NA	NA
C2-Phenanthrenes/Anthracenes		--	NA	NA
C3-Chrysenes		--	NA	NA
C3-Dibenzothiophenes		--	NA	NA
C3-Fluoranthenes/Pyrenes		--	NA	NA
C3-Fluorenes		--	NA	NA
C3-Naphthalenes		--	NA	NA
C3-Phenanthrenes/Anthracenes		--	NA	NA
C4-Chrysenes		--	NA	NA
C4-Naphthalenes		--	NA	NA
C4-Phenanthrenes/Anthracenes		--	NA	NA
Semivolatile Organic Compounds, µg/kg	CAS			
Phthalate Esters				
Dibutyl phthalate		84-74-2	0.027	0.2
Dimethyl phthalate		131-11-3	0.013	0.2
Diethyl phthalate		84-66-2	0.026	0.2
Butyl benzyl phthalate		85-68-7	0.026	0.2
Bis(2-ethylhexyl) phthalate		117-81-7	0.27	2
Di-n-octyl phthalate		117-84-0	0.032	0.2
Phenols				
Phenol		108-95-2	0.02	0.48
2-Chlorophenol		95-57-8	0.015	0.5
2,4-Dichlorophenol		120-83-2	0.024	0.5
2,4,5-Trichlorophenol		95-95-4	0.025	0.5
2,4,6-Trichlorophenol		88-06-2	0.037	0.5

Table A6-7. Analytes, Method Detection Limits, and Method Reporting Limits for Equipment Blanks.

			Equipment Blank Samples	
Analyte	**Lab**	**CAS number**	**MDL**	**MRL[a]**
2,3,4,6-Tetrachlorophenol		58-90-2	NA	NA
Tetrachlorophenol (2,3,4,5 and 2,3,5,6)		25167-83-3	NA	NA
Pentachlorophenol		87-86-5	0.029	0.96
2-Methylphenol		95-48-7	0.06	0.48
2,4-Dimethylphenol		105-67-9	0.32	2
2-Nitrophenol		88-75-5	0.014	0.5
4-Nitrophenol		100-02-7	0.54	2
2,4-Dinitrophenol		51-28-5	0.53	4
4-Chloro-3-methylphenol		59-50-7	0.029	0.5
4,6-Dinitro-2-methylphenol		534-52-1	0.013	2
Other Semivolatile Organic Compounds				
1,2,4-Trichlorobenzene		120-82-1	0.016	0.2
1,2-Dichlorobenzene		95-50-1	0.015	0.2
1,2-Diphenylhydrazine		122-66-7	NA	NA
1,3-Dichlorobenzene		541-73-1	0.011	0.2
1,4-Dichlorobenzene		106-46-7	0.014	0.2
2,4-Dinitrotoluene		121-14-2	0.019	0.2
2,6-Dinitrotoluene		606-20-2	0.0088	0.2
2-Chloronaphthalene		91-58-7	0.015	0.2
2-Nitroaniline		88-74-4	0.015	0.2
3,3'-Dichlorbenzidine		91-94-1	0.43	2
3-Nitroaniline		99-09-2	0.23	1
4-Bromophenyl phenyl ether		101-55-3	0.018	0.2
4-Chloroaniline		106-47-8	0.017	0.2
4-Chlorophenyl-phenyl ether		7005-72-3	0.0084	0.2
4-Methylphenol		106-44-5	0.051	0.5
4-Nitroaniline		100-01-6	0.16	1
Aniline		62-53-3	NA	1
Benzoic Acid		65-85-0	1.71	5
Benzyl alcohol		100-51-6	0.43	4.8
Bis-(2-chloroethoxy) methane		111-91-1	0.012	0.2
Bis-(2-chloroethyl) ether		111-44-4	0.014	0.2
Bis(2-chloroisopropyl) ether		39638-32-9	0.014	0.2
Hexachlorobenzene		118-74-1	0.0037	0.012
Hexachlorobutadiene		87-68-3	0.0043	0.012
Hexachlorocyclopentadiene		77-47-4	0.041	1
Hexachloroethane		67-72-1	0.019	0.2
Isophorone		78-59-1	0.0084	0.2
Nitrobenzene		98-95-3	0.0074	0.2
n-Nitrosodimethylamine		62-75-9	0.00026	0.002
n-Nitroso-di-n-propylamine		621-64-7	0.032	0.2
n-Nitrosodiphenylamine		86-30-6	0.028	0.2
Chlorinated Dioxins and Furans, pg/L	Axys			
2,3,7,8-TCDD		1746-01-6	0.361	1.9
2,3,7,8-TCDF		51207-31-9	0.37	1.9
1,2,3,7,8-PeCDD		40321-76-4	1.35	9.49
1,2,3,7,8-PeCDF		57117-41-6	0.009	0.13
2,3,4,7,8-PeCDF		57117-31-4	1.2	9.49
1,2,3,4,7,8-HxCDD		39227-28-6	1.26	9.49

Table A6-7. Analytes, Method Detection Limits, and Method Reporting Limits for Equipment Blanks.

Analyte	Lab	CAS number	Equipment Blank Samples MDL	Equipment Blank Samples MRL[a]
1,2,3,6,7,8-HxCDD		57653-85-7	1.76	9.49
1,2,3,7,8,9-HxCDD		19408-74-3	1.19	9.49
1,2,3,4,7,8-HxCDF		70648-26-9	0.894	9.49
1,2,3,6,7,8-HxCDF		57117-44-9	0.797	9.49
1,2,3,7,8,9-HxCDF		72918-21-9	1.4	9.49
2,3,4,6,7,8-HxCDF		60851-34-5	1.07	9.49
1,2,3,4,6,7,8-HpCDD		35822-46-9	1.25	9.49
1,2,3,4,6,7,8-HpCDF		67562-39-4	1.2	9.49
1,2,3,4,7,8,9-HpCDF		55673-89-7	0.942	9.49
OCDD		3268-87-9	2.4	19
OCDF		39001-02-0	2.76	19
Total tetrachlorinated dioxins		--	--	--
Total pentachlorinated dioxins		--	--	--
Total hexachlorinated dioxins		--	--	--
Total heptachlorinated dioxins		--	--	--
Total tetrachlorinated furans		--	--	--
Total pentachlorinated furans		--	--	--
Total hexachlorinated furans		--	--	--
Total heptachlorinated furans		--	--	--
PCB Congeners, pg/L	Axys/Vista			
Dioxin-like PCB congeners (WHO list)				
PCB-77		32598-13-3	2.66	3.8
PCB-81		70362-50-4	2.83	3.8
PCB-105		32598-14-4	0.843	3.8
PCB-114		74472-37-0	1.54	3.8
PCB-118		31508-00-6	2.31	3.8
PCB-123		65510-44-3	3.04	3.8
PCB-126		57465-28-8	1.39	3.8
PCB-156/157		--	1.17	7.59
PCB-156		38380-08-4	--	--
PCB-157		69782-90-7	--	--
PCB-167		52663-72-6	1.41	3.8
PCB-169		32774-16-6	1.15	3.8
PCB-189		39635-31-9	1.9	3.8
187 non-planar PCB congeners		--	0.775 - 98.2	11.4 - 99.5

Notes:

[a] The MRL generally represents the level of lowest calibration standard (i.e., the practical quantitation limit).

CAS laboratory - Columbia Analytical Services
CAS number - Chemical Abstract Services number
MDL - method detection limit
MRL - method reporting limit
PCB - polychlorinated biphenyl
WHO - World Health Organization

Table A7-1. Laboratory Control Limits for Surrogate Recoveries in Tissue Samples.

Surrogate Compound	Control Limits for Percent Recovery
Columbia Analytical Services	
Butyltin Compounds	
Tri-n-proplytin	10-114
Polycyclic Aromatic Hydrocarbons & Alkylated PAHs	
2-Methylnaphthalene-d10	70-130
Fluoranthene-d10	48-108
Fluorene-d10	40-97
Terphenyl-d14	49-137
Phthalate Esters and Selected SVOCs	
2,4,6-Tribromophenol	47-152
2-Fluorobiphenyl	43-133
2-Fluorophenol	41-112
Fluoranthene-d10	48-108
Fluorene-d10	40-97
Nitrobenzene-d5	35-128
Phenol-d6	43-133
Terphenyl-d14	49-137
Axys Analytical	
Organochlorine pesticides	
$^{13}C_6$-Chlorobenzene	10-130
$^{13}C_6$-1,4-Dichlorobenzene	15-130
$^{13}C_6$-1,2,3-Trichlorobenzene	20 - 130
$^{13}C_6$-1,2,3,4-Tetrachlorobenzene	20-130
$^{13}C_6$-Pentachlorobenzene	20-150
$^{13}C_6$-Hexachlorobenzene	20-150
$^{13}C_6$-beta-HCH	30-150
$^{13}C_6$-delta-HCH	30-150
$^{13}C_6$-gamma-HCH	30-150
$^{13}C_{10}$-Heptachlor	30-150
$^{13}C_{12}$-Aldrin	30-150
$^{13}C_{10}$-Oxychlordane	30-200
$^{13}C_{10}$-trans-Chlordane	30-200
$^{13}C_{12}$-o,p'-DDE	40-150
$^{13}C_{12}$-p,p'-DDE	40-150
$^{13}C_{10}$-trans-Nonachlor	30-150
$^{13}C_{10}$-cis-Nonachlor	30-150

Table A7-1. Laboratory Control Limits for Surrogate Recoveries in Tissue Samples.

Surrogate Compound	Control Limits for Percent Recovery
$^{13}C_{12}$-p,p'-DDD	40-150
$^{13}C_{12}$-o,p'-DDT	40-150
$^{13}C_{12}$-p,p'-DDT	40-150
$^{13}C_{10}$-Mirex	30-150
PCB Congeners	
$^{13}C_{12}$-2-MonoCB	15-150
$^{13}C_{12}$-4-MonoCB	15-150
$^{13}C_{12}$-2,2'-DiCB	25-150
$^{13}C_{12}$-4,4'-DiCB	25-150
$^{13}C_{12}$-2,2',6-TriCB	25-150
$^{13}C_{12}$-3,4,4'-TriCB	25-150
$^{13}C_{12}$-2,2',6,6'-TetraCB	25-150
$^{13}C_{12}$-3,3',4,4'-TetraCB	25-150
$^{13}C_{12}$-3,4,4',5-TetraCB	25-150
$^{13}C_{12}$-2,2',4,6,6'-PentaCB	25-150
$^{13}C_{12}$-2,3,3',4,4'-PentaCB	25-150
$^{13}C_{12}$-2,3,4,4',5-PentaCB	25-150
$^{13}C_{12}$-2,3',4,4',5-PentaCB	25-150
$^{13}C_{12}$-2',3,4,4',5-PentaCB	25-150
$^{13}C_{12}$-3,3',4,4',5-PentaCB	25-150
$^{13}C_{12}$-2,2',4,4',6,6'-HexaCB	25-150
$^{13}C_{12}$-2,3,3',4,4',5-HexaCB	25-150
$^{13}C_{12}$-2,3,3',4,4',5'-HexaCB	25-150
$^{13}C_{12}$-2,3',4,4',5,5'-HexaCB	25-150
$^{13}C_{12}$-3,3',4,4',5,5'-HexaCB	25-150
$^{13}C_{12}$-2,2',3,4',5,6,6'-HeptaCB	25-150
$^{13}C_{12}$-2',3,3',4,4',5,5'-HeptaCB	25-150
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OctaCB	25-150
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OctaCB	25-150
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NonaCB	25-150
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NonaCB	25-150
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DecaCB	25-150

Table A7-1. Laboratory Control Limits for Surrogate Recoveries in Tissue Samples.

Surrogate Compound	Control Limits for Percent Recovery
Dioxins and Furans	
$^{13}C_{12}$-2,3,7,8-TCDD	25-130
$^{13}C_{12}$-2,3,7,8-TCDF	24-130
$^{13}C_{12}$-1,2,3,7,8-PeCDD	25-130
$^{13}C_{12}$-1,2,3,7,8-PeCDF	24-130
$^{13}C_{12}$-2,3,4,7,8-PeCDF	21-130
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	32-130
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	28-130
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	26-130
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	26-123
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	29-130
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	28-130
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	23-130
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	28-130
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	26-130
$^{13}C_{12}$-OCDD	17-130

Note:
Control limits are updated periodically by the laboratories. Control limits that are in effect at the laboratory at the time of analysis will be used for sample analysis and data validation. These may differ slightly from the control limits shown in this table.

Table A7-2. Laboratory Control Limits for Surrogate Recoveries in Collocated Sediment Samples.

Surrogate Compound	Control Limits for Percent Recovery
Columbia Analytical Services	
Butyltins	
Tri-n-proplytin	20-121
Organochlorine pesticides	
Tetrachloro-meta-xylene	19-134
Decachlorobiphenyl	26-144
Polycyclic aromatic hydrocarbons & Alkylated PAHs	
2-Methylnapthalene-d10	70-130
Fluoranthene-d10	10-136
Fluorene-d10	10-123
p-Terphenyl-d14	32-123
Phthalate Esters and Selected SVOCs	
2,4,6-Tribromophenol	16-122
2-Fluorobiphenyl	10-107
2-Fluorophenol	12-88
Nitrobenzene-d5	10-97
Phenol-d6	20-101
Terphenyl-d14	28-135
Dioxins and Furans	
^{13}C-2,3,7,8-TCDD	25-164
^{13}C-1,2,3,7,8-PeCDD	25-181
^{13}C-1,2,3,4,7,8-HxCDD	32-141
^{13}C-1,2,3,6,7,8-HxCDD	28-130
^{13}C-1,2,3,4,6,7,8-HpCDD	23-140
^{13}C-OCDD	17-157
^{13}C-2,3,7,8-TCDF	24-169
^{13}C-1,2,3,7,8-PeCDF	24-185
^{13}C-2,3,4,7,8-PeCDF	21-178
^{13}C-1,2,3,4,7,8-HxCDF	26-152
^{13}C-1,2,3,6,7,8-HxCDF	26-123
^{13}C-2,3,4,6,7,8-HxCDF	28-136
^{13}C-1,2,3,4,6,7,8-HpCDF	28-143
^{13}C-1,2,3,4,7,8,9-HpCDF	26-138
Vista Analytical Laboratory	
PCB Congeners	
^{13}C-2-MonoCB	25-150
^{13}C-4-MonoCB	25-150
^{13}C-2,2'-DiCB	25-150
^{13}C-2,5-DiCB	25-150
^{13}C- 2,2',6-TriCB	25-150

Table A7-2. Laboratory Control Limits for Surrogate Recoveries in Collocated Sediment Samples.

Surrogate Compound	Control Limits for Percent Recovery
^{13}C-2,4,4'-TriCB	25-150
^{13}C-2,4',6-TriCB	25-150
^{13}C-3,4,4'-TriCB	25-150
^{13}C-2,2',5,5'-TetraCB	25-150
^{13}C-2,2',6,6'-TetraCB	25-150
^{13}C-3,3',4,4'-TetraCB	25-150
^{13}C-3,3',4,4'-TetraCB	25-150
^{13}C-2,2',3,5',6-PentaCB	25-150
^{13}C-2,2',4,5,5'-PentaCB	25-150
^{13}C-2,2',4,6,6'-PentaCB	25-150
^{13}C-2,3,3',4,4'-PentaCB	25-150
^{13}C-2,3,4,4',5-PentaCB	25-150
^{13}C-2,3',4,4',5-PentaCB	25-150
^{13}C-2',3,4,4',5-PentaCB	25-150
^{13}C-3,3',4,4',5-PentaCB	25-150
^{13}C-2,2',4,4',5,5'-HexaCB	25-150
^{13}C- 2,2',4,4',6,6'-HexaCB	25-150
^{13}C-2,3,3',4,4',5-HexaCB	25-150
^{13}C-2,3,3',4,4',5'-HexaCB	25-150
^{13}C-2,3',4,4',5,5'-HexaCB	25-150
^{13}C-3,3',4,4',5,5'-HexaCB	25-150
^{13}C-2,2',3,3',4,4',5-HeptaCB	25-150
^{13}C-2,2',3,3',5,5',6-HeptaCB	25-150
^{13}C-2,2',3,4,4',5,5'-HeptaCB	25-150
^{13}C- 2,2',3,4',5,6,6'-HeptaCB	25-150
^{13}C- 2,3,3',4,4',5,5'-HeptaCB	25-150
^{13}C-2,2',3,3',4,4',5,5'-OctaCB	25-150
^{13}C-2,2',3,3',5,5',6,6'-OctaCB	25-150
^{13}C-2,2',3,3',4,4',5,5',6-NonaCB	25-150
^{13}C-2,2',3,3',4,5,5',6,6'-NonaCB	25-150
^{13}C-DecaCB	25-150

Notes:

Control limits are updated periodically by the laboratories. Control limits that are in effect at the laboratory at the time of analysis will be used for sample analysis and data validation. These may differ slightly from the control limits shown in this table.

Table A7-3. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples in Tissue Samples.

Analysis	Accuracy		Precision	
	Matrix Spike Recovery (percent)	LCS Recovery (percent)	Type of Duplicate	Control Limit (RPD)
Columbia Analytical Services				
Conventional Analyses				
Percent moisture	NA	NA	LD	20
Metals				
Aluminum	70-130	85-115	LD/LCSD	30
Antimony	70-130	85-115	LD/LCSD	30
Arsenic	70-130	85-115	LD/LCSD	30
Cadmium	70-130	85-115	LD/LCSD	30
Chromium	70-130	85-115	LD/LCSD	30
Copper	70-130	85-115	LD/LCSD	30
Lead	70-130	85-115	LD/LCSD	30
Manganese	70-130	85-115	LD/LCSD	30
Mercury	60-130	85-115	LD/LCSD	30
Nickel	70-130	85-115	LD/LCSD	30
Selenium	70-130	85-115	LD/LCSD	30
Silver	70-130	85-115	LD/LCSD	30
Thallium	70-130	85-115	LD/LCSD	30
Zinc	70-130	85-115	LD/LCSD	30
Butyltins				
Monobutyltin	10-113	10-115	MSD/LCSD	40
Dibutyltin	10-121	10-94	MSD/LCSD	40
Tributyltin	10-113	10-97	MSD/LCSD	40
Tetrabutyltin	10-125	10-110	MSD/LCSD	40
Polycyclic Aromatic Hydrocarbons				
2-Methylnaphthalene	39-116	12-159	MSD/LCSD	40
Acenaphthene	52-112	44-120	MSD/LCSD	40
Acenaphthylene	44-126	44-124	MSD/LCSD	40
Anthracene	51-121	47-128	MSD/LCSD	40
Benz(a)anthracene	49-127	51-135	MSD/LCSD	40
Benzo(a)pyrene	50-126	49-144	MSD/LCSD	40
Benzo(b)fluoranthene	46-124	54-138	MSD/LCSD	40
Benzo(g,h,i)perylene	46-124	45-138	MSD/LCSD	40
Benzo(k)fluoranthene	51-126	55-141	MSD/LCSD	40
Chrysene	59-123	59-132	MSD/LCSD	40
Dibenz(a,h)anthracene	42-144	36-155	MSD/LCSD	40
Dibenzofuran	57-115	11-168	MSD/LCSD	40
Fluoranthene	49-130	47-139	MSD/LCSD	40
Fluorene	53-120	49-120	MSD/LCSD	40
Indeno(1,2,3-cd)pyrene	30-148	37-148	MSD/LCSD	40
Naphthalene	30-112	42-116	MSD/LCSD	40
Phenanthrene	58-108	49-121	MSD/LCSD	40
Pyrene	58-110	52-129	MSD/LCSD	40
Additional Alkylated Polycyclic Aromatic Hydrocarbons				
1-Methylnaphthalene	46-104	10-174	MSD/LCSD	40
C1-Chrysenes	70-130	70-130	MSD/LCSD	40
C1-Dibenzothiophenes	70-130	70-130	MSD/LCSD	40
C1-Fluoranthenes/Pyrenes	70-130	70-130	MSD/LCSD	40

Table A7-3. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples in Tissue Samples.

Analysis	Accuracy		Precision	
	Matrix Spike Recovery (percent)	LCS Recovery (percent)	Type of Duplicate	Control Limit (RPD)
C1-Fluorenes	70-130	70-130	MSD/LCSD	40
C1-Phenanthrenes/Anthracenes	70-130	70-130	MSD/LCSD	40
C2-Chrysenes	70-130	70-130	MSD/LCSD	40
C2-Dibenzothiophenes	70-130	70-130	MSD/LCSD	40
C2-Fluoranthenes/Pyrenes	70-130	70-130	MSD/LCSD	40
C2-Fluorenes	70-130	70-130	MSD/LCSD	40
C2-Naphthalenes	70-130	70-130	MSD/LCSD	40
C2-Phenanthrenes/Anthracenes	70-130	70-130	MSD/LCSD	40
C3-Chrysenes	70-130	70-130	MSD/LCSD	40
C3-Dibenzothiophenes	70-130	70-130	MSD/LCSD	40
C3-Fluoranthenes/Pyrenes	70-130	70-130	MSD/LCSD	40
C3-Fluorenes	70-130	70-130	MSD/LCSD	40
C3-Naphthalenes	70-130	70-130	MSD/LCSD	40
C3-Phenanthrenes/Anthracenes	70-130	70-130	MSD/LCSD	40
C4-Chrysenes	70-130	70-130	MSD/LCSD	40
C4-Naphthalenes	70-130	70-130	MSD/LCSD	40
C4-Phenanthrenes/Anthracenes	70-130	70-130	MSD/LCSD	40
Phthalate esters				
Bis(2-ethylhexyl) phthalate	53-172	39-174	MSD/LCSD	40
Butyl benzyl phthalate	39-171	56-145	MSD/LCSD	40
Diethyl phthalate	60-139	61-133	MSD/LCSD	40
Dimethyl phthalate	68-124	60-120	MSD/LCSD	40
Di-n-butyl-phthalate	47-161	60-146	MSD/LCSD	40
Di-n-octyl phthalate	35-184	49-156	MSD/LCSD	40
Semivolatile organic compounds				
1,2,4-Trichlorobenzene	59-109	41-114	MSD/LCSD	40
1,2-Dichlorobenzene	51-106	41-117	MSD/LCSD	40
1,2-Diphenylhydrazine	70-130	53-104	MSD/LCSD	40
1,3-Dichlorobenzene	43-104	41-112	MSD/LCSD	40
1,4-Dichlorobenzene	45-101	41-109	MSD/LCSD	40
2,4-Dinitrotoluene	73-137	54-136	MSD/LCSD	40
2,6-Dinitrotoluene	71-138	48-138	MSD/LCSD	40
2-Chloronaphthalene	37-137	39-133	MSD/LCSD	40
2-Nitroaniline	62-131	47-122	MSD/LCSD	40
3,3'-Dichlorbenzidine	70-130	35-135	MSD/LCSD	40
3-Nitroaniline	10-122	50-121	MSD/LCSD	40
4-Bromophenyl phenyl ether	66-119	57-123	MSD/LCSD	40
4-Chloroaniline	10-104	39-95	MSD/LCSD	40
4-Chlorophenyl phenyl ether	63-123	58-115	MSD/LCSD	40
4-Methylphenol	10-103	34-93	MSD/LCSD	40
4-Nitroaniline	10-122	51-134	MSD/LCSD	40
Aniline	70-130	10-78	MSD/LCSD	40
Benzoic Acid	10-214	10-214	MSD/LCSD	40
Benzyl alcohol	46-133	30-122	MSD/LCSD	40
Bis-(2-chloroethoxy) methane	56-124	46-116	MSD/LCSD	40
Bis-(2-chloroethyl) ether	56-112	52-109	MSD/LCSD	40
Bis(2-chloroisopropyl) ether	10-105	39-91	MSD/LCSD	40
Hexachlorobenzene	62-121	59-124	MSD/LCSD	40
Hexachlorobutadiene	43-114	51-105	MSD/LCSD	40

Table A7-3. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples in Tissue Samples.

Analysis	Accuracy		Precision	
	Matrix Spike Recovery (percent)	LCS Recovery (percent)	Type of Duplicate	Control Limit (RPD)
Hexachlorocyclopentadiene	10-105	19-123	MSD/LCSD	40
Hexachloroethane	29-116	51-110	MSD/LCSD	40
Isophorone	61-147	54-127	MSD/LCSD	40
Nitrobenzene	54-119	48-117	MSD/LCSD	40
n-Nitrosodimethylamine	55-111	24-115	MSD/LCSD	40
n-Nitroso-di-n-propylamine	48-139	42-132	MSD/LCSD	40
n-Nitrosodiphenylamine	71-132	58-132	MSD/LCSD	40
Phenols				
Phenol	70-130	39-124	MSD	40
2-Chlorophenol	21-107	40-100	MSD	40
2,4-Dichlorophenol	17-106	40-97	MSD	40
2,4,5-Trichlorophenol	11-107	42-90	MSD	40
2,4,6-Trichlorophenol	10-91	10-92	MSD	40
2,3,4,6-Tetrachlorophenol	10-134	13-119	MSD	40
Tetrachlorophenol (2,3,4,5 and 2,3,5,6)	16-92	40-89	MSD	40
Pentachlorophenol	10-98	34-90	MSD	40
2-Methylphenol	13-99	42-91	MSD	40
2,4-Dimethylphenol	--	--	MSD	40
2-Nitrophenol	18-104	40-98	MSD	40
4-Nitrophenol	10-129	40-113	MSD	40
2,4-Dinitrophenol	10-138	32-104	MSD	40
4-Chloro-3-methylphenol	21-92	43-90	MSD	40
4,6-Dinitro-2-methylphenol	--	--	MSD	40
Axys Analytical Services				
Lipids	NA	NA	LD/LCSD	20
Organochlorine Pesticides				
2,4'-DDD	70-130	70-130	MSD/LCSD	40
2,4'-DDE	70-130	70-130	MSD/LCSD	40
2,4'-DDT	70-130	70-130	MSD/LCSD	40
4,4'-DDD	70-130	70-130	MSD/LCSD	40
4,4'-DDE	70-130	70-130	MSD/LCSD	40
4,4'-DDT	70-130	70-130	MSD/LCSD	40
Aldrin	70-130	70-130	MSD/LCSD	40
alpha-BHC	70-130	70-130	MSD/LCSD	40
beta-BHC	70-130	70-130	MSD/LCSD	40
delta-BHC	60-130	60-130	MSD/LCSD	40
gamma-BHC (Lindane)	70-130	70-130	MSD/LCSD	40
alpha-Chlordane	70-130	70-130	MSD/LCSD	40
gamma-Chlordane	70-130	70-130	MSD/LCSD	40
Oxychlordane	70-130	70-130	MSD/LCSD	40
cis -Nonachlor	70-130	70-130	MSD/LCSD	40
trans -Nonachlor	70-130	70-130	MSD/LCSD	40
Dieldrin	60-130	60-130	MSD/LCSD	40
Endosulfan I	70-130	70-130	MSD/LCSD	40
Endosulfan II	70-130	70-130	MSD/LCSD	40
Endosulfan sulfate	70-130	70-130	MSD/LCSD	40
Endrin	60-130	60-130	MSD/LCSD	40
Endrin aldehyde	50-130	50-130	MSD/LCSD	40

Table A7-3. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples in Tissue Samples.

Analysis	Accuracy		Precision	
	Matrix Spike Recovery (percent)	LCS Recovery (percent)	Type of Duplicate	Control Limit (RPD)
Endrin ketone	60-130	60-130	MSD/LCSD	40
Heptachlor	70-130	70-130	MSD/LCSD	40
Heptachlor epoxide	60-130	60-130	MSD/LCSD	40
Methoxychlor	60-130	60-130	MSD/LCSD	40
Toxaphene	70-130	70-130	MSD/LCSD	40
Hexachlorobenzene	70-130	70-130	MSD/LCSD	40
Hexachlorobutadiene	--	--	MSD/LCSD	40
Hexachloroethane	70-130	70-130	MSD/LCSD	40
Mirex	70-130	70-130	MSD/LCSD	40
Dioxins and Furans				
1,2,3,4,6,7,8-HpCDD	NA	70-130	LCSD	40
1,2,3,4,6,7,8-HpCDF	NA	82-122	LCSD	40
1,2,3,4,7,8,9-HpCDF	NA	78-130	LCSD	40
1,2,3,4,7,8-HxCDD	NA	70-130	LCSD	40
1,2,3,4,7,8-HxCDF	NA	72-130	LCSD	40
1,2,3,6,7,8-HxCDD	NA	76-130	LCSD	40
1,2,3,6,7,8-HxCDF	NA	84-130	LCSD	40
1,2,3,7,8,9-HxCDD	NA	70-130	LCSD	40
1,2,3,7,8,9-HxCDF	NA	78-130	LCSD	40
1,2,3,7,8-PeCDD	NA	70-130	LCSD	40
1,2,3,7,8-PeCDF	NA	80-130	LCSD	40
2,3,4,6,7,8-HxCDF	NA	70-130	LCSD	40
2,3,4,7,8-PeCDF	NA	70-130	LCSD	40
2,3,7,8-TCDD	NA	70-130	LCSD	40
2,3,7,8-TCDF	NA	75-130	LCSD	40
OCDD	NA	78-130	LCSD	40
OCDF	NA	70-130	LCSD	40
Total tetrachlorinated dioxins	--	--	LCSD	40
Total pentachlorinated dioxins	--	--	LCSD	40
Total hexachlorinated dioxins	--	--	LCSD	40
Total heptachlorinated dioxins	--	--	LCSD	40
Total tetrachlorinated furans	--	--	LCSD	40
Total pentachlorinated furans	--	--	LCSD	40
Total hexachlorinated furans	--	--	LCSD	40
Total heptachlorinated furans	--	--	LCSD	40

Table A7-3. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples in Tissue Samples.

Analysis	Accuracy		Precision	
	Matrix Spike Recovery (percent)	LCS Recovery (percent)	Type of Duplicate	Control Limit (RPD)
PCB Congeners (209)				
Dioxin-like PCB congeners (WHO list)				
PCB-77	NA	50-150	LCSD	40
PCB-81	NA	50-150	LCSD	40
PCB-105	NA	50-150	LCSD	40
PCB-114	NA	50-150	LCSD	40
PCB-118	NA	50-150	LCSD	40
PCB-123	NA	50-150	LCSD	40
PCB-126	NA	50-150	LCSD	40
PCB-156/157	NA	50-150	LCSD	40
PCB-167	NA	50-150	LCSD	40
PCB-169	NA	50-150	LCSD	40
PCB-170	NA	50-150	LCSD	40
PCB-180	NA	50-150	LCSD	40
PCB-189	NA	50-150	LCSD	40
187 non-planar PCB congeners	NA	50-150	LCSD	40

Notes:

LCS - laboratory control sample
LCSD - laboratory control sample duplicate
LD - laboratory duplicate
MSD - matrix spike duplicate
NA - Not applicable
RPD - relative percent difference
WHO - World Health Organization

Control limits are updated periodically by the laboratories and may differ slightly from the control limits shown in this table. During data validation, low recoveries will be evaluated for analytes with wide laboratory control limits to evaluate potential bias of the data. Data will be qualified if a bias is identifed.

Table A7-4. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples in Sediment Samples.

Analysis	Accuracy		Precision	
	Matrix Spike Recovery (percent)	LCS Recovery (percent)	Type of Duplicate	Control Limit RPD
Columbia Analytical Services				
Conventional Analyses				
Grain size	NA	NA	Triplicate	Note-1
Total organic carbon	75-125	85-115	LD	20
Total solids	NA	NA	LD	20
Metals				
Aluminum	75-125	61-152	LD	30
Antimony	10-125	32-162	LD	30
Arsenic	61-128	80-115	LD	30
Cadmium	79-127	79-127	LD	30
Chromium	22-184	93-123	LD	30
Copper	51-147	85-118	LD	30
Lead	51-155	81-129	LD	30
Manganese	70-130	80-120	LD	30
Mercury	60-123	75-118	LD	30
Nickel	74-126	92-123	LD	30
Selenium	45-132	76-136	LD	30
Silver	72-121	76-128	LD	30
Thallium	82-123	82-123	LD	30
Zinc	32-168	88-126	LD	30
Butyltins				
Monobutyltin	10-78	10-101	MSD	40
Dibutyltin	10-150	27-163	MSD	40
Tributyltin	10-146	26-131	MSD	40
Tetrabutyltin	11-151	17-135	MSD	40
Organochlorine pesticides				
2,4'-DDD	39-139	64-131	MSD	40
2,4'-DDE	45-135	59-133	MSD	40
2,4'-DDT	40-136	66-130	MSD	40
4,4'-DDD	32-156	74-130	MSD	40
4,4'-DDE	35-146	73-126	MSD	40
4,4'-DDT	31-161	75-132	MSD	40
Aldrin	39-143	67-120	MSD	40
alpha-BHC	41-148	67-130	MSD	40
alpha-Chlordane	40-140	72-116	MSD	40
beta-BHC	37-152	66-134	MSD	40
cis -Nonachlor	70-130	61-121	MSD	40
delta-BHC	35-162	78-139	MSD	40
Dieldrin	48-142	74-121	MSD	40
Endosulfan I	10-141	50-112	MSD	40
Endosulfan II	21-135	59-116	MSD	40
Endosulfan sulfate	39-148	70-124	MSD	40
Endrin	44-146	76-127	MSD	40
Endrin aldehyde	18-137	29-138	MSD	40
Endrin ketone	37-149	72-123	MSD	40
gamma-BHC (Lindane)	45-153	70-125	MSD	40
gamma-Chlordane	33-161	74-117	MSD	40
Heptachlor	35-151	69-120	MSD	40

Table A7-4. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples in Sediment Samples.

Analysis	Accuracy		Precision	
	Matrix Spike Recovery (percent)	LCS Recovery (percent)	Type of Duplicate	Control Limit RPD
Heptachlor epoxide	37-148	70-117	MSD	40
Hexachlorobenzene	70-130	36-114	MSD	40
Hexachlorobutadiene	70-130	70-130	MSD	40
Hexachloroethane	70-130	70-130	MSD	40
Methoxychlor	35-158	68-137	MSD	40
Mirex	68-119	43-136	MSD	40
Oxychlordane	70-130	55-115	MSD	40
Toxaphene	52-156	52-142	MSD	40
trans -Nonachlor	70-130	58-117	MSD	40
Polycyclic Aromatic Hydrocarbons				
2-Methylnaphthalene	13-126	42-121	MSD/LCSD	40
Acenaphthene	18-125	50-110	MSD/LCSD	40
Acenaphthylene	21-121	50-111	MSD/LCSD	40
Anthracene	19-133	52-115	MSD/LCSD	40
Benz(a)anthracene	12-139	51-118	MSD/LCSD	40
Benzo(a)pyrene	10-148	56-122	MSD/LCSD	40
Benzo(b)fluoranthene	12-144	55-125	MSD/LCSD	40
Benzo(g,h,i)perylene	10-148	49-125	MSD/LCSD	40
Benzo(k)fluoranthene	11-145	55-124	MSD/LCSD	40
Chrysene	12-145	54-120	MSD/LCSD	40
Dibenz(a,h)anthracene	12-143	37-135	MSD/LCSD	40
Dibenzofuran	21-126	50-115	MSD/LCSD	40
Fluoranthene	10-149	55-121	MSD/LCSD	40
Fluorene	22-125	52-112	MSD/LCSD	40
Indeno(1,2,3-cd)pyrene	10-151	42-133	MSD/LCSD	40
Naphthalene	10-121	48-107	MSD/LCSD	40
Phenanthrene	10-143	53-112	MSD/LCSD	40
Pyrene	10-150	47-129	MSD/LCSD	40
Additional Alkylated Polycyclic Aromatic Hydrocarbons				
1-Methylnaphthalene	23-121	46-119	MSD/LCSD	40
C1-Chrysenes	70-130	70-130	MSD/LCSD	40
C1-Dibenzothiophenes	70-130	70-130	MSD/LCSD	40
C1-Fluoranthenes/Pyrenes	70-130	70-130	MSD/LCSD	40
C1-Fluorenes	70-130	70-130	MSD/LCSD	40
C1-Phenanthrenes/Anthracenes	70-130	70-130	MSD/LCSD	40
C2-Chrysenes	70-130	70-130	MSD/LCSD	40
C2-Dibenzothiophenes	70-130	70-130	MSD/LCSD	40
C2-Fluoranthenes/Pyrenes	70-130	70-130	MSD/LCSD	40
C2-Fluorenes	70-130	70-130	MSD/LCSD	40
C2-Naphthalenes	70-130	70-130	MSD/LCSD	40
C2-Phenanthrenes/Anthracenes	70-130	70-130	MSD/LCSD	40
C3-Chrysenes	70-130	70-130	MSD/LCSD	40
C3-Dibenzothiophenes	70-130	70-130	MSD/LCSD	40
C3-Fluoranthenes/Pyrenes	70-130	70-130	MSD/LCSD	40
C3-Fluorenes	70-130	70-130	MSD/LCSD	40
C3-Naphthalenes	70-130	70-130	MSD/LCSD	40
C3-Phenanthrenes/Anthracenes	70-130	70-130	MSD/LCSD	40
C4-Chrysenes	70-130	70-130	MSD/LCSD	40
C4-Naphthalenes	70-130	70-130	MSD/LCSD	40
C4-Phenanthrenes/Anthracenes	70-130	70-130	MSD/LCSD	40

Table A7-4. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples in Sediment Samples.

Analysis	Accuracy		Precision	
	Matrix Spike Recovery (percent)	LCS Recovery (percent)	Type of Duplicate	Control Limit RPD
Phthalate esters				
Bis(2-ethylhexyl) phthalate	10-150	47-124	MSD	40
Butyl benzyl phthalate	21-130	48-119	MSD	40
Dibutyl phthalate	15-133	51-111	MSD	40
Diethyl phthalate	23-123	48-107	MSD	40
Dimethyl phthalate	24-118	48-99	MSD	40
Di-n-octyl phthalate	28-122	41-123	MSD	40
Semivolatile Organic Compounds, µg/kg				
1,2,4-Trichlorobenzene	10-102	43-89	MSD	40
1,2-Dichlorobenzene	10-96	45-89	MSD	40
1,2-Diphenylhydrazine	70-130	53-104	MSD	40
1,3-Dichlorobenzene	10-92	42-86	MSD	40
1,4-Dichlorobenzene	10-91	42-84	MSD	40
2,4-Dinitrotoluene	16-127	50-114	MSD	40
2,6-Dinitrotoluene	22-117	50-105	MSD	40
2-Chloronaphthalene	--	--	MSD	40
2-Nitroaniline	20-116	42-104	MSD	40
3,3'-Dichlorbenzidine	10-98	10-109	MSD	40
3-Nitroaniline	10-105	38-103	MSD	40
4-Bromophenyl phenyl ether	10-121	46-101	MSD	40
4-Chloroaniline	10-83	17-95	MSD	40
4-Chlorophenyl phenyl ether	10-118	45-101	MSD	40
4-Methylphenol	10-103	34-93	MSD	40
4-Nitroaniline	10-109	38-107	MSD	40
Aniline	10-73	10-80	MSD	40
Benzoic Acid	10-127	10-100	MSD	40
Benzyl alcohol	26-93	41-93	MSD	40
Bis-(2-chloroethoxy) methane	14-103	44-90	MSD	40
Bis-(2-chloroethyl) ether	10-106	44-89	MSD	40
Bis(2-chloroisopropyl) ether	10-105	39-91	MSD	40
Hexachlorobenzene	15-120	49-104	MSD	40
Hexachlorobutadiene	10-123	41-88	MSD	40
Hexachlorocyclopentadiene	10-80	23-92	MSD	40
Hexachloroethane	10-89	37-90	MSD	40
Isophorone	29-108	47-101	MSD	40
Nitrobenzene	20-92	40-91	MSD	40
n-Nitrosodimethylamine	21-101	31-103	MSD	40
n-Nitroso-di-n-propylamine	18-111	40-100	MSD	40
n-Nitrosodiphenylamine	22-130	47-108	MSD	40
Phenols, ug/kg				
2,3,4,6-Tetrachlorophenol	70-130	39-124	MSD	40
2,4,5-Trichlorophenol	21-107	40-100	MSD	40
2,4,6-Trichlorophenol	17-106	40-97	MSD	40
2,4-Dichlorophenol	11-107	42-90	MSD	40
2,4-Dimethylphenol	10-91	10-92	MSD	40
2,4-Dinitrophenol	10-134	13-119	MSD	40
2-Chlorophenol	16-92	40-89	MSD	40
2-Methylphenol	10-98	34-90	MSD	40
2-Nitrophenol	13-99	42-91	MSD	40
4,6-Dinitro-2-methylphenol	--	--	MSD	40

Table A7-4. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples in Sediment Samples.

Analysis	Accuracy		Precision	
	Matrix Spike Recovery (percent)	LCS Recovery (percent)	Type of Duplicate	Control Limit RPD
4-Chloro-3-methylphenol	18-104	40-98	MSD	40
4-Nitrophenol	10-129	40-113	MSD	40
Pentachlorophenol	10-138	32-104	MSD	40
Phenol	21-92	43-90	MSD	40
Tetrachlorophenol (2,3,4,5 and 2,3,5,6)	--	--	MSD	40
Chlorinated Dioxins and Furans				
1,2,3,4,6,7,8-HpCDD	50-150	50-150	LCSD	50
1,2,3,4,6,7,8-HpCDF	50-150	50-150	LCSD	50
1,2,3,4,7,8,9-HpCDF	50-150	50-150	LCSD	50
1,2,3,4,7,8-HxCDD	50-150	50-150	LCSD	50
1,2,3,4,7,8-HxCDF	50-150	50-150	LCSD	50
1,2,3,6,7,8-HxCDD	50-150	50-150	LCSD	50
1,2,3,6,7,8-HxCDF	50-150	50-150	LCSD	50
1,2,3,7,8,9-HxCDD	50-150	50-150	LCSD	50
1,2,3,7,8,9-HxCDF	50-150	50-150	LCSD	50
1,2,3,7,8-PeCDD	50-150	50-150	LCSD	50
1,2,3,7,8-PeCDF	50-150	50-150	LCSD	50
2,3,4,6,7,8-HxCDF	50-150	50-150	LCSD	50
2,3,4,7,8-PeCDF	50-150	50-150	LCSD	50
2,3,7,8-TCDD	50-150	50-150	LCSD	50
2,3,7,8-TCDF	50-150	50-150	LCSD	50
OCDD	50-150	50-150	LCSD	50
OCDF	50-150	50-150	LCSD	50
Total heptachlorinated dioxins	--	--	LCSD	50
Total heptachlorinated furans	--	--	LCSD	50
Total hexachlorinated dioxins	--	--	LCSD	50
Total hexachlorinated furans	--	--	LCSD	50
Total pentachlorinated dioxins	--	--	LCSD	50
Total pentachlorinated furans	--	--	LCSD	50
Total tetrachlorinated dioxins	--	--	LCSD	50
Total tetrachlorinated furans	--	--	LCSD	50

Table A7-4. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples in Sediment Samples.

Analysis	Accuracy		Precision	
	Matrix Spike Recovery (percent)	LCS Recovery (percent)	Type of Duplicate	Control Limit RPD
Alta Analytical Services				
PCB Congeners				
Dioxin-like PCB congeners (WHO list)	NA	50-150	LD	50
PCB-77	NA	50-150	LD	50
PCB-81	NA	50-150	LD	50
PCB-105	NA	50-150	LD	50
PCB-114	NA	50-150	LD	50
PCB-118	NA	50-150	LD	50
PCB-123	NA	50-150	LD	50
PCB-126	NA	50-150	LD	50
PCB-156	NA	50-150	LD	50
PCB-157	NA	50-150	LD	50
PCB-167	NA	50-150	LD	50
PCB-169	NA	50-150	LD	50
PCB-170	NA	50-150	LD	50
PCB-180	NA	50-150	LD	50
PCB-189	NA	50-150	LD	50
187 non-planar PCB congeners	NA	50-150	LD	50

Notes:

Note 1 - RPD control limit is not applicable. Laboratory control limit is ± 10 percent in the weight of the fraction.

LCS - laboratory control sample
LCSD - laboratory control sample duplicate
LD - laboratory duplicate
MSD - matrix spike duplicate
NA - Not applicable
RPD - relative percent difference
WHO - World Health Organization

Control limits are updated periodically by the laboratories. Control limits that are in effect at the laboratory at the time of analysis will be used for sample analysis and data validation. These may differ slightly from the control limits shown in this table.

Table B2-1. Sample Containers, Preservation, Holding Times, and Sample Volume Requirements.

Analysis	Lab	Container Type	Jar	Sample Size[a,b]	Preservation	Holding Time
Fish and Shellfish Tissue						
Polycyclic aromatic hydrocarbons [c]	CAS	WMG	2 oz.	10g	Deep Frozen (-20±4°C)	1 year
SVOCs, including phthalate esters and phenols	CAS			10g		1 year
Mercury	CAS	WMG	2 oz.	5g	Deep Frozen (-20±4°C)	6 months [d]
Metals and percent moisture	CAS			5g		1 year
Butyltin compounds	CAS			10g		1 year
PCB congeners [e]	Axys	WMG	2 oz.	10g	Deep Frozen (-20±4°C)	1 year
PCDD/PCDFs [e]	Axys			10g		
Pesticides and lipids [e]	Axys			10g		
Archival	CAS	WMG	Four 4-oz jars	N/A	Deep Frozen (-20±4°C)	N/A
Sediment Samples						
Grain size [f]	CAS	G/P	8 oz	100g	4±2°C	6 months
Total organic carbon	CAS	G/P	8 oz	1g	4±2°C	28 days [g]
Mercury	CAS	WMG	16 oz	5g	4±2°C	28 days [d]
Metals and total solids	CAS			10g		6 months [g]
Butyltins	CAS			20g		14 days [g]
PAHs [c], SVOCs (including phthalates and phenols)	CAS			50g		14 days [g]
Pesticides, PCB Aroclors	CAS			40g		14 days [g]
PCDDs/PCDFs	CAS	WMG	8 oz	50g	4±2°C	30 days [g]
PCB congeners	Vista	WMG	8 oz	10g	Deep Frozen (-20°C)	1 year
Archival	CAS	WMG	Two 8-oz jars	N/A	Deep Frozen (-20°C)	1 year

Table B2-1. Sample Containers, Preservation, Holding Times, and Sample Volume Requirements.

Analysis	Lab	Container Type	Jar	Sample Size[a,b]	Preservation	Holding Time
Equipment Rinsate Blanks for Sediment Collection and Tissue Homogenization						
Metals and Mercury	CAS	HDPE	500 mL	100 mL	5 ml of 1:1&	6 months
Mercury	CAS			100 mL	HNO_3 & 4±2°C	28 days
Butyltins	CAS	Polycarbonate	500 mL	500 mL	Dark; 4±2°C	7 days
SVOCS (including PAHs, phthalates, and phenols)	CAS	AG	500 mL	500 mL	Dark; 4±2°C	7 days
Pesticides	Axys, CAS	AG	500 mL	500 mL	Dark; 4±2°C	7 days
PCBs	Axys, CAS	AG	500 mL	500 mL	Dark; 4±2°C	7 days
PCDD/Fs	Axys, CAS	AG	500 mL	500 mL	Dark; 4±2°C	1 year
PCB congeners	Axys, Vista	AG	500 mL	500 mL	Sulfuric acid to pH 2-3; 4±2°C	1 year

Notes:

WMG = wide mouth glass
HDPE = high density polyethylene
AG = amber glass
G/P = glass or plastic

[a] The size and number of containers may be modified by analytical laboratory.

[b] Matrix spike samples and duplicates will be prepared by the laboratory only if sufficient sample volume is available.

[c] Clam tissue samples and collocated sediments associated with clam samples will be analyzed for alkylated PAHs.

[d] The holding time for mercury in frozen samples is 180 days, as approved by EPA (2002, pers. comm.).

[e] For clam and sculpin samples, PCB congeners, PCDD/Fs, pesticides and lipids will be analyzed using a single 10-g sample for samples with limited tissue mass.

[f] For grain-size analysis, 16 oz. of sediment will be collected for lab QC at 5% of the stations. 16 oz. of sediment will also be collected for sandy samples.

[g] Holding times for frozen samples are as follows: total organic carbon, 1 year; metals (except mercury), 2 years; butyltin species, 1 year; PAHs, SVOCs, pesticides, PCB Aroclors, and PCDD/Fs, 1 year.