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May 22, 2014

Via E-Mail

Sean Sheldrake
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Re: Supplemental RI/FS Work at the River Mile 11E Project Area
Portland Harbor Superfund Site (PHSS)
Transmittal of the Final Porewater Sampling and Analysis Plan

Dear Mr. Sheldrake:

Attached for your review and approval is the Final Porewater Sampling and Analysis Plan (SAP) for the River Mile 11E Project Area. The Final SAP addresses the final round of comments that the U.S. Environmental Protection Agency (EPA) transmitted to the River Mile (RM) 11E on April 25, 2014. To assist with the review of the document, we have attached a copy of the comment table that EPA and the RM11E Group have been exchanging that denotes (in red) where the SAP has been updated to address each of EPA's comments.

Please do not hesitate to contact me if you have any questions.

Sincerely yours,

Jacqueline Thiell Wetzsteon
RM11E Project Coordinator

ec: River Mile 11E Respondents
AOC Notice Recipients (Paragraph 97.c through m)
Paul Fuglevand

Porewater Sampling and Analysis Plan

River Mile 11 East

Willamette River
Portland, Oregon

May 2014

Prepared for

RM11E GROUP

Prepared by

SEE LLC
Science & Engineering for the Environment

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GSI Water Solutions, Inc.

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Attachment 6	SOP Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Bio solids and Tissue by Isotope Dilution HRGC/HRMS. ALS SOP Code: HMS-1668
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Abbreviations and Acronyms

µg/kg	microgram(s) per kilogram
ALS	ALS Environmental
AOPC	Area of Potential Concern
BMC	Ballard Marine Construction
bml	below mudline
Cargill	Cargill, Inc.
CFR	Code of Federal Regulations
City	City of Portland
cm	centimeters
COC	contaminants of concern
dGPS	differential global positioning system
DM	Data Manager
DOD	U.S. Department of Defense
DOF	Dalton, Olmsted & Fuglevand, Inc.
EMNR	Enhanced Monitored Natural Recovery
EPA	U.S. Environmental Protection Agency
EqP	equilibrium partitioning model
FD	Field Director
FS	feasibility study
Glacier NW	Glacier Northwest, Inc.
GPS	global positioning system
GSI	GSI Water Solutions, Inc.
HSP	Health and Safety Plan
ID	identification number
IDW	investigation-derived waste
LWG	Lower Willamette Group
MDL	method detection limit
MIT	Massachusetts Institute of Technology
MRL	method reporting limit
NAD 83	North American Datum of 1983
NAVD 88	North American Vertical Datum of 1988
ng/kg	nanograms per kilogram
ng/L	nanograms per liter
ORS	Oregon Revised Statute
PCB	polychlorinated biphenyl
PE	polyethylene
PED	polyethylene device

PM	Project Manager
Portland Harbor	Portland Harbor Superfund Site
PRC	performance reference compounds
PRG	Preliminary Remediation Goal
PSD	passive sampling device
QA	quality assurance
QAPP	Quality Assurance Project Plan
QA/QC	quality assurance/quality control
QC	quality control
RAL	Remedial Action Level
RAO	remedial action objectives
RI	remedial investigation
RI/FS	remedial investigation and feasibility study
RM	river mile
RM11E	River Mile 11 East
RM11E Group	Cargill, Inc.; CBS Corporation; City of Portland; DIL Trust; Glacier Northwest, Inc.; and PacifiCorp
RPM	Remedial Project Manager
RSS	Research Support Systems, Inc.
SAC	Sampling and Analysis Coordinator
SAP	sampling and analysis plan
SCRA	Site Characterization and Risk Assessment
SEE	Science and Engineering for the Environment, LLC
SOP	standard operating procedure
SOW	statement of work
SRM	Standard Reference Material
TBD	to be determined
TOC	total organic carbon
TPL	Technical Project Lead
TS	total solids
USGS	U.S. Geological Survey
Willamette CRA	Willamette Cultural Resources Associates, LTD
Work Plan	Supplemental Remedial Investigation and Feasibility Study Work Plan

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Section 1. Introduction

This Porewater Sampling and Analysis Plan (SAP), prepared for the River Mile 11 East (RM11E) Project Area, is submitted by Cargill, Inc. (Cargill); CBS Corporation; City of Portland (City); DIL Trust; Glacier Northwest, Inc. (Glacier NW); and PacifiCorp, collectively referred to as the RM11E Group. This SAP was prepared on behalf of the RM11E Group by Science and Engineering for the Environment, LLC (SEE); Dalton, Olmstead and Fuglevand, Inc. (DOF); and GSI Water Solutions, Inc. (GSI).

This SAP is part of the RM11E Supplemental Remedial Investigation and Feasibility Study (Supplemental RI/FS). The Final Supplemental RI/FS Work Plan for RM11E (Work Plan; GSI and DOF, 2013) provides a detailed description of the work being conducted pursuant to the Statement of Work (SOW) contained within the Administrative Settlement Agreement and Order on Consent (Settlement Agreement) (U.S. Environmental Protection Agency [EPA] Region 10, CERCLA Docket No. 10-2013-0087). The RM11E investigation supplements the Portland Harbor Superfund Site (Portland Harbor) Draft Final Remedial Investigation Report (Draft Final RI Report; Integral et al., 2011) and the Draft Feasibility Study Report (Draft FS Report; Anchor QEA et al., 2012) to inform selection and design of a final remedy at the RM11E Project Area (Integral et al., 2011; Anchor QEA et al., 2012). The submission of this SAP is in response to EPA's communications to the RM11E Group in September 2013 requiring the performance of a porewater investigation as part of the data gathering effort under the Work Plan.

The RM11E Project Area is shown in Figure 1-1. The project area lies between approximately RM 10.9 and RM 11.6 along the east bank of the Willamette River and includes Area of Potential Concern (AOPC) 25 (from the Draft FS for Portland Harbor) and the riverbank area to the top of the bank.

1.1 Purpose and Objectives

The purpose of the work described in this SAP is to meet a specific data need identified in Section 2.4 of the SOW:

> *Porewater and Empirical Data to Inform Cap Design: Porewater quality, hydraulic gradients, sediment texture, and other empirical data will be collected and evaluated for input into a sediment cap isolation (advection/diffusion) model in order to inform cap design.*

This SAP is for the collection of porewater and associated sediment data for the purpose of supporting sediment cap isolation modeling, which will inform remedy selection and cap design. The objective of this SAP is to collect data for use as input parameters into models to support remedial selection and design for alternatives defined in the Draft FS Report. Site remedies that may be evaluated for RM11E included Enhanced Monitored Natural Recovery

(EMNR), engineered cap, in situ treatment, and removal[1]. These evaluations will be used for both the Recontamination Assessment and the Implementability Study, as described in the Work Plan.

1.2 Summary of Existing Sediment Data

Extensive bedded sediment, suspended sediment, and bank soil data have been collected in the RM11E Project Area. As part of the Portland Harbor RI/FS process, the Lower Willamette Group (LWG) and the City have collected and analyzed more than 60 surface sediment grab samples in the RM11E Project Area. Several additional surface sediment samples have been collected by shoreline property owners to support activities such as environmental permitting and maintenance dredging at waterfront facilities. Previous sediment investigations that have been conducted in the RM11E Project Area are described in Section 3.3 and Section 6.2.1 of the Work Plan. Existing sediment data are included in the LWG's Site Characterization and Risk Assessment (SCRA) Database and the FS Sediment Database, and are discussed in the Draft Final RI Report and Draft FS Report. New sediment data collected by the RM11E Group as part of the Supplemental RI/FS will be documented in the forthcoming Field and Data Report, but new data results germane to this porewater investigation have been incorporated into this SAP where applicable.

1.3 SAP Preparation

This SAP is consistent with the approach and methodologies set forth in Appendix A (Quality Assurance Project Plan) and Appendix C (Surface Sediment Sampling and Analysis Plan Addendum) of the Work Plan (GSI and DOF, 2013).

The results of the Phase 1 Porewater Characterization, as described in Section 6.2.4.1 of the Work Plan, will be presented in the Porewater Characterization Report.

[1] Removal as an alternative will be evaluated both as dredging to a clean z-layer, and dredging to a specified depth coupled with a sand cover, an engineered cap, or the addition of an amendment as part of an engineered cap at the bottom of the dredge cut.

Section 2. Porewater Study Design

The purpose of the porewater study is to provide additional data to inform remedy selection, and cap design, consistent with Section 2.4 of the SOW. Polychlorinated biphenyls (PCB) in sediments principally have defined the proposed remedial alternative footprint for RM11E in the Draft FS Report (Anchor et al., 2012). As such, PCBs are the focus of this porewater study.

The general approach to the project involves the following key steps:

1. Selection of analytical tools (models) that will be used to meet project objectives.
2. Identification of data needs to meet the model input requirements.
3. Identification of sampling methods to satisfy data needs.
4. Identification of sampling locations that will provide data representative of a range of site conditions.
5. Sample collection and laboratory analysis.

Steps 1 to 4 are described in this section. Step 5 is detailed in Section 5.

2.1 Data to Inform Remedy Selection

The design of the porewater sampling program at RM11E is intended to provide measures of the flux of PCBs within sediments, and a measure of the freely dissolved PCBs that move out of the sediment and into the river. Selection and design of specific remedies within RM11E will consider bulk sediment concentrations in the surface and subsurface sediments, as well as the potential for advective and/or diffusive flux of PCBs from those sediments. The two controlling factors to assess PCB flux are (1) site-specific partitioning constants and (2) the rate of exchange from underlying contaminated sediments into the overlying surface sediments and to the Willamette River.

Site-specific partitioning and rate of flux of PCBs are needed to adequately frame the models that will inform both the Recontamination Assessment and the Implementability Study. To define the data needed, it is first necessary to identify the models that may be used to evaluate PCB flux including : (1) site-specific sediment to porewater partitioning constants and (2) the rate of sediment porewater and surface water movement (flux) between sediment and overlying surface water.

2.1.1 Remedial Design Models

The following models have been identified for use in the Recontamination Assessment and Implementability Study: a two-carbon partitioning model to estimate the concentrations of dissolved PCBs in porewater, and cap/amendment addition models. Each is described below. While selection of specific models will be made as part of the Recontamination Assessment and Implementability Study, the purpose of this SAP is to gather the data to support these models.

1. **Two-Carbon Partitioning Model for Determining Dissolved PCB Interstitial Water Concentrations.** An equilibrium partitioning model (EqP) was used in the Draft Final RI Report to estimate flux from sediments into the overlying river water. These estimates were based on theoretical calculations of dissolved PCBs in porewater using the bulk sediment PCB concentration and the levels of total organic carbon (TOC).

   A refinement of the EqP model, developed by EPA, is the two-carbon model (EPA, 2012a; Gschwend et al., 2011, 2013). The two-carbon partitioning model requires measurement of PCBs in bulk sediments, PCBs in porewater, TOC, and black carbon levels[2]. Collecting empirical data for these parameters through the porewater study will allow calibration of a site-specific partitioning model. The site-specific model will allow for a more reliable estimation of dissolved PCBs in surface and subsurface porewater across the RM11E Project Area. The output from the two-carbon partitioning model will be used in the Recontamination Assessment and Implementability Study to evaluate the potential recontamination of surface sediment, monitored natural recovery, design thickness and efficacy of a conventional cap, levels of in situ treatment (such as activated carbon to sequester PCBs,) and the relative risks of remaining PCBs in post-dredging residuals (Greenberg et al., 2014).

2. **Cap/Amendment Addition Models.** The model to evaluate conventional caps, in situ remedy design, and efficacy for the Recontamination Assessment and Implementability Study will be either the RECOVERY model developed by the U.S. Army Corps of Engineers (Boyer et al., 1994; Ruiz and Gerald, 2001), or the model CAPSIM from Texas Tech University (Lampert et al., 2012; Reible and Lampert, 2014). The RECOVERY model is a relatively simple compartment model that principally models advective flux by compression during cap placement and passive flux by diffusion thereafter. CAPSIM is a more input-data-intensive model that typically is used for final design. However, it allows for the evaluation of multiple sediment layers, active flux of contaminants from groundwater intrusion, and a more explicit consideration of an active carbon addition. Model selection and justification will be presented in the Recontamination Assessment Report.

2.1.2 Inputs to the Model

Table 2-1 presents the required model parameters and their data sources for the two-carbon partitioning model and for the two cap/amendment models under consideration. A considerable amount of data was generated in connection with the Draft Final RI Report and the subsequent RM11E studies (including those being conducted under the Work Plan) to develop parameters for at least the cap/amendment models. Data that will be collected as part of this porewater study will be considered for use in calibrating the two-carbon partitioning model.

Sediment PCB congener data, collected as part of this SAP, will allow comparison to PCB porewater congener data and also will be used to evaluate correlations between congeners and Aroclors measured in the RM11E Project Area. Likewise, data on the proportion of black

[2] EPA (2012a) defines black carbon as "A form of carbon produced by the burning of biomass and fossil fuels that can accumulate in sediments. This form of carbon has a large affinity for hydrophobic contaminants of concern (COC) and can substantially reduce bioaccessibility and bioavailability."

carbon, as a proportion of the TOC values previously reported, are lacking and will be collected in this porewater study.

For the cap/amendment models, parameter data needs for sediment, surface water, and general system properties (e.g., surface area being modeled, water flow through the site) can be met with the existing information (Table 2-1). Groundwater data are being collected under the Upland Groundwater and Bank Soil SAP (Appendix B to the Work Plan) and will be evaluated in the Recontamination Assessment and Implementability Study. Data not currently available include PCB partitioning and PCB porewater concentrations, both of which will be addressed through this porewater study.

Maximum and minimum groundwater seepage (flux) rate(s) will be estimated based on a standard approach that uses the hydraulic gradients (i) between upland wells and the Willamette River (taking seasonal maximum and minimum gradients and diurnal tidal variation into consideration) and an estimated range of hydraulic conductivity values (k) from geologic samples from the shoreline wells and sediments collected in the area. A version of this method (Lampert and Reible steady-state model, 2009) was used to evaluate capping in the Portland Harbor FS. Seepage velocity will be calculated by multiplying gradient and hydraulic conductivity. Mass flux will be estimated by multiplying seepage velocity by porewater concentration.

Based on this evaluation, the data needs identified for this porewater study are:

- Concentrations of dissolved PCB congeners in porewater
- Concentrations of PCBs in bedded sediments as both Aroclors and congeners3
- Concentrations of TOC and black carbon in bedded sediment
- Concentrations of dissolved PCB congeners in surface water immediately above the sediment porewater sampling locations
- Concentrations of dissolved PCB congeners in surface water immediately upstream of the porewater sampling locations

While not a specific data need per se, estimated concentrations of dissolved PCBs based on the EqP model used for the Draft Final RI Report will be compared to the measured PCB concentrations in porewater based on the sum of all congeners, and then compared to the estimates made using the calibrated two-carbon partitioning model. If advective flux is observed, existing subsurface sediment data will be used to calculate subsurface porewater concentrations and as model inputs for subsurface sediment concentrations.

The associated field and analytical activities to address these data needs are detailed in Section 4.

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[3] Bulk sediment samples will be collected as close as practicable to the porewater sampler. In discussions with MIT it was decided that collecting a core at the time of passive sampler deployment could impact the sediment column close to the passive sampler (leaving a hole in the bedded sediment), and thus could compromise the porewater sampler exposure evaluation. This study will collect that sample at the time of passive sampler retrieval.

2.2 Sampling Methods

The RM11E porewater sampling will employ passive samplers developed at the Massachusetts Institute of Technology (MIT). Passive samplers are capable of sorbing hydrophobic organic compounds and have been demonstrated to be effective in quantifying PCBs in porewater at a number of EPA sites (EPA, 2012b; Greenberg et al., 2014)), including recent work on the Lower Duwamish Waterway (Gschwend et al., 2013; HDR et al., 2013). Cleaned, inert sheets of polyethylene (PE) are placed into rigid frames and a passive sampling device is pushed into the sediment. In nature, organic carbon is the dominant sorption phase of hydrophobic organic compounds such as PCBs (EPA, 2012). While there are many mass transfer processes that control desorption of PCBs into porewater, and then sorption into a passive sampler, a large body of research has demonstrated that plastic samplers such as PE act essentially as another carbon sink, and that the PCBs will come into equilibrium between the three phases: sediment carbon, dissolved in porewater, and in the passive sampler (Mayer et al., 2014).

If left sufficiently long in the sediment, until steady-state conditions exist, the PE will equilibrate with the PCBs in porewater. However, for highly chlorinated PCBs, this can take months or up to a year or longer (Tomaszewski and Luthy, 2008; Gschwend et al., 2012a; Ghosh et al., 2014). To facilitate the use of a shorter PE exposure period, while still being able to calculate the PCB concentration at equilibrium, performance reference compounds (PRC) will be impregnated into the PE. These PRCs are essentially internal standards whose loss from the PE is used to correct for the samplers' incomplete equilibration with the sediments (Fernandez et al., 2009). For this porewater study, these PRCs will be stable isotope-labeled or deuterated forms of the congeners with similar partitioning coefficients (log $K_{ow)}$ as the target congeners.

The data collection is intended to reflect remedial design conditions. Chemical isolation design requires consideration of advective loading from subsurface sediment to surface sediment and from surface sediment to surface water, both of which are driven in part by the groundwater flux rate through the sediment. A controlling remedial design condition for groundwater flux is the period when the river is at its lowest stage, such that the hydraulic head in groundwater between the upland and the river is greatest. The low river stage for the Willamette River normally occurs during August through November each year, and is, if practicable, the preferred time to collect porewater data (Figure 2-1).

2.3 Porewater Sampling Program

2.3.1 Defining the Sampling Area

The sampling area is a subset of the overall RM11E Project Area and was established with consideration of the following (Figure 2-2):

- Comprehensive Benthic Risk Area identified in Draft FS Report
- Alternative F Remedial Action Level footprint in the Draft FS Report
- Engineered Cap and In-Situ Treatment Areas identified in Draft FS Report
- Availability of paired Surface-Subsurface Previous Sampling Locations
- Surface and subsurface concentrations of PCBs and TOC

Porewater inert-samples will be collected from an area with a wide range of PCB concentrations in surface and subsurface sediment. Therefore, the results from this study will allow for the assessment of porewater as a potential source of recontamination in other locations within the RM11E Project Area.

2.3.1.1 Comprehensive Benthic Risk Area

The Draft FS Report identifies a near shore area of benthic concern between approximately RM 11.25 and RM 11.50. For RM11E, this is, in part defined by bioassays, but also by the EPA guidelines for assessing benthic risk for contaminants of concern (COC). For RM11E, PCBs represent the principal COC that defines this Comprehensive Benthic Risk Area (shown as a hatched area in Figure 2-2). The porewater sampling area generally is contained within the Comprehensive Benthic Risk Area for the RM11E Project Area because it represents a known area of benthic concern.

2.3.1.2 Alternative F Remedial Action Level Footprint and Engineered Cap and In Situ Treatment Areas

The Work Plan states that Alternative F from the Draft FS Report will be used as a conservative footprint for the alternative analysis. Alternative F is defined by the 75 micrograms per kilogram (µg/kg) PCB Remedial Action Level (RAL), and the Draft FS Report identified engineered capping and in situ treatment as integrated remedial alternatives for RM11E in areas located primarily beneath and behind the Glacier NW and Cargill docks (Figure 2-2). For the purpose of the RM11E study, in situ treatment will be considered to be part of an engineered capping remedy as it involves introducing sorbent amendments such as activated carbon (AC) into contaminated sediments. Porewater data collected over a range of physical and chemical conditions are intended to inform design and potential recontamination of both engineered caps and in situ treatment areas. Consequently, the porewater sampling area includes the engineered cap and in situ treatment areas shown in Figure 2-2 and is congruent with the Comprehensive Benthic Risk Area.

2.3.1.3 Paired Surface-Subsurface Previous Sampling Locations with Elevated PCBs

To understand the nature of potential flux of PCBs from subsurface contaminated sediments, it is beneficial to place porewater sampling stations at existing sediment sampling locations with both surface and subsurface measurements of PCBs in sediment. Stations with elevated PCB surface sediment concentrations are presented in Figure 2-3, and those with elevated subsurface sediment concentrations in Figure 2-4.

Several paired surface and subsurface sediment PCB measurements are located within the Comprehensive Benthic Risk Area, in the 75 µg/kg RAL, and in the vicinity of the Draft FS Report-identified integrated engineered cap and in situ treatment footprints. Table 2-2 presents sampled locations within the 75 µg/kg RAL footprint, including the physical (grain size, total solids, TOC) characteristics and total PCB concentrations. To generate a wide range of measured porewater data for calibrating the partitioning and cap models, these existing sediment data were used in Section 2.3.2 to select porewater sampling stations located across a

range of PCB sediment concentrations, TOC concentrations, and estimated porewater concentrations[4].

2.3.2 Porewater Sampling Stations

Figure 2-1 shows the proposed porewater sampling stations. Generally, the stations are numbered from downstream to upstream (left to right in Figure 2-1) and from onshore to offshore. The factors considered for their selection are presented in Table 2-3 and are discussed on the basis of the grab (G) and core (C) pairings. Three stations are arrayed in a transect along the Draft FS Report-identified integrated engineered cap/in situ treatment area (RM11E-PW002, RM11E-PW003, and RM11E-PW006), and in two transects extending offshore: RM11E-PW003, RM11E-PW004, RM11E-PW005 and RM11E-PW002, RM11E-PW001. With the exception of RM11E-PW001, all of these data were collected in the 2009 sediment sampling event (Table 2-3). The data for RM11E-PW001 were collected in 2007, with a re-collection of the surface sediment sample in 2013. The bulk sediment data are of sufficient recency and quality to be used in this study.

Sampling stations are presented in Table 2-3 and described below.

2.3.2.1 RM11E-PW001

This station is located at the paired surface/subsurface sediment sampling locations RM11E-G079 and LWG-UG02/UC02. RM11E-PW001 is within the Comprehensive Benthic Risk Area, but is an area targeted for removal in the Draft FS Report. The station is represented by two surface grab samples at approximately the same location, but at different concentrations; 5,900 µg/kg at UG02 from the Draft FS Report, and 2,200 µg/kg at G079 from the recent Supplemental Sediment Sampling. The TOCs are low (<0.5 percent) at both intervals, and the corresponding PCB porewater concentrations ($C_{pw\text{-}s}$) are the highest estimated within the RM11E Project Area. Subsurface PCB concentrations are non-detect. This station provides a high total PCB value (> 2,000 µg/kg) with a low TOC, which will provide useful data for calibration of the two-carbon partitioning model. Furthermore, it would be expected that any flux of dissolved PCBs into the overlying water column would be solely from the surface sediment.

2.3.2.2 RM11E-PW002

This station is located at the paired surface/subsurface sediment sampling locations RM11E-G022/C019. RM11E-PW002 is within the Comprehensive Benthic Risk Area and is located in an area identified for possible in situ treatment in the Draft FS Report. While not indicated in the Draft FS, capping may also be possible in this area. PCB concentrations in the upper 1.5 meters are relatively low (178 to 340 µg/kg), and higher at 1.5 to 2.0 meters below mudline (bml) (6,200 µg/kg). TOC averages approximately 2 percent through the sediment column, and in the

[4] Porewater concentrations were estimated in Table 2-3 (EPA, 2012a) based on total PCB concentrations, TOC, and the same octanol-water coefficient used in the Bioaccumulation Modeling Report (K_{ow} = 6.14) (Windward, 2009). Individual PCB congeners that were not detected were not included in the total PCB porewater estimation. Consistent with EPA guidance and the Draft Final RI Report, calculating EqP porewater concentrations require a detectable PCB and > 0.2 percent TOC. Where either of those conditions did not exist, the PCB porewater concentrations were not calculated.

surface intervals the calculated C_{pw} (18.3 – 36.1 nanograms/liter [ng/L]) is near the PRG for ecological receptors (RAO7 – Table 2-4) of 14 ng/L. In the 1.5- to 2.0-meter interval, the C_{pw} is 868 ng/L, and at 5 ng/L in the lowest interval. Data from this station will be used to represent conditions where dissolved PCBs may be advecting into surface-sediment porewater from the elevated PCBs in subsurface sediment. These data may indicate whether the higher levels of TOC in the sediment column effectively sequester PCBs, which may be indicated by lower flux into the overlying surface water.

2.3.2.3 RM11E-PW003

This station is located at the paired surface/subsurface sediment sampling locations RM11E-G026/C022. RM11E-PW003 is within the Comprehensive Benthic Risk Area and is located in an area identified for potential in situ treatment in the Draft FS Report. Capping may also be possible in this location. PCB concentration levels are relatively high in the surface sediment (1,400 µg/kg), lower in the middle section of the subsurface sediment column (30.9 to 90.5 centimeters [cm] at 370 µg/kg), and elevated in the deepest segment down to 140 cm (1,500 µg/kg). TOCs range from 1.35 to 1.91 percent; the calculated C_{pw} is highest in the surface interval (217 ng/L) and lower below. Data from this station will be used to represent conditions where PCBs may be advecting into surface-sediment porewater from the elevated dissolved PCBs in subsurface sediment.

2.3.2.4 RM11E-PW004

This station is located at the paired surface/subsurface sediment sampling locations RM11E-G027/C023. RM11E-PW004 is within the Comprehensive Benthic Risk Area, and outside of the Draft FS Report-designated cap or in situ treatment area. This location was selected because of its relatively low PCB surface sediment concentration (133 µg/kg), with elevated PCB concentrations in the subsurface sediments (650 to 3,300 µg/kg) down to approximately 3 meters bml. TOC and estimated C_{pw} are low (0.46 percent and 57.2 ng/L) at the surface interval, and are higher at depth (0.86 to 1.6 percent TOC and 105 to 802 ng/L). Data from this station will be used to represent conditions where dissolved PCBs may be advecting into surface-sediment porewater from the elevated PCBs in subsurface sediment. With the low TOC level in surface sediments, flux measured in the overlying surface water may be attributable to flux from the subsurface sediments.

2.3.2.5 RM11E-PW005

This station is located at the paired surface/subsurface sediment sampling locations RM11E-G028/C024. RM11E-PW005 is outside of the Comprehensive Benthic Risk Area, but is within the 75 µg/kg total PCB RAL in the region targeted in the Draft FS Report for removal. It has a relatively moderate surface sediment PCB concentration (373 µg/kg) and low subsurface contamination levels (<4 .4 µg/kg TOC is 1.4 percent in the surface sediment, but less than 0.1 percent in the lower intervals). C_{pw} is estimated at 49 µg/kg in the surface, but cannot be estimated for the lower intervals. This station provides an intermediate total PCB value (less than 500 µg/kg) within the range of data needed for calibration of the two-carbon partitioning model. Furthermore, it would be expected that any flux of dissolved PCBs into the overlying water column would be solely from the surface sediment.

2.3.2.6 RM11E-PW006

This station is located at the paired surface/subsurface sediment sampling locations RM11E-G033 and C029. This station is within the Comprehensive Benthic Risk Area, and was identified as a possible in situ treatment site in the Draft FS Report. It is also a potential site for capping. PCB concentrations at the surface are 2,000 µg/kg and then are non-detect to a depth of at least 4 meters. The TOC is low (0.46 percent), and the estimated C_{pw} is 908 ng/L. This station provides a high total PCB value (> 1,000 µg/kg) with a low TOC, which will provide data for calibration of the two-carbon partitioning model. Furthermore, it would be expected that any flux of dissolved PCBs into the overlying water column would be solely from the surface sediment.

2.3.2.7 RM11E-PW007 – Upriver Reference Location

This station, will be located at the previous upstream surface sediment sampling location RM11E-C043. This station (sediment, porewater, and surface water) may provide insight on which PCB congeners are contributed by upstream sources. Data from this station will provide an upriver measure of dissolved porewater PCBs in a relatively uncontaminated (non-detected at 1.7 µg/kg) area, and also as a measure of the upriver concentrations of dissolved PCBs in the overlying water (Figure 2-1). The in-sediment analytical data from the upstream station should also serve a second function in defining the lower boundaries (lowest PCB congener concentration) for the two-carbon partitioning model.

2.4 Preliminary Remediation Goals as Design Inputs

Additional inputs to the porewater study design are the remedial action objectives (RAO) and preliminary remediation goals (PRG) that are being developed for Portland Harbor. Table 2-4 shows the PCB RAOs and PRGs applicable to this porewater study (as provided by EPA to the LWG on March 3, 2014). To the extent practical, bulk sediment analytical reporting limits should be below the lowest sediment PRG of 4 µg/kg, while the porewater measurements should target a total PCB reporting limit of 0.0064 ng/L. Reporting limits are discussed further in Section 5.

Section 3. Project Organization

This section summarizes the organizational structure, responsibilities, and resources employed to support this SAP, including field activities, laboratory services, data validation, data management, and reporting.

3.1 Team Organization and Responsibilities

This SAP will be implemented by a team of consultants and subcontractors that will be retained by DOF. DOF is the primary common consultant retained by the RM11E Group and will contract principally with SEE and GSI to accomplish this work

3.1.1 Project Manager (PM)

Paul Fuglevand, PE (DOF), is the senior Project Manager (PM). In this role, he will provide general oversight of the work and will be the point of contact for the RM11E Group. Paul will work closely with SEE and GSI, as discussed below, and other project staff members to ensure that the project objectives are achieved. Principal deviations from the SAP will not be made without prior approval from the PM.

The PM generally is responsible for the following:

- Oversee the planning and implementation of all field sampling efforts in accordance with this SAP.
- Coordinate with the Field Director (FD) to address any field problems and approve deviations from this SAP.
- Communicate with the RM11E Group regarding the schedule, performance, and any anticipated deviations from sampling and analysis activities.

3.1.2 Field Director (FD)

Tim Thompson (SEE) is the Technical Project Lead (TPL) for the porewater investigations. He will serve as the lead scientist and FD for all sampling activities. He will report directly to the PM and coordinate with other project staff members. The FD generally is responsible for the following:

- Direct the planning and implementation of all field sampling efforts, including arranging for necessary sampling equipment and overseeing the operations of vessel subcontractors (described below).
- Mobilize for field work and direct all aspects of the sampling to ensure that the appropriate procedures and methods are used in accordance with this SAP.
- Coordinate closely with the PM, Sampling and Analysis Coordinator (SAC), and field staff members to address any field problems, deviations from this SAP, or emergencies that may arise.

- Function as the Field Safety Officer and ensure that the sampling activities adhere to the Health and Safety Plan (HSP).
- Track the schedule and performance of the sampling and analysis activities according to this SAP in direct coordination with the PM.

The FD will work closely with the PM and SAC to fulfill the listed responsibilities and may be assisted at times by other project staff members.

3.1.3 Sampling and Analysis Coordinator (SAC)

Erin Carroll Hughes (GSI) will be the SAC and will work closely with the PM and FD. The SAC generally is responsible for the following:

- Coordinate with the owners of waterfront properties with active working docks, marine operations, and vessel traffic to ensure that, to the extent possible, the consultant team's access to these properties will not interfere with the normal activities conducted at these properties, and will accommodate periodic operational and security limitations resulting from these operational activities.
- Receive and maintain copies of field documentation and laboratory chain-of-custody forms.
- Assist the FD in tracking the schedule and performance of the sampling and analysis activities according to this SAP.
- Assist with sampling efforts.
- Assist with safety operations.
- Assist with investigation-derived waste (IDW) management and ensure that it is removed in a timely manner from properties owned and/or operated by RM11E Group members.

The SAC may be assisted at times by the FD, PM, and other project staff members.

3.1.4 Field Support

Subcontractors anticipated to be used to support this work are listed below by work type:

- **Ballard Marine Construction (BMC).** Commercial divers from BMC will be used to: (1) conduct an initial pre-deployment field reconnaissance of the proposed porewater sampling locations; (2) place the porewater samplers at predetermined locations; and (3) after approximately 60 days, retrieve the porewater samplers and take surface sediment cores at the same locations where the samplers were retrieved.
- **Willamette Cultural Resources Associates, LTD. (Willamette CRA)**. David Ellis of Willamette CRA and his team will provide cultural resource monitoring services, as necessary, during sample processing (see Section 4.5).

3.1.5 Data Validation and Management Support

The data validation and data management subcontractors are listed below by work type:

- **Pyron Environmental, LLC.** Mingta Lin of Pyron Environmental will serve as Chemical Quality Assurance (QA) Manager responsible for laboratory coordination and oversight, and will conduct the quality review of analytical data. The Chemical QA Manager will be the final arbiter of any data qualifiers that may be needed to the laboratory-reported result. A data validation report will be written for the project, and will be included in the appendices of the final report. Mr. Lin previously worked with SEE, ALS Environmental (ALS), and MIT on the Lower Duwamish porewater sampling program, and is familiar with the methods, QA and quality control (QC) requirements, and the data validation procedures for this PE sampling.
- **Data Management (GSI).** GSI will assign a Data Manager (DM) who will maintain the project database, and will coordinate directly with the PM, FD, SAC, Chemical QA Manager, and primary contract laboratory, as needed. Validated laboratory results will be provided as electronic deliverables to the DM by the Chemistry QA Manager. The DM will coordinate with the Chemistry QA Manager to determine the appropriate database structure, verify the satisfactory electronic transfer of validated data, maintain the integrity of the database, and oversee all data queries and reporting.

3.1.6 Senior Chemist

Dr. Philip Gschwend, along with his staff at MIT, will participate in the project as the Senior Chemist for conducting the porewater sampling at RM11E. He will serve as an advisor for the preparation of the passive samplers, on how to impregnate the samplers with PRCs, and aid in the interpretation of the results. The methods being used for this project were developed by his laboratory at MIT. He will coordinate with the primary contract laboratory, to prepare the passive samplers, impregnate the samplers with PRCs, and aid in the interpretation of the results. In addition, a subset of collected field sediment samples and passive samplers will be sent to MIT as part of the project QC program as a check on the reliability and accuracy of the analytical method. The accuracy of porewater measures in assessing PCBs is expected to be similar to those reported by Gschwend et al. (2011).

3.1.7 Laboratory Services

ALS, of Kelso, Washington, is the primary contract laboratory and will perform chemical analyses on the passive samplers and the sediment samples collected. Jeff Christian will serve as the Laboratory Project Manager to oversee laboratory performance in accordance with the QAPP Addendum (Appendix A of the Work Plan). He has served as ALS's (formerly Columbia Analytical Services) project manager for a number of sediment characterizations conducted by DOF and SEE, and is familiar with the analytical objectives of this project. ALS is a certified by the National Environmental Laboratory Accreditation Program; method certifications for the laboratory are included in Appendix A of the Work Plan.

3.2 Regulatory Oversight

Regulatory oversight will be provided by EPA. Sean Sheldrake is the site Remedial Project Manager (RPM) for EPA and he may be supported at times by other technical staff members and consultants. As the lead agency for all activities related to site assessment for the Portland Harbor RI/FS, EPA will oversee implementation of the activities associated with the RM11E Settlement Agreement and SOW and coordinate feedback on deliverables from other agencies

and the tribes that are overseeing the work to be performed by the RM11E Group. As noted in Section 4.5 of this SAP, a Field Change Request form will be submitted to Sean Sheldrake via email if sample locations need to be moved more than 10 meters from the initial target. Contact information for these requests is:

Sean Sheldrake, Unit Diving Officer, RPM
EPA Region 10, 1200 Sixth Ave., Suite 900
Seattle, WA 98101
sheldrake.sean@epa.gov

3.3 Health and Safety

The primary hazards for the sampling event are physical hazards associated with the river environment and working on a vessel with heavy and mobile equipment in and around working docks and marine operations with frequent vessel traffic. Diving for sediment sample collection also requires careful adherence to safety procedures and a diving-specific HSP prepared by the diving contractor. The field crew will exercise sound field judgment and practices to maintain a safe working environment during sample collection and all other field activities. The field crew will comply with HAZWOPER regulations under 29 CFR 1910.120, exercise due care to maintain the integrity of in-water structures, avoid interfering with marine operations and other activities conducted at and around the working docks, and comply with all operational and security limitations, as directed by the FD and the vessel operator.

GSI prepared a project-specific HSP (Appendix D of the Work Plan) for sediment grab sampling. BMC prepared a diver HSP for under-dock sediment sampling. Both HSPs incorporated EPA review comments. Those approved plans will be used as appropriate in the development of supplemental HSPs for the Porewater SAP activities for the field staff and divers. The HSPs for the Porewater SAP will be submitted to EPA at least 6 weeks before the planned mobilization for porewater sampling.

As noted above, the FD will function as the Field Safety Officer during the field work and will coordinate with the diver Field Safety Officer to determine the limits of safe practice and operating conditions during field activities. The FD will confirm that field personnel have up-to-date 8-hour HAZWOPER refresher safety training and medical monitoring, if required. The FD will lead the safety meeting at the beginning of the field work and daily safety briefings each morning before beginning field activities. The FD also will provide a safety briefing to any new participant involved in the field activities.

Section 4. Field Activities to Support Porewater Sampling

This section describes the activities to support porewater sampler placement and retrieval, as well as the collection of surface sediment samples (30 cm bml) from the porewater sampling locations upon retrieval. As noted previously, the field methods and analyses are similar to those employed on the Lower Duwamish Waterway in Seattle, Washington (Gschwend et al., 2013; HDR et al., 2013). An overview of the field placement of passive samplers in the Lower Duwamish Waterway in 2012 is posted on the EPA Region 10 Dive Unit Web site.[5]

4.1 Overview of the Porewater Sampling Approach

For the RM11E porewater sampling, the passive samplers will be impregnated with the PRCs at ALS's laboratory at Kelso, Washington, and mounted into the sampling frame just before deployment. Each frame will have 50 x 10 cm strips of PRC-labelled PE. At each sampling location, two passive sampling devices (PSD) will be deployed, approximately 8 inches apart. Divers will install the samplers at the selected sampling locations, to a target depth of 30 cm bml, leaving 20 cm of the PE extending above the mudline in the overlying surface water. The sampler will be left to interrogate the sediment and overlying water for approximately 60 days.

At the time of retrieval, prior to removal of the sampler, a 40- x 10-cm-diameter core will be inserted between the two PSDs at each station, and then the PSDs will be removed. Once the PSDs are removed and secured on the support vessel, the core will be extracted. The PE frame itself will be labeled, wrapped in foil, and stored on ice for transport. Likewise, the collected cores will be capped and labeled in the field; both the PE and collected sediment will be transported in separate ice-filled coolers to ALS's laboratory at Kelso, Washington, for further processing.

At ALS, the PE will be removed from each frame and cut initially into two sections: the sediment (lower 30 cm) section and the surface water (upper 20 cm) section. These sections will be further cut into 5-cm-wide longitudinal strips thereby creating and making available replicate sediment and surface water samples representing the full depth profile available for analysis or archiving at each station. The individual strips will be wrapped in aluminum foil, labeled, and placed into labeled jars at ALS. For each sampling location, the PE will be extracted and analyzed for the 209 PCB congeners, as well as the 5 PRC congeners. The PE from at least two locations will be sent to MIT for confirmation sampling analysis.

The collected sediment cores provided to ALS will be extruded, logged, photographed, and composited for analysis of total solids, grain size, PCB Aroclors, PCB congeners, TOC, and black carbon (Table 4-1). Only the top 30 cm of sediment in the core tube will be collected for analysis.

[5] http://www.epa.gov/region10/pdf/diveteam/duwamish_passive_sampling_2013.pdf

Methods for laboratory analyses are discussed in Section 5. The remainder of this section covers field activities.

4.2 Sample Location and Sample Designations

Sampling locations are presented in Table 2-3 and Figure 2-2. Sampling locations include six onsite RM11E locations, and one upriver location, with one field replicate for a total of eight sets of analyses at ALS. The number and type of chemical analyses are presented in Table 4-1. The location and identification of each sample are presented in Table 4-2. Location identifications for these stations are noted as RM11E-PW for "porewater" and are numbered 001 – 007 (e.g., RM11E-PW007). The field replicate currently is targeted for collection at station RM11E-PW004 and as such would be designated as PW504. The field replicate station may change based on sediment characteristics and/or physical obstructions encountered during sampling. Sample identification number (ID) are further delineated as "PWP" for porewater sample, "PWW" for the overlying surface water passive sample, and "PWG" for the grab sediment sample. For the MIT confirmation samples, "b" is added to the sample designation. For example, RM11E-PWW005b is the surface water passive sample from station 5.

The specific sampling and analyses scheme is provided in Table 4-3.

4.3 Navigation and Sample Locating

This Porewater SAP incorporates by reference the Sediment Sampling SAP Addendum (Appendix C of the Work Plan) Section 3.2 on Navigation Sampling and Station Locating. Appendix C of the RM11E Work Plan describes the navigation equipment and procedures, which are consistent with those previously employed for previous sediment sampling. This includes both horizontal and vertical measures of station position. In addition, this Porewater SAP includes a Standard Operating Procedure (SOP) for Diver-Placement and Retrieval of Passive Samplers and Co-located Sediment Samples (Attachment 3). The FD will coordinate safety, navigation, positioning, and communication with the divers during placement and retrieval. A copy of the SOPs, which contain more detailed information on sample location and positioning, will be on board the sampling vessel throughout deployment, retrieval, and sediment sampling activities.

4.4 Field Porewater Reconnaissance Survey

A pre-deployment field reconnaissance and initial trial placement of the passive samplers was conducted on May 2, 2014. The intent of the reconnaissance survey was to determine whether the gravel, sand, and anthropogenic materials found during the 2009 grab sampling and coring would either prevent effective sampler insertion or, alternatively, damage the PE during placement and retrieval.

Prior to undertaking the reconnaissance survey, modifications to the sampler and procedures described in the March 3, 2014, draft SAP and SOP were made. Modifications were based upon EPA comments dated April 3, 2014, and the RM11E Group response to comments dated April 17, 2014, which was discussed with EPA in a conference call on April 22, 2014. EPA acknowledged the RM11E Group Response to EPA Comments on April 24, 2014. These changes included modifying the sampler size, adding of a detachable diver-operated hammer assembly

to assist installation of the PE sampler into the substrate, and adding a removable stainless-steel cover over the PE to protect the sampler during insertion. Those modifications were incorporated into the Passive Sampling SOP (Attachment 3) and were implemented during the reconnaissance survey.

The results of the reconnaissance survey are presented in Attachment 4. Placement and retrieval of the passive samplers were tested at the three inshore stations where anthropogenic debris was observed during the 2009 sampling: RM11E-PW002, RM11E-PW003, and RM11E-PW006. Successful insertion and retrieval was achieved at all three locations, with no tears or other visible stress in the PE sheets.

The results of the reconnaissance survey indicate that these methods will be suitable for successful deployment and retrieval of the passive samplers in the RM11E Project Area.

4.5 Field Deployment and Retrieval Methods

Field deployment and retrieval methods are documented in the Passive Sampling SOP (Attachment 3). Those methods were developed following the SOPS developed by MIT for passive sampler deployment (Gschwend et al., 2012b). The detailed procedures for PE sampler deployment, retrieval, and collection of the co-located sediment sample at retrieval are covered in Sections 6 through 9 of the Passive Sampling SOP (Attachment 3) and briefly summarized below. The relevant portions of the Passive Sampling SOP (Attachment 3) will be reviewed at the start of each day's dive operations with the field and dive team.

Health and safety procedures for diving onsite were developed by BMC for the reconnaissance survey, and followed during field deployment. Passive samplers will be pre-loaded with the PCB PRCs at ALS following the ALS SOP for Preparing Polyethylene (PE) Passive Samplers and PCB Congener Performance Reference Compounds Loading (Attachment 5). The FD will load the PE into the passive sampling device at ALS, wrap each sampler individually in aluminum foil, and bring the passive samplers to the site in an ice-filled cooler

Two passive samplers will be deployed and separately anchored at each station. This replication is in part to ensure against the potential loss of a sampler, and to ensure adequate sample for replication and archive. The potential for sampler loss necessitates a different deployment and anchoring approach for the relatively protected inshore stations, versus those stations offshore of the docks within the river. These differences are noted below, and in detail in the Passive Sampling SOP (Attachment 3).

- For the three inshore stations (RM11E-PW002, RM11E-PW003, and RM11E-PW006), the passive samplers will be tethered to either dock pilings or other suitable structures. Two tether lines per sampler will be deployed.
- For the three locations that are offshore on the riverward side of the Glacier dock (RM11E-PW001, RM11E-PW004, and RM11E-PW005, two anchor lines per sampler will be deployed and attached to sediment anchors.
- For each offshore station, pingers (Benthos ALP365 Pinger) will be attached to the samplers to assist in location and retrieval at the end of the exposure period.

- Upstream location RM11E-PW007 is exposed to river currents, but it is anticipated there will be a tether point (potentially onshore) for the anchor line. If no tether point is available, the samplers will be anchored to the sediment and equipped with a pinger for future location.
- Buoys will not be attached to the lines to minimize any possibility of public interference with the samplers.

At each location, the passive sampler(s) will be taken out of the ice-filled cooler, fitted into the insertion assembly, fitted with stainless steel covers, and attached via a carabiner to a weighted tag line. The tag line serves two purposes: (1) to provide an additional anchor to secure the sampler frames during deployment and (2) to help locate the sampler again at the end of the deployment period. The sampling area is an active shipping area with vessels docking regularly at both the Glacier NW and Cargill docks. The offshore tag lines will need to be anchored using a shore anchor, or similarly constructed anchor, driven into the sediment.

The divers will be equipped with underwater video and surface communications. To the extent practicable, the sample location will be established by the differential global positioning system (dGPS) on board the vessel. An anchored tag line will be dropped to the river floor at the established station coordinates, and the diver will follow that line down to the sediments to drive the samplers. Once on bottom, the diver will operate to minimize disturbance of the surface sediment. The FD will view the general sample location on video, look for areas of soft sediment and minimal debris, and coordinate the selection of the actual sampling location with the diver. If the diver is required to move the location, the line anchor will be moved to the actual sampling location, and a new coordinate fix will be taken.

Sample locations will be maintained within 10 meters (approximately 30 feet) of the proposed locations in Table 4-2 and target areas with comparable sediment characteristics to satisfy the rationale and objectives for the sample. If sample locations must be moved more than 10 meters from the initial target, a RM11E Group representative will call and/or e-mail the EPA Remedial Program Manager (RPM) to discuss proposed field changes. If the RPM cannot be reached, the RM11E Group representative will call and/or e-mail the Project Manager for EPA's oversight contractor, CDM, to discuss proposed field changes. A Field Change Request form will be submitted via e-mail as a followup to any sampling location changes (see Attachment 1 for an example). Decisions to relocate samples and new sampling coordinates will be recorded in the field logbook.

After a sampling location is confirmed, the diver will manually insert the passive sampler 30 cm into the sediment and then remove the stainless steel PE covers. The passive sampler insertion assembly has a built-in stop so that the sampler cannot penetrate deeper than 30 cm. Should the diver be unable to successfully insert the sampler, it will be withdrawn and inspected. If the stainless steel PE protective plates are still in place, the diver may proceed with a second insertion attempt. If the protective plates are not in place the diver will inspect the PE for any rips or tears. If the diver notes any rips or tears in the PE membrane before or during insertion, sample deployment must be aborted and a new sampler obtained in order to proceed.

The on-board field staff will record the time of sampling, dGPS coordinates, diver observations during placement, mudline elevation, and depth of sampler penetration into the sediment. Upon successful insertion of the first passive sampler, a second sampler will be lowered to the

diver. The second sampler will be inserted approximately 8 inches apart (two diver-gloved fist-widths), parallel to the first sampler. After insertion of the samplers, the diver will secure the tag lines to anchors, or tether the lines to fixed piling or other suitable in-water structure.

After approximately 60 days, the dive/field team will return to the sampling locations to retrieve the passive samplers, as further described in the Passive Sampling SOP (Attachment 3). For the inshore stations (RM11E-PW002, RM11E-PW003, and RM11E-PW006), and the upriver station (PW007), the tag line previously secured to a piling or other tie-point will be located, and the diver will follow the line to the samplers. For the offshore stations (RM11E-PW001, RM11E-PW004, and RM11E-PW005), the passive samplers will be located using a hand-held pinger locator (Benthos DPL-275).

Before pulling out the passive sampler, the diver will measure the length of the PE exposed to the water (i.e., the portion of the PE above the sediment surface), and report it to the FD who will record that in the field notebook The diver will then push the sediment core sampler. Care will be taken by the diver to ensure:

- That the core is driven between the two PE samplers approximately 2 inches (2 diver-gloved fingers) from each sampler
- That the core drive is strictly vertical in order to not contact and potentially damage the PE sheets
- That the integrity of the sediment stratigraphy for core logging at the laboratory is preserved

The diver will communicate to the on-board field staff the approximate length of core drive, which will be recorded in the field notebook. After driving the sediment core sampler, it will remain in place until the diver removes and secures the passive samplers and then notifies the support team that the passive sampler may be lifted to the surface via the tag line. Once those are secure on the boat, the diver may withdraw the sediment sampler, placing a cap over the bottom and top of the core tube immediately after removal from the sediment. The sediment sampler is then secured to a tag line, and raised to the boat.

Onboard, the passive samplers will be rinsed immediately with deionized water and rubbed with a clean Kimwipe™ to remove any adhering sediment. The general condition of the passive sampler will be noted in the field log (e.g., intact, tears in the PE, biofouling, presence of oil), the sample's ID will be recorded onto the sampler sleeve, and then the entire sampler will be wrapped in aluminum foil. The station ID will be recorded again with an indelible marker on the aluminum foil, along with the date and time. The passive sampler then will be placed into the ice-filled cooler for transport to ALS in Kelso, Washington, for processing and analysis.

Sediment core samples collected also will have the station ID recorded onto the sample tube; this can be scratched directly onto the tube. The penetration depth will be again confirmed with the diver, and the amount of material retained in the core tube will be measured and recorded in the field log. Minimum acceptable recovery requirements will follow the SOP Section 8-10. The core lids will then be secured with duct tape, and the sample ID, date, and time will be written using an indelible marker onto the taped lids with an arrow indicating the direction to the surface of the sediment. "TOP" will be written onto the taped lid indicating the sediment

surface. These secured cores then will be placed upright into the ice-filled cooler for transport and processing and analysis at ALS.

The samples must remain in the custody of the FD or SAC at all times during the transport to and processing at ALS.

4.6 Post-Processing PE Samplers

All post-processing will be done by the FD and SAC at ALS' Kelso, Washington, facility. At ALS, the sampler first will be photographed, and then the PE will be removed from the frame. The PE surface will then be wiped and rinsed free of surface particles and coatings. This may include a brief (<1 minute) wiping with a hexane-soaked Kimwipe® (or equivalent) to remove oily or tarry exterior staining. If still wet, the PE surface will be blotted dry with a clean wipe. The PE is then cut with a clean razor blade initially into two sections as shown in Table 4-3: the sediment section (lower 30 cm) and the surface water (upper 20 cm) section. These segments will be further cut longitudinally into widths of 5 cm by 20 or 30 cm long for the water exposure and sediment exposures, respectively.[6] To immediately analyze the samples, the cut PE will be placed into labeled jars. Samples to be archived will be re-wrapped into clean aluminum foil envelopes, labeled, rolled, and then inserted into a 1-liter clean borosilicate jar. A separate label will be affixed to the outside of the jar. The sampling labels will note the sample ID, date of sampling, personnel processing the sample, and analyses to be run or archived. A small quantity of laboratory-grade water will be placed into the passive sample jar to prevent dehydration. The amount of laboratory-grade water added to the sample jar will be recorded.

4.7 Sediment Core Sample Processing

Processing of the diver-collected cores also will occur at ALS in Kelso, Washington, by the FD and SAC. Before processing, each core tube will be measured to establish the acquisition (recovery) as measured by the amount of material retained in the tube. The diver-noted penetration, along with the acquisition measure, will be recorded as percent recovery on the core log.

It is anticipated that these short cores will be able to be extruded onto a clean sheet of aluminum foil for logging and compositing. The time, date, personnel present, sediment type, stratigraphic features, and presence or absence of any visible contamination will be recorded in the core log for each core. In addition, photographs of each core will be taken. Each core photograph will include a label denoting station, time and date, and a scale (tape measure) showing depth bml. Each core will be examined by a senior sediment scientist who is familiar with site conditions, and those observations will be recorded in a sediment coring log.

Only the top 30 cm of the cored material will be placed into a decontaminated stainless-steel bowl for homogenizing. Rock larger than coarse gravel (16 millimeters), large organisms, and

[6] If the mudline at the time of retrieval as measured by the diver is higher or lower than when the sampler was installed, the analyzed sections will need to be adjusted in the laboratory. If sediment has accreted, the section that is above the original 30 cm sediment exposure will be cut out separately and archived, but not analyzed. Where erosion has occurred, the new section exposed to the water will be excised and archived, and the sediment-exposed PE segment adjusted accordingly.

pieces of debris will be removed and noted in the field logbook. The sediments will be mixed to visible uniformity[7], and subsamples from this homogenate then will be transferred into the appropriate containers for individual analyses. The samples required and the jar size are presented in Table 4-4.

4.8 Sample Handling, Custody, and Transport

This study will follow the handling, chain-of-custody, and transport requirements defined in the RM11E Focused Sediment Characterization SAP (GSI, 2009) and summarized in Section 3.4 of the Sediment Sampling SAP Addendum (Appendix C of the Work Plan). Transfer of both the passive samples and the sediment samples to ALS at Kelso, Washington, will occur after completion of the post-processing by the FD and SAC.

4.9 Cultural Resource Monitoring

David Ellis and Matt Goodwin of Willamette CRA have been retained to oversee potential cultural resources found in the RM11E Project Area. David has provided training to GSI field staff to define what kinds of artifacts and deposits require examination and documentation by a professional archaeologist. A Willamette CRA archaeologist was present during the initial Supplemental RI/FS sampling conducted in November 2013 to inspect surface sediment grab samples. No sensitive prehistoric or historic artifacts were found during this sampling or previous RM11E sediment sampling conducted in this area. This archaeologist will be on-call during logging and processing of diver-collected surface sediment samples at the laboratory. If archaeologically sensitive prehistoric or historic artifacts are discovered at this time, the FD will contact the archeologist and follow the procedures described in the Inadvertent Discovery Plan (Attachment 2 of this SAP). All personnel will follow Oregon State Historic Preservation Office guidelines for known sites and isolated finds (Oregon Revised Statute [ORS] 358.905-358.955) or sites along scenic waterways (ORS 390.805-390.925).

[7] Visible uniformity means a homogenous mixture of color, texture, density, and particle density that is free of any clay clasts

Section 5. Laboratory Analyses

This section: (1) describes the chemical parameters to be analyzed, (2) identifies analytical laboratories and analytical methodologies, (3) defines data quality goals, and (4) specifies analytical data reporting and validation requirements. Except as noted in this Porewater SAP, all laboratory QA and QC procedures will follow those prescribed in Section 3 of the Quality Assurance Project Plan Addendum (Attachment A) of the Work Plan. Significant deviations from these procedures will be documented in the Porewater Characterization Report.

5.1 Chemical Analyses

The analytical approach for this sampling event corresponds to the project objectives stated in Section 2. The analytical parameters include:

- Porewater
    - PCB congeners (including PCB PRCs) extracted from passive samplers
- Sediment
    - Total solids (TS)
    - Grain size
    - TOC
    - PCB congeners
    - PCB Aroclors
    - Black carbon

The analytical schedule for each sample location is summarized in Table 4-1 and detailed in Table 4-3.

5.2 Porewater Samplers

The analytical methods for the passive samplers have been developed at ALS with assistance from MIT. The analytical methods at ALS were developed generally following the MIT-developed SOPs procedures for passive sampling listed below:

- Passive PE Sampling in Support of In Situ Remediation of Contaminated Sediments: Standard Operating Procedure for polyethylene device (PED) Preparation (Gschwend et al., 2012b)
- Passive PE Sampling in Support of In Situ Remediation of Contaminated Sediments: Standard Operating Procedure for PE Analysis (Gschwend et al., 2012c)

The ALS SOP for sampler preparation and PRC loading is substantively similar to the MIT SOP, with modification noted for PRCs used and reporting limits as described below. ALS' SOP is presented in Attachment 5 of this SAP. The ALS SOP for analysis of the PE by EPA Method 1668C is presented in Attachment 6 of this SAP.

Chemical analyses run on the PE sheets include the following:

- **PRC Loading Validation**: During the preparation of the passive sampling device for field deployment and before field deployment, a single piece of the PRC-loaded PE will be extracted for ***each*** sample location set and analyzed at ALS in Houston, Texas, to validate initial PRC loading concentrations. Select PCB congeners in Table 5-1 will be used as PRCs. PRC confirmation will be done by EPA Method 1668c (see below); recovery standards employed by ALS also are listed in Table 5-1.
- **PCB Congeners in the Field-Samplers:** Passive samples will be extracted using methylene chloride and run by EPA Method 1668c at ALS in Houston, Texas. This method uses high-resolution gas chromatography/high resolution mass spectrophotometry. It is commonly referred to as high resolution GC/MS. Attachment 6 to this SAP is the ALS SOP for conducting PCB congener analyses by EPA Method 1668c. Samples will be cut into longitudinal strips as discussed in Section 4.6, and analyzed as noted in Table 4-3. The anticipated method detection limit (MDL), method reporting limit (MRL), and QC parameters for the 209 PCB congeners in PE are presented in Table 5-2. Split samples sent to MIT will be run by high-resolution gas chromatography/low-resolution mass spectrometry (HGRC/LRMS) (Gschwend et al., 2012c). This method is commonly referred to as low-resolution GC/MS.

5.3 Sediment Analyses

Specific sample preparation and analytical methods for each of the analytes are summarized in Table 5-4. These are described briefly below.

5.3.1 Conventionals

All sediment will be analyzed by ALS in Kelso, Washington, for a limited set of conventional analytes including total solids, TOC, and grain size. The analyses of these parameters will follow respective protocols specified in the following documents:

- EPA *Test Methods for Evaluating Solid Waste (SW-846).*Third Edition and Revised Update IIIA. Office of Solid Waste and Emergency Response, Washington, D.C. April 1998.
- EPA *Methods for Chemical Analysis of Water and Wastes* (*MCAWW)* EPA/600/4-79-020, Revised March 1983.
- PSEP *Recommended Protocols for Measuring Conventional Sediment Variables in Puget Sound,* Puget Sound Water Quality Authority, March 1986.

5.3.2 PCB Aroclors and Congeners

Analyses of PCB Aroclors in sediment will be performed by ALS in Kelso, Washington, while PCB congeners will be performed by ALS in Houston, Texas. The PCB homologues and congeners analyses at ALS will follow the protocols below:

- EPA *Method 8082A Polychlorinated Biphenyls (PCBs) by Gas Chromatography*, February 2007.
- EPA *Method 1668C - Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS,* April 2010.

PCB congener (EPA Method 1668C) data will be evaluated and reported down to the MDL.

Samples sent to MIT will be prepared using solvent extraction with an accelerated solvent extraction system (ASE) followed by HRGC/LRM –SIM.

5.3.3 Black Carbon

The analysis of black carbon will be performed by ALS in Phoenix, Arizona, following the protocols below:

- *Standard Operating Procedure for Sample Preparation for Black Carbon (Soot) in Sediment by Chemothermal Oxidation Pretreatment and Combustion/ Thermoconductivity or Infrared Detection.* CAS SOP 9/20/2010. (Attachment #7 to this SAP).

5.3.4 Standard Reference Material

Standard Reference Material (SRM) analyses are required for parameters at a frequency of one analysis per 20 samples or one analysis per preparation batch, whichever is more frequent. Specific SRM should be used for respective type of analysis, as specified below:

- National Institute of Standards and Technologies 1941b for TOC
- SRM-911 for PCB Aroclors

The acceptance range for SRM recovery is ±20 percent of the certified 95 percent confidence interval. Any corrective actions necessary will be taken in accordance with recovery outliers per the U.S. Department of Defense (DOD) Quality Systems Manual, Appendix DoD-B (DOD, 2010).

5.4 Laboratory QA/QC Procedures

Laboratory QA/QC procedures are defined in Appendix A of the Work Plan, which follows the use of standard EPA methods and other accepted methods and standard analytical procedures for the target analytes. Analytical methods and QC measurements and criteria will be based on current Contract Laboratory Program and SW-846 requirements, and EPA guidance. The parameters used to evaluate data quality are listed in Table 5-5. For sediments, the ALS MDLs and MRLs are given in Table 5-6, with accuracy and precision control criteria listed in Table 5-7. Finally, the data quality goals for PCB congeners in sediments are presented in Table 5-8.

Additional laboratory methods, QA procedures, and QA/QC requirements for the sampling as well as data validation procedures are included in the QAPP Addendum (Appendix A of the Work Plan).

5.5 Archived Samples

Both PE samples and sediment samples for each station will be archived according to the conditions listed in Table 4-4. Upon receipt of validated data, all archived samples will be disposed of by ALS.

Section 6. Data Management

Data management protocols for both field data and electronic data will be consistent with those used throughout the Supplemental RI/FS and the previous RM11E Focused Sediment Characterization (GSI, 2009a, 2009b, 2010a). The objective is to provide consistent, accurate, and defensible documentation of data quality, and incorporate data management protocols used for the Portland Harbor RI/FS (Integral, 2007), RM11E Focused Sediment Characterization, and Downtown Portland Sediment Characterization (GSI, 2009c).

Data generated in the field will be documented and managed as described in the RM11E Focused Sediment Characterization SAP (GSI, 2009a) and Section 7 of the Work Plan. Significant deviations from those documents, as well as key components worthy of restatement, are described in this section.

6.1 Field Data Management

6.1.1 Field Documentation

Field activities and observations will be documented in field logbooks during implementation of the sampling activities. Field forms describing the sediment characteristics will be completed for all sediment samples. Chain-of-custody forms will be maintained as part of the field records. Examples of field data sheets and forms are provided in Attachment 1 of this SAP.

The field records will be kept in the project file as a permanent record of the sampling or field measurement activities. All field records will be copied, scanned, and/or entered into an electronic spreadsheet to create an electronic record for the project file. QA reviews by the FD or SAC will check for electronic/hard copy consistencies and identify anomalous values or erroneous entries.

6.1.2 Sample Identification

All samples will be assigned a unique ID consisting of five components that will indicate the sampling event, type of study, type of sample, station ID, and field QC sample type (if applicable):

- The first component is “RM11E,” identifying the data as belonging to the RM11E Project Area.
- The second component begins with a “PW”, designating the porewater study.
- The third component will be either a “P” indicating a PE porewater exposure, a “W” indicating a PE overlying water exposure, or a “G,” representing the diver grab surface sediment sample type.
- This will be followed by the unique station ID. For this study, the first unique station will be designated 0001.

- The final component identifies the QC sample type. For split samples, a number of 500 will be integrated with the station number of the original sample. For equipment rinsate blanks, a number of 900 will be integrated with the station number.

Examples of the unique sample identifiers are shown in Table 4-2 and below:

- RM11E-PWP001: A porewater sample from Station 001.
- RM11E-PWG502: Duplicate grab sediment sample from Station 002.

6.2 Electronic Data Management

The electronic field data will be incorporated into the project database by the DM. Management of electronic data files is described in Section 7 of the Work Plan and data from this and the other supplemental investigations will be managed in accordance with those guidelines.

Section 7. Schedule and Reporting

The schedule for deliverables is presented in Table 7-1. A pre-deployment field reconnaissance took place in conjunction with the under-dock sediment sampling on May 1 and 2, 2014. The findings of the reconnaissance and modifications to the sampling technique have been included in this SAP

As discussed in Section 2.2 the preferred time to collect porewater data from sediment at RM11E is when the river is at its lowest stage, which normally occurs during August through November each year (Figure 4-1). Based on these seasonal fluctuations, the placement of the passive samplers currently is scheduled to occur in early August 2014 (Table 7-1). Timing of the placement of samplers, to the extent practicable, will be timed to avoid potential periods of non-typical sediment disturbance in the RM11E Project Area, such as dredging or dock repairs.

Passive samplers will be deployed for a period of approximately 60 days. This deployment schedule is longer than that used previously on the Lower Duwamish Waterway, but based on those data; Dr. Gschwend recommended a longer exposure period to allow for better equilibration and porewater measurements for the Willamette River program. Actual deployment and retrieval times may vary from the 60-day target based on vessel traffic considerations at the Glacier NW and Cargill docks.

Based on an anticipated deployment in early August 2014, samplers will be retrieved in early October 2014. Chemical analyses will take 30 days; validated data then would be expected 3 weeks after that. The draft Porewater Characterization Report will be delivered to EPA within 90 days of retrieval of the samplers.

Reporting will be consistent with that described in Section 9.4 of the Work Plan. The Porewater Characterization Report will document field activities and analytical results from the porewater investigation and describe any deviations from this SAP. The Porewater Characterization Report also will include the evaluations described in Section 6.2.4 of the Work Plan for the Phase 1 Porewater Characterization and compare the measured porewater data from this study with (1) calculated porewater values from the two-carbon partitioning model and (2) estimated porewater PCB concentrations using the same Equilibrium Partitioning approach used in the Draft Final RI Report and the Draft FS Report. The capping models will be developed separately as part of the RM11E Implementability Study.

As discussed in Section 2.1.1, porewater data and associated modeling will be used in the Recontamination Assessment and Implementability Study to evaluate the potential recontamination of surface sediment, monitored natural recovery, design thickness and efficacy of a conventional cap, levels of in situ treatment (such as activated carbon to sequester mobile PCBs), and the relative risks of remaining PCBs in post-dredging residuals. To appropriately consider the findings of the porewater sampling program in the Recontamination Assessment and Implementability Study, those draft reports will be submitted 60 days after submission of the Porewater Characterization Report.

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Table 2-1. Two-Carbon EqP and Cap Model Parameters and Data Source

Model Parameters		Data Source
Contaminant Properties		
Contaminant		PCBs
Sediment-water partitioning coefficients		Porewater Study
Water Diffusivity		Porewater Study
Flux of PCBs sediment surface to overlying water		Porewater Study
Flux of subsurface PCBs to surface sediments [1]		Porewater Study
Estimated diffusive flux rates		Porewater Study
Contaminated Sediment		
Depth of surface layer		Portland Harbor RI
Depth(s) of contaminated sediment layer(s)		RM11E Database
Surface sediment area		RM11E GIS-calculated
Subsurface sediment area(s)		RM11E GIS-calculated
Contaminant Concentrations		RM11E Database
	Surface sediment	2012 Supplemental Data
	Subsurface sediment layer(s)	Porewater Study Data
Fraction Organic Carbon		RM11E Database
	Surface sediment	2012 Supplemental Data
	Subsurface sediment layer(s)	Porewater Study Data
Fraction Black Carbon		RM11E Database
	Surface sediment	2012 Supplemental Data
	Subsurface sediment layer(s)	Porewater Study Data
System Properties		
Suspended solids concentration in water (g/m^3)		Portland Harbor RI
Sediment porosity		RM11E Recontamination Assessment
Sediment particle density		RM11E Recontamination Assessment
Wind speed		Portland Harbor RI
Resuspension velocity		RM11E Hydrodynamic Study
Burial velocity		RM11E Hydrodynamic Study
Settling velocity		RM11E Hydrodynamic Study
Surface Water Column		
Flow through (m^3/y)		Portland Harbor RI
Residence time, year		Portland Harbor RI
Project surface area		RM11E GIS-calculated
Project water depth		RM11E Bathymetric Data
Concentration of contaminant in surface water (µg/L)		Portland Harbor RI
Subsurface Water		
Hydraulic Gradient		RM11E Recontamination Assessment
Hydraulic Conductivity (by sediment layer)		RM11E Recontamination Assessment
Effective Porosity (by sediment layer)		RM11E Recontamination Assessment
Darcy Velocity		RM11E Recontamination Assessment
Average Linear Groundwater Velocity		RM11E Recontamination Assessment

Table 2-1. Two-Carbon EqP and Cap Model Parameters and Data Source

Model Parameters	Data Source
Contaminant concentration in upland groundwater wells	RM11E Supplemental RI/FS
Advective flux of contaminants to surface sediments	Porewater Study
Estimated advective flux rates	Porewater Study
Active Cap Properties	
Active Cap Layer thickness	TBD
Cap Materials -Granular (G) or Consolidated Silty/Clay (C)	TBD
Active Cap consolidation depth	TBD
Underlying sediment consolidation due to cap placement	TBD
Porosity	TBD
Particle Density	TBD
Active Layer Loading $kg/m^2/cm$	TBD
Kd in active layer	Porewater Study
Depth of Interest, *z- from cap-water interface*	Porewater Study
Fraction organic carbon at depth of interest	Porewater Study
Sediment/Conventional Cap Properties	
Contaminant Pore Water Concentration	Porewater Study
Conventional Cap layer fraction organic carbon	TBD
Colloidal Organic Carbon Concentration	TBD
Conventional Cap Placed thickness	TBD
Conventional Cap Consolidation Depth	TBD
Conventional Cap Layer Thickness	TBD
Cap Materials -Granular (G) or Consolidated Silty/Clay (C)	TBD
Conventional Cap Layer Porosity	TBD
Conventional Cap Layer Particle Density	TBD
Conventional Cap Layer Diffusion Coefficient	TBD

Note:

[1] If advective flux is observed, existing subsurface sediment data would be used to calculate subsurface porewater concentrations and as model inputs for subsurface sediment concentrations.

Table 2-2. Physical Characteristics and Total PCB Concentrations of Select Sediment Samples.

Sample Location											Conventionals		Grain Size	Total PCBs	
Proposed Porewater Location ID	Sample Name	Depth Class	Location Name	Sample Coordinates[1]		Elevation	River Mile	Sample Date	Sampling Interval (cm)		Total organic carbon	Total solids	Percent Fines (clay + silt)	Total PCBs[2]	Qualifier
				Northing	Easting				Start	End	pct	pct	pct	ug/kg	
PW001[3]	LW3-UG02	Surface Sediment	UG02	689302.7147	7643999.701	-40	11.2	1/31/2007	0	23	0.36	79.5	3.66	6000	JT
	LW3-UC02-B	Subsurface Sediment	UC02	689306.2653	7644001.036	-40	11.2	2/7/2007	30	90	0.11	79.6	0.99	2.2	UT
	LW3-UC02-C	Subsurface Sediment	UC02	689306.2653	7644001.036	-40	11.2	2/7/2007	90	216	0.16	86.9	1.39	2	UT
PW002	RM11E-G022	Surface Sediment	G022	689344.289	7644176.022	-23	11.3	5/7/2009	0	21	2.03	60.8	37.5	180	JT
	RM11E-C019-C	Subsurface Sediment	C019	689335.281	7644172.393	-28	11.3	5/21/2009	91.4	152.4	1.96	59.8	--	340	T
	RM11E-C019-D	Subsurface Sediment	C019	689335.281	7644172.393	-28	11.3	5/21/2009	152.4	213.4	2.1	59.1	63.2	9000	T
	RM11E-C019-B	Subsurface Sediment	C019	689335.281	7644172.393	-28	11.3	5/21/2009	30.5	91.4	1.92	57.8	57.1	210	T
	RM11E-C019-E	Subsurface Sediment	C019	689335.281	7644172.393	-28	11.3	5/21/2009	213	301	2.65	61	--	75	T
PW003	RM11E-G026	Surface Sediment	G026	689248.567	7644329.234	-16	11.3	5/8/2009	0	17	1.35	56.5	33.8	1400	JT
	RM11E-C022-B	Subsurface Sediment	C022	689257.569	7644334.365	-15	11.3	5/20/2009	30.5	91.4	1.67	66.9	--	390	JT
	RM11E-C022-C	Subsurface Sediment	C022	689257.569	7644334.365	-15	11.3	5/20/2009	91.4	140	1.91	61.6	55.4	2800	T
PW004	RM11E-G027	Surface Sediment	G027	689168.259	7644261.375	-43	11.3	5/8/2009	0	22	0.4	67	--	140	JT
	RM11E-C023-B	Subsurface Sediment	C023	689165.162	7644265.515	-42	11.3	5/21/2009	30.5	91.4	0.86	74.8	9.3	4300	T
	RM11E-C023-C	Subsurface Sediment	C023	689165.162	7644265.515	-42	11.3	5/21/2009	91.4	152.4	1.17	68.1	--	2100	T
	RM11E-C023-D	Subsurface Sediment	C023	689165.162	7644265.515	-42	11.3	5/21/2009	152.4	213.4	1.62	64.9	--	2200	JT
	RM11E-C023-E	Subsurface Sediment	C023	689165.162	7644265.515	-42	11.3	5/21/2009	213.4	301	1.29	63.9	28.7	670	T
PW005	RM11E-G028	Surface Sediment	G028	689072.902	7644163.446	-51	11.3	5/8/2009	0	28	1.4	65.3	17.8	380	JT
	RM11E-C024-B	Subsurface Sediment	C024	689065.852	7644169.518	-51	11.3	5/27/2009	30.5	91.4	0.06	79.3	0.73	1	UT
	RM11E-C024-C	Subsurface Sediment	C024	689065.852	7644169.518	-51	11.3	5/27/2009	91.4	152.4	0.04	79.2	--	8.4	T
	RM11E-C024-D	Subsurface Sediment	C024	689065.852	7644169.518	-51	11.3	5/27/2009	152.4	213.4	0.06	84.9	--	1	UT
PW006	RM11E-G033	Surface Sediment	G033	689004.644	7644579.379	-47	11.4	5/13/2009	0	25	0.46	67.1	8.58	2000	JT
	RM11E-C029-B	Subsurface Sediment	C029	689005.433	7644581.765	-22	11.4	6/23/2009	30.5	91.4	0.84	64.1	62.9	1.3	UT
	RM11E-C029-C	Subsurface Sediment	C029	689005.433	7644581.765	-22	11.4	6/23/2009	91.4	152.4	0.45	69.9	--	1.3	UT
	RM11E-C029-D	Subsurface Sediment	C029	689005.433	7644581.765	-22	11.4	6/23/2009	152.4	213.4	0.89	65.8	--	1.3	UT
	RM11E-C029-E	Subsurface Sediment	C029	689005.433	7644581.765	-22	11.4	6/23/2009	213.4	304.8	1	67.4	--	1.3	UT
	RM11E-C029-F	Subsurface Sediment	C029	689005.433	7644581.765	-22	11.4	6/23/2009	304.8	396.2	0.45	68.4	--	1.3	UT
	RM11E-C029-G	Subsurface Sediment	C029	689005.433	7644581.765	-22	11.4	6/23/2009	396.2	436	0.26	67.5	--	1.3	UT
--	LW3-G776	Surface Sediment	G776	689693	7643560	-39	11.1	12/5/2007	0	22	0.75	66.2	14	2700	JT
--	LW3-UG01	Surface Sediment	UG01	689712.7928	7643655.366	-22	11.1	2/1/2007	0	19	1.12	71.3	4.88	320	NJT
--	RM11E-C010-A (R2)	Surface Sediment	C010-R2	689650.969	7643668.096	-50	11.1	5/22/2009	0	30	1.73	76.7	21.2	100	T
--	RM11E-G006	Surface Sediment	G006	689808.294	7643449.952	-48	11.1	5/5/2009	0	12	1.95	67	--	230	JT
--	RM11E-G008	Surface Sediment	G008	689807.121	7643609.54	-15	11.1	5/6/2009	0	17	3.06	44.8	32.8	35	JT
--	RM11E-G010	Surface Sediment	G010	689647.586	7643468.155	-52	11.1	5/6/2009	0	20	0.32	83.6	--	10	JT
--	RM11E-G013	Surface Sediment	G013	689650.365	7643666.758	-47	11.1	6/15/2009	0	17	1.27	82.3	--	94	JT
--	RM11E-G076	Surface Sediment	G076	689723.505	7643543.729	-42	11.1	10/31/2013	0	25	0.974	69.7	8.99	110	T
--	RM11E-C010-B	Subsurface Sediment	C010-R2	689650.969	7643668.096	-50	11.1	5/22/2009	30.5	91.4	2.25	78	--	63	T
--	RM11E-C010-C	Subsurface Sediment	C010-R2	689650.969	7643668.096	-50	11.1	5/22/2009	91.4	152.4	0.17	74.8	--	11	JT
--	RM11E-C010-D	Subsurface Sediment	C010-R2	689650.969	7643668.096	-50	11.1	5/22/2009	152.4	169	0.05	88.1	--	1	UT

Table 2-2. Physical Characteristics and Total PCB Concentrations of Select Sediment Samples.

Sample Location											Conventionals		Grain Size	Total PCBs	
Proposed Porewater Location ID	Sample Name	Depth Class	Location Name	Sample Coordinates[1]		Elevation	River Mile	Sample Date	Sampling Interval (cm)		Total organic carbon	Total solids	Percent Fines (clay + silt)	Total PCBs[2]	Qualifier
				Northing	Easting				Start	End	pct	pct	pct	ug/kg	
--	LW3-G777	Surface Sediment	G777	689429	7643780	-39	11.2	12/5/2007	0	21	0.17	84.1	1.68	57	JT
--	RM11E-C012-A	Surface Sediment	C012	689614.395	7643820.979	-37	11.2	5/20/2009	0	30.5	1.35	68.3	--	98	JT
--	RM11E-G015	Surface Sediment	G015	689614.385	7643820.976	-36	11.2	6/16/2009	0	29	1.57	67.4	27	120	T
--	RM11E-G019	Surface Sediment	G019	689411.595	7643927.274	-44	11.2	6/16/2009	0	23	0.81	76.8	--	110	T
--	RM11E-G020	Surface Sediment	G020	689313.605	7643838.02	-55	11.2	5/7/2009	0	27	0.2	86	--	91	JT
--	RM11E-G065	Surface Sediment	G065	689449.982	7644061.258	-12	11.2	6/18/2009	0	28	1.24	60.8	--	170	JT
--	RM11E-C012-B	Subsurface Sediment	C012	689614.395	7643820.979	-37	11.2	5/20/2009	30.5	91.4	1.82	66.5	30.5	160	T
--	RM11E-C012-C	Subsurface Sediment	C012	689614.395	7643820.979	-37	11.2	5/20/2009	91.4	152.4	0.21	88.4	--	5.7	T
--	RM11E-C012-D	Subsurface Sediment	C012	689614.395	7643820.979	-37	11.2	5/20/2009	152.4	213.4	0.07	86	--	1	UT
--	RM11E-C012-E	Subsurface Sediment	C012	689614.395	7643820.979	-37	11.2	5/20/2009	213	274	0.02 U	82.7	--	1.3	UT
--	LW3-G778	Surface Sediment	G778	689191	7644130	-38	11.3	12/5/2007	0	22	0.4	74.8	6.01	580	T
--	LW3-G779	Surface Sediment	G779	688958	7644450	-41	11.3	12/5/2007	0	23	0.43	79.1	4.77	200	JT
--	LW3-GCA11E-C00	Surface Sediment	GCA11E	689225.7872	7644252.526	-22	11.3	12/6/2007	0	24	0.81	64	6.58	6600	JT
--	LW3-GCRSP11E	Surface Sediment	GCRSP11E	689209.5283	7644357.169	-12	11.3	10/18/2007	0	12	1.13	64.1	24.2	11	T
--	RM11E-G023	Surface Sediment	G023	689279.58	7644091.381	-51	11.3	5/7/2009	0	17	0.51	69.5	--	1000	JT
--	RM11E-G024	Surface Sediment	G024	689175.117	7643998.105	-52	11.3	5/7/2009	0	20	0.36	78.2	4.67	77	JT
--	RM11E-G025	Surface Sediment	G025	689032.375	7643983.176	-52	11.3	5/8/2009	0	25	1.4	63.5	--	96	JT
--	RM11E-G029	Surface Sediment	G029	689157.349	7644476.222	-33	11.3	5/12/2009	0	19	1.18	69.4	5.95	2300	JT
--	RM11E-G030	Surface Sediment	G030	689066.829	7644394.977	-51	11.3	5/8/2009	0	20	0.89	65.6	--	190	JT
--	RM11E-G066	Surface Sediment	G066	689066.88	7644539.042	-27	11.3	6/18/2009	0	23	0.99	68.8	18.8	350	JT
--	RM11E-G067	Surface Sediment	G067	689290.948	7644246.88	-22	11.3	6/15/2009	0	26	0.33	71	8.31	56	T
--	RM11E-G082	Surface Sediment	G082	689107.134	7644280.414	-35.2	11.3	10/31/2013	0	20	0.626	69.3	4.81	910	T
--	RM11E-G036	Surface Sediment	G036	688889.563	7644708.497	-23	11.4	6/15/2009	0	25	1.4	64.9	17.4	1300	JT
--	RM11E-G037	Surface Sediment	G037	688803.942	7644574.373	-56	11.4	5/13/2009	0	28	1.1	64.2	--	110	JT
--	RM11E-G039	Surface Sediment	G039	688796.273	7644755.595	-23	11.4	6/15/2009	0	28	0.61	70.5	18.8	22	JT
--	RM11E-G064	Surface Sediment	G064	688687.254	7644823.096	-27	11.4	6/15/2009	0	15	0.59	85.5	--	1600	JT
--	RM11E-C033A	Surface Sediment	C033A	688819.388	7644600.851	-53.65	11.4	6/4/2009	0	30.5	1.9	61.3	--	190	UT
--	RM11E-G043	Surface Sediment	G043	688577.551	7644827.386	-48.15	11.4	7/26/2009	0	30	1.77	53.4	--	16.3	JT

Notes:

1. Northing and easting coordinates exist in the following coordinate system: NAD 83 Oregon State Plane North Zone, International Feet.

2. "Total PCBs" reflect total PCB congener results where available, and total PCB Aroclor results where congener analysis was not conducted. Analyte group totals follow the FS data summation as described in Appendix R of the Draft FS report for the Portland Harbor (Anchor QEA et al., 2012). Calculated totals are the sum of all detected concentrations, and non-detected results are included in the summation at one-half the method detection limit (MDL). If none of the analytes is detected for a given sample, then the highest MDL is used for the summation.

3. Sample LW3-UG02 was collected by the Lower Willamette Group. Sample RM11E-G079 was collected in 2013 from approximately the same location. Both station results are reported here.

Acronyms:

PCB = Polychlorinated biphenyl
TOC = Total Organic Carbon
µg/kg = micrograms per kilogram
-- No Data

U = The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J = The associated numerical value is an estimated quantity.
T = The associated numerical value was mathematically derived (e.g., from summing multiple analyte results such as Aroclors, or calculating the average of multiple results for a single analyte).

Table 2-3. Location and Rationale for Porewater Sampling Locations. Stations presented from downstream to upstream.

Proposed Porewater Location ID	Previous Sample Locations	Sample Coordinates[1]		Previous Sample Code	Sample Date	Start Depth (cm)	End Depth (cm)	Total PCB Aroclors (µg/kg)[2]		TOC (%)	Sum PCB Aroclor C_{pw} (ng/L)[3,4]	Comprehensive Benthic Risk Area	In-Situ Treatment/ Engineered Cap	Removal	Subsurface Total PCB Concentration >1,000 µg/kg
PW001[5]	UG02	689302.7	7643999.7	LW3-UG02	1/31/2007	0	23	6000	JT	0.36	3417.1	Yes	No	Yes	No
	G079	689302.8	7644009.5	RM11E-G079	10/31/2013	0	23	2300	JT	0.491	936.1				
	UC02	689306.3	7644001.0	LW3-UC02-B	2/7/2007	30	90	2.2	UT	0.11	not calculated				
	UC02	689306.3	7644001.0	LW3-UC02-C	2/7/2007	90	216	2	UT	0.16	not calculated				
PW002	G022	689344.3	7644176.0	RM11E-G022	5/7/2009	0	21	180	JT	2.03	18.3	No	Yes	No	Yes
	C019	689335.3	7644172.4	RM11E-C019-B	5/21/2009	31	91	210	T	1.92	22.7				
		689335.3	7644172.4	RM11E-C019-C	5/21/2009	91	152	340	T	1.96	36.1				
		689335.3	7644172.4	RM11E-C019-D	5/21/2009	152	213	9000	T	2.1	868.2				
		689335.3	7644172.4	RM11E-C019-E	5/21/2009	213	301	75	T	2.65	5.5				
PW003	G026	689248.6	7644329.2	RM11E-G026	5/8/2009	0	17	1400	JT	1.35	216.7	Yes	Yes	No	Yes
	C022	689257.6	7644334.4	RM11E-C022-B	5/20/2009	31	91	390	JT	1.67	46.7				
		689257.6	7644334.4	RM11E-C022-C	5/20/2009	91	140	2800	T	1.91	164.1				
PW004	G027	689168.3	7644261.4	RM11E-G027	5/8/2009	0	22	140	JT	0.4	57.2	Yes	No	Yes	Yes
	C023	689165.2	7644265.5	RM11E-C023-B	5/21/2009	31	91	4300	T	0.86	801.7				
		689165.2	7644265.5	RM11E-C023-C	5/21/2009	91	152	2100	T	1.17	214.3				
		689165.2	7644265.5	RM11E-C023-D	5/21/2009	152	213	2200	JT	1.62	229.5				
		689165.2	7644265.5	RM11E-C023-E	5/21/2009	213	301	670	T	1.29	104.9				
PW005	G028	689072.9	7644163.4	RM11E-G028	5/8/2009	0	28	380	JT	1.4	49.05	No	No	Yes	No
	C024	689065.9	7644169.5	RM11E-C024-B	5/27/2009	31	91	1.0	UT	0.06	not calculated				
	C024	689065.9	7644169.5	RM11E-C024-C	5/27/2009	91	152	8.4	T	0.04	not calculated				
	C024	689065.9	7644169.5	RM11E-C024-D	5/27/2009	152	213	1	UT	0.06	not calculated				
PW006	G033	689004.6	7644579.4	RM11E-G033	5/13/2009	0	25	2000	JT	0.46	908.4	Yes	Yes	No	No
	C029	689005.4	7644581.8	RM11E-C029-B	6/23/2009	31	91	1.3	UT	0.84	1.0				
	C029	689005.4	7644581.8	RM11E-C029-C	6/23/2009	91	152	1.3	UT	0.45	not calculated				
	C029	689005.4	7644581.8	RM11E-C029-D	6/23/2009	152	213	1.3	UT	0.89	not calculated				
	C029	689005.4	7644581.8	RM11E-C029-E	6/23/2009	213	305	1.3	UT	1	not calculated				
	C029	689005.4	7644581.8	RM11E-C029-F	6/23/2009	305	396	1.3	UT	0.45	not calculated				
	C029	689005.4	7644581.8	RM11E-C029-G	6/23/2009	396	436	1.3	UT	0.26	not calculated				
PW007 (Reference)	C043	687388.4	7645488.7	RM11E-C043-A	5/19/2009	0	30	1.7	UT	6.23	not calculated	Proposed Upstream Reference Area			
	C043	687388.4	7645488.7	RM11E-C043-B	5/19/2009	31	95	1	UT	0.83	not calculated				

Notes:

1. Northing and easting coordinates exist in the following coordinate system: NAD 83 Oregon State Plane North Zone, International Feet.
2. Total PCB Aroclors were calculated using the FS data summation rules as described in Appendix R of the Draft FS report for the Portland Harbor (Anchor QEA et al., 2012). Calculated totals are the sum of all detected concentrations, and non-detected results are included in the summation at one-half the method detection limit (MDL). If none of the analytes is detected for a given sample, then the highest MDL is used for the summation.
3. EPA Guidance for calculating EqP porewater concentrations (C_{pw}) requires a detectable PCB and > 0.2% TOC. Where either of those conditions existed, the C_{pw} was not calculated
4. Only the detected Aroclor concentrations were used to estimate the C_{pw} for each individual congener expected to be in the RM11E sediments, and then summing those individual congener porewater concentrations.
5. Sample LW3-UG02 was collected by the Lower Willamette Group. Sample RM11E-G079 was collected in 2013 from approximately the same location. Both station results are reported here.

Acronyms:

PCB = Polychlorinated biphenyl
TOC = Total Organic Carbon
µg/kg = micrograms per kilogram
ng/L = nanogram/Liter

U = The material was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
J = The associated numerical value is an estimated quantity.
T = The associated numerical value was mathematically derived (e.g., from summing multiple analyte results such as Aroclors, or calculating the average of multiple results for a single analyte).

Table 2-4. Portland Harbor Superfund Site Preliminary Remediation Goals (EPA Recalculation March 2014)

Preliminary Remediation Action Objectives (RAOs)	Total PCBs[1] PRG
Sediment	**(µg/kg)**
RAO 1 – Human Health Sediments: Reduce to acceptable levels human health risks from exposure to contaminated sediments resulting from incidental ingestion of and dermal contact with sediments, and comply with identified ARARs.	370
RAO 2 – Human Health Biota Ingestion: Reduce to acceptable levels human health risks from indirect exposures to COCs through ingestion of fish and shellfish that occur via bioaccumulation pathways from sediment and/or surface water and comply with identified ARARs.	6
RAO 5 – Ecological Sediments: Reduce to acceptable levels the risks to ecological receptors resulting from the ingestion of and direct contact with contaminated sediments and comply with identified ARARs.	64
RAO 6 – Ecological Biota (Prey) Ingestion: Reduce to acceptable levels risks to ecological receptors from indirect exposures through ingestion of prey to COCs in sediments via bioaccumulation pathways from sediment and/or surface water and comply with identified ARARs.	31
Water (Surface and Groundwater)	**(ng/L)**
RAO 3 – Human Health Surface Water: Reduce risks from COCs in surface water at the Site to acceptable exposure levels that are protective of human health risks from ingestion of, inhalation of, and dermal contact with surface water; protect the drinking water beneficial use of the Willamette River at the Site; and comply with identified ARARs.	0.0064
RAO 4 – Human Health Groundwater: Reduce to acceptable levels human health risks resulting from direct exposure to contaminated groundwater and indirect exposure to contaminated groundwater through fish and shellfish consumption, and comply with identified ARARs.	500
RAO 7 – Ecological Surface Water: Reduce risks from COCs in surface water at the Site to acceptable exposure levels that are protective of ecological receptors based on the ingestion of and direct contact with surface water and comply with identified ARARs.	14
RAO 8 – Ecological Groundwater: Reduce to acceptable levels the risks to ecological receptors resulting from the ingestion of and direct contact with contaminated groundwater and indirect exposures through ingestion of prey via bioaccumulation pathways from groundwater, and comply with identified ARARs.	---

Notes:

1. "Total PCBs" reflect total PCB congener results where available, and total PCB Aroclor results where congener analysis was not conducted.

Acronyms:

PCB = Polychlorinated biphenyl

µg/kg = Micrograms per kilogram

ng/L = nanogram/Liter

Table 3-1. Project Contact Information

Contact/Company	Title	E-Mail Address	Phone Number
Paul Fuglevand, DOF	Project Manager	pfuglevand@dofnw.com	425-827-4588
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Laboratory	**Project Manager**	**E-Mail Address**	**Phone Number**
ALS Life Sciences Division/ Environmental	Jeff Christian	Jeff.Christian@alsglobal.com	360-501-3316
Oversight	**Remedial Project Manager**	**E-Mail Address**	**Phone Number**
Environmental Protection Agency (EPA)	Sean Sheldrake	sheldrake.sean@epa.gov	206-553-1220

Table 4-1. Sampling Media, Number of Samples, and Analyte Groups

Data Needed to Address the Objectives	Associated Field and Analytical Actions	Number of Samples			
		PCB Congeners	PCB Aroclors	Conventionals [1]	Black Carbon
Quantify PCB congener levels in sediment porewater.	Performance Reference Compound Confirmation	8	--	--	--
	• Onsite porewater samples (6) • Onsite field replicate (1) • Upriver porewater sample (1)	8	--	--	--
Quantify PCB congeners in overlying surface water immediately above porewater sample location.	• Onsite porewater samples (6) • Onsite field replicate (1) • Upriver porewater sample (1)	8	--	--	--
Bulk sediments analyses	• Onsite porewater samples (6) • Onsite field replicate (1) • Upriver porewater sample (1)	8	8	8	8

Notes:

1. Conventionals = Total Solids, Total Organic Carbon, Grain Size

Table 4-2. Sample Coordinates, Location ID, and Sample ID

Location ID	Grab/Core Pair	Sample Location [1,2]				Sample Matrix	Sample ID
		Northing	Easting	Latitude	Longitude		
RM11E-PW001	G079 and UG02/UC02	689302.7	7643999.7	45.536728	-122.678338	PE Porewater	RM11E-PWP001
						PE Surface Water	RM11E-PWW001
						Sediment	RM11E-PWG001
RM11E-PW002	G022/C019	689344.3	7644176.0	45.536855	-122.677654	PE Porewater	RM11E-PWP002
						PE Surface Water	RM11E-PWW002
						Sediment	RM11E-PWG002
RM11E-PW003	G026/C022	689248.6	7644329.2	45.536604	-122.677047	PE Porewater	RM11E-PWP003
						PE Surface Water	RM11E-PWW003
						Sediment	RM11E-PWG003
RM11E-PW004	G027/C023	689168.3	7644261.4	45.536379	-122.677303	PE Porewater	RM11E-PWP004
						PE Surface Water	RM11E-PWW004
						Sediment	RM11E-PWG004
RM11E-PW005	G028/C024	689072.9	7644163.4	45.53611	-122.677675	PE Porewater	RM11E-PWP005
						PE Surface Water	RM11E-PWW005
						Sediment	RM11E-PWG005
RM11E-PW006	G033/C029	689004.6	7644579.4	45.535954	-122.676045	PE Porewater	RM11E-PWP006
						PE Surface Water	RM11E-PWW006
						Sediment	RM11E-PWG006
RM11E-PW007	C043	687388.4	7645488.7	45.53159	-122.672328	PE Porewater	RM11E-PWP007
						PE Surface Water	RM11E-PWW007
						Sediment	RM11E-PWG007
RM11E-PW504 [4]	G027/C023	689168.3	7644261.4	45.536379	-122.677303	PE Porewater	RM11E-PWP504
						PE Surface Water	RM11E-PWW504
						Sediment	RM11E-PWG504

Notes:

1. Northing and easting coordinates exist in the following coordinate system: NAD 83 Oregon State Plane North Zone, International Feet.
2. Latitude and longitude coordinates exist in the following coordinate system: “WGS 1984, decimal degrees”
3. Sample RM11E-PW504 will be a field replicate of sample RM11E-PW004.

Table 4-3. Specific Samples, Analyses, Assigned Lab and Station Labelling

Location ID	Original Grab/Core Pair	Sample Method	Portion of Sampler for Analysis (5cm widths)	Analyses [1]	Laboratory	Labeling
PW001	G079 and UG02/UC02	PE Sampler 1	0-20 cm in water	PCB Congeners by EPA 1668C	ALS	RM11E-PWW001
			0-30 cm in sediment			RM11E-PWP001
			0-20 cm in water	PCB Congeners by HRGC/LRM -SIM	MIT	RM11E-PWW001b
			0-30 cm in sediment			RM11E-PWP001b
		PE Sampler 2	0-20 cm in water	Archive 2 PE subsamples	ALS	RM11E-PWW001
			0-30 cm in sediment			RM11E-PWP001
		Bulk Sediment Core Sample	0-30 cm in sediment	PCB Aroclors, Black Carbon, Conventionals	ALS	RM11E-PWG001
				Black Carbon CTO-375; Total Organic Carbon; PCB Congeners by HRGC/LRM -SIM	MIT	RM11E-PWG001b
PW002	G022/C019	PE Sampler	0-20 cm in water	PCB Congeners by EPA 1668C	ALS	RM11E-PWW002
			0-30 cm in sediment			RM11E-PWP002
			0-20 cm in water	Archive 2 PE subsamples		RM11E-PWW002
			0-30 cm in sediment			RM11E-PWP002
		Bulk Sediment Core Sample	0-30 cm in sediment	PCB Aroclors, Black Carbon, Conventionals		RM11E-PWG002
PW003	G026/C022	PE Sampler	0-10cm in water	PCB Congeners by EPA 1668C	ALS	RM11E-PWW003
			0-30 cm in sediment			RM11E-PWP003
			0-20 cm in water	Archive 2 PE subsamples		RM11E-PWW003
			0-30cm in sediment			RM11E-PWP003
		Bulk Sediment Core Sample	0-30 cm in sediment	PCB Aroclors, Black Carbon, Conventionals [2]		RM11E-PWG003
PW004	G027/C023	PE Sampler 1	0-20 cm in water	PCB Congeners by EPA 1668C	ALS	RM11E-PWW004
			0-30 cm in sediment			RM11E-PWP004
			0-20 cm in water	PCB Congeners by HRGC/LRM -SIM (MIT)	MIT	RM11E-PWW004b
			0-30 cm in sediment			RM11E-PWP004b
		PE Sampler 2	0-20 cm in water	Field Replicate (PW504) Archive 1 PE subsample	ALS	RM11E-PWW504
			0-30 cm in sediment			RM11E-PWP504
		Bulk Sediment Core Sample	0-30 cm in sediment	PCB Aroclors, Black Carbon, Conventionals		RM11E-PWG004
		Bulk Sediment Core Sample	0-30 cm in sediment	PCB Aroclors, Black Carbon, Conventionals	MIT	RM11E-PWG004b
PW504	G027/C023 FIELD REPLICATE	PE Sampler	0-20 cm in water	PCB Congeners by EPA 1668C	ALS	RM11E-PWW504
			0-30 cm in sediment			RM11E-PWP504
		Bulk Sediment Core Sample	0-30 cm in sediment	PCB Aroclors, Black Carbon, Conventionals		RM11E-PWG504

Table 4-3. Specific Samples, Analyses, Assigned Lab and Station Labelling

Location ID	Original Grab/Core Pair	Sample Method	Portion of Sampler for Analysis (5cm widths)	Analyses [1]	Laboratory	Labeling
PW005	G028/C024	PE Sampler	0-20 cm in water	PCB Congeners by EPA 1668C	ALS	RM11E-PWW005
			0-30 cm in sediment			RM11E-PWP005
			0-20 cm in water	Archive 2 PE subsamples		RM11E-PWW005
			0-30 cm in sediment			RM11E-PWP005
		Bulk Sediment Core Sample	0-30 cm in sediment	PCB Aroclors, Black Carbon, Conventionals		RM11E-PWG005
PW006	G033/C029	PE Sampler	0-10cm in water	PCB Congeners by EPA 1668C	ALS	RM11E-PWW006
			0-30 cm in sediment			RM11E-PWP006
			0-10cm in water	Archive 2 PE subsamples		RM11E-PWW006
			0-30cm in sediment			RM11E-PWP006
		Bulk Sediment Core Sample	0-30 cm in sediment	PCB Aroclors, Black Carbon, Conventionals		RM11E-PWG006
PW007	C043 (reference)	PE Sampler	0-10cm in water	PCB Congeners by EPA 1668C	ALS	RM11E-PWW007
			0-30 cm in sediment			RM11E-PWP007
			0-20 cm in water	Archive 2 PE subsamples		RM11E-PWW007
			0-30 cm in sediment			RM11E-PWP007
		Bulk Sediment Core Sample	0-30 cm in sediment	PCB Aroclors, Black Carbon, Conventionals		RM11E-PWG007

Notes:

1. Laboratory QA replicates are not identified on this table

2. Conventionals include total solids, total organic carbon, and grain size

Acronyms

ALS = ALS Laboratories

MIT = Massachusetts Institute of Technology

PCB = Polychlorinated biphenyl

PE = Polyethylene

HRGC/LRM-SIM = High Resolution Gas Chromatography/Low Resolution Mass Spectrophotometry - Selected Ion Monitoring. This method is commonly referred to as low resolution GC/MS.

Table 4-4. Container and Preservation Requirements, Holding Times for Samples

Parameter	Minimum Sample Size (1)	Container Description (2)		Preservation Requirements	Holding Time
		Container	Volume (mL)		
Sediment Conventionals					
Grain size	100 g	16-oz glass or HDPE	480	4°C ± 2°C	6 months
Total Organic Carbon	30 g	8-oz glass or HDPE	240	4°C ± 2°C	14 days
				-20°C ± 2°C	6 months
Total Black Carbon	30 g			4°C ± 2°C	14 days
				-20°C ± 2°C	6 months
Total Solids	30 g			4°C ± 2°C	14 days
				-20°C ± 2°C	6 months
Sediment Chemistry					
Total Polychlorinated Biphenyls (PCBs) as Aroclors	100 g	8-oz glass	480	4°C ± 2°C	14 days
				-20°C ± 2°C	1 year
Low-Level PCBs (209 congeners)	100 g	8-oz amber glass	240	4°C ± 2°C	14 days
				-20°C ± 2°C	1 year
Passive Sampler Testing					
Performance Reference Compounds	100 mg	Polyethylene Sheet	---	4°C ± 2°C	1 year
Low-Level PCBs (209 congeners)	100 mg				
Archive					
Sediment Chemistry Archive (4)	250 mL	One (1) 8-oz glass	480	4°C ± 2°C	14 days
				-20°C ± 2°C	1 year
PE PCB Chemistry Archive	100 mg	Polyethylene Sheet	---	4°C ± 2°C	1 year

Notes:

1. Recommended minimum field sample sizes for one lab analysis. Actual volumes to be collected have been increased to provide a margin of error and allow for retests.
2. HDPE - high density polyethylene
3. During transport to the lab, samples will be stored on ice.
4. For every sample location, an 8-oz container is archived for any potential analysis/re-analysis deemed necessary.

Table 5-1. Range of Properties of ^{13}C-labelled PCB Congeners Used as Performance Reference Compounds and Recovery Standards

Labelled Congener	Number Chlorines	Congener Number	log K_{ow}	log K_{oc}	log $K_{pe\text{-}w}$
Performance Reference Compounds					
$^{13}C_{12}$-2,4'-DiCB	2	8	5.10	4.72	4.81
$^{13}C_{12}$-2,4,4'-TriCB	3	28	5.67	5.05	5.38
$^{13}C_{12}$-2,2',4,4'-TetraCB	4	47	5.85	5.23	5.62
$^{13}C_{12}$-2,3',4',5-TetraCB	4	70	6.2	5.40	5.91
$^{13}C_{12}$-2,2',3',4,5-PentaCB	5	97	6.29	5.74	6.00
$^{13}C_{12}$-2,3,3',5,5'-PentaCB	5	111	6.76	5.60	6.24
$^{13}C_{12}$-2,2',4,4',5,5'-HexaCB	6	153	6.92	6.06	6.81
$^{13}C_{12}$-2,2'3,3'5,5'6-HeptaCB	7	178	7.14	6.24	6.85
Recovery Standards					
$^{13}C_{12}$-2,5-DiCB	2	9	5.06	4.67	4.77
$^{13}C_{12}$-2,2'5,5'-TetraCB	4	52	5.54	5.20	5.55
$^{13}C_{12}$-2,2',4,5,5'-PentaCB	5	101	6.38	5.64	6.18
$^{13}C_{12}$-2,2',3,4,4',5'-HexaCB	6	138	6.86	6.19	6.82
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OctaCB	8	194	7.80	6.96	7.15

Notes:
Log Kow (octanol-water partitioning coefficient) values from Hawker and Connell, 1988
Log Koc (organic carbon-water partitioning coefficient) values from Hansen et al., 1999
Log Kpe-w (polyethylene-water partitioning coefficient) values from Ghosh et al., 2014

Table 5-2. Data Quality Goals for PCB Congeners in Polyethylene

Analyte	CAS No.	Reporting Limits in Polyethylene		Method Detection Limit (MDL) in Porewater		LCS	MS/MSD	Precision	Labeled Compound
		MDL	MRL	Log K_{pe-w}	C_w (ng/L)	% Recovery	% Recovery	Relative Percent Difference	% Recovery
		(ng/g PE)							
PCB 1	2051-60-7	0.2	0.5	4.173	1.34E-02	50-150	50-150	50	--
PCB 2	2051-61-8	0.01	0.03	4.403	3.95E-04	--	--	--	--
PCB 3	2051-62-9	0.2	0.5	4.403	8.90E-03	50-150	50-150	50	--
PCB 4	13029-08-8	0.4	1	4.363	1.84E-02	50-150	50-150	50	--
PCB 5	16605-91-7	0.03	0.1	4.733	4.62E-04	--	--	--	--
PCB 6	25569-80-6	0.03	0.1	4.770	4.25E-04	--	--	--	--
PCB 7	33284-50-3	0.05	0.1	4.780	8.30E-04	--	--	--	--
PCB 8	34883-43-7	0.3	1	4.733	5.55E-03	--	--	--	--
PCB 9	34883-39-1	0.05	0.1	4.773	8.43E-04	--	--	--	--
PCB 10	33146-45-1	0.05	0.1	4.553	1.40E-03	--	--	--	--
PCB 11	2050-67-1	0.3	3	4.993	2.54E-03	--	--	--	--
PCB 12	2974-92-7	0.08	0.3	4.933	8.75E-04	--	--	--	--
PCB 13	2974-90-5	0.08	0.3	5.003	7.45E-04	--	--	--	--
PCB 14	34883-41-5	0.08	0.3	4.993	7.62E-04	--	--	--	--
PCB 15	2050-68-2	0.5	0.1	5.013	4.37E-03	50-150	50-150	50	--
PCB 16	38444-78-9	0.1	0.3	4.873	1.34E-03	--	--	--	--
PCB 17	37680-66-3	0.2	0.5	4.963	2.45E-03	--	--	--	--
PCB 18	37680-65-2	0.5	1	4.953	5.57E-03	--	--	--	--
PCB 19	38444-73-4	0.1	0.3	4.733	1.85E-03	50-150	50-150	50	--
PCB 20	38444-84-7	0.5	1	5.283	2.48E-03	--	--	--	--
PCB 21	55702-46-0	0.1	0.5	5.223	7.48E-04	--	--	--	--
PCB 22	38444-85-8	0.2	0.5	5.293	1.15E-03	--	--	--	--
PCB 23	55720-44-0	0.1	0.5	5.283	6.51E-04	--	--	--	--
PCB 24	55702-45-9	0.1	0.5	5.063	1.08E-03	--	--	--	--

Table 5-2. Data Quality Goals for PCB Congeners in Polyethylene

Analyte	CAS No.	Reporting Limits in Polyethylene		Method Detection Limit (MDL) in Porewater		LCS	MS/MSD	Precision	Labeled Compound
		MDL	MRL	Log K_{pe-w}	C_w (ng/L)	% Recovery	% Recovery	Relative Percent Difference	% Recovery
		(ng/g PE)							
PCB 25	55712-37-3	0.1	0.5	5.383	5.17E-04	--	--	--	--
PCB 26	38444-81-4	0.2	0.5	5.373	8.47E-04	--	--	--	--
PCB 27	38444-76-7	0.2	0.5	5.153	1.05E-03	--	--	--	--
PCB 28	7012-37-5	0.5	1	5.383	1.97E-03	--	--	--	--
PCB 29	15862-07-4	0.2	0.5	5.313	9.73E-04	--	--	--	--
PCB 30	35693-92-6	0.5	1	5.153	3.52E-03	--	--	--	--
PCB 31	16606-02-3	0.4	1	5.383	1.55E-03	--	--	--	--
PCB 32	38444-77-8	0.2	0.5	5.153	1.41E-03	--	--	--	--
PCB 33	38444-86-9	0.1	0.5	5.313	6.08E-04	--	--	--	--
PCB 34	37680-68-5	0.2	0.5	5.373	7.41E-04	--	--	--	--
PCB 35	37680-69-6	0.2	0.5	5.533	5.86E-04	--	--	--	--
PCB 36	38444-87-0	0.2	0.5	5.593	5.11E-04	--	--	--	--
PCB 37	38444-90-5	0.3	1	5.543	9.31E-04	50-150	50-150	50	--
PCB 38	53555-66-1	0.2	0.5	5.473	6.73E-04	--	--	--	--
PCB 39	38444-88-1	0.2	0.5	5.603	5.61E-04	--	--	--	--
PCB 40	38444-93-8	0.3	1	5.373	1.27E-03	--	--	--	--
PCB 41	52663-59-9	0.3	1	5.403	1.19E-03	--	--	--	--
PCB 42	36559-22-5	0.2	0.5	5.473	5.05E-04	--	--	--	--
PCB 43	70362-46-8	0.2	1	5.463	7.75E-04	--	--	--	--
PCB 44	41464-39-5	0.5	1	5.463	1.64E-03	--	--	--	--
PCB 45	70362-45-7	0.1	0.5	5.243	7.14E-04	--	--	--	--
PCB 46	41464-47-5	0.3	0.5	5.243	1.43E-03	--	--	--	--
PCB 47	2437-79-8	0.5	1	5.563	1.30E-03	--	--	--	--
PCB 48	70362-47-9	0.2	0.5	5.493	6.43E-04	--	--	--	--

Table 5-2. Data Quality Goals for PCB Congeners in Polyethylene

Analyte	CAS No.	Reporting Limits in Polyethylene		Method Detection Limit (MDL) in Porewater		LCS	MS/MSD	Precision	Labeled Compound
		MDL	MRL	Log K_{pe-w}	C_w (ng/L)	% Recovery	% Recovery	Relative Percent Difference	% Recovery
		(ng/g PE)							
PCB 49	41464-40-8	0.3	1	5.563	7.52E-04	--	--	--	--
PCB 50	62796-65-0	0.2	0.5	5.343	6.81E-04	--	--	--	--
PCB 51	68194-04-7	0.1	0.5	5.343	5.67E-04	--	--	--	--
PCB 52	35693-99-3	0.5	1	5.553	1.33E-03	--	--	--	--
PCB 53	41464-41-9	0.2	0.5	5.333	6.97E-04	--	--	--	--
PCB 54	15968-05-5	0.3	1	4.923	3.58E-03	50-150	50-150	50	--
PCB 55	74338-24-2	0.3	1	5.823	4.51E-04	--	--	--	--
PCB 56	41464-43-1	0.3	0.5	5.823	3.76E-04	--	--	--	--
PCB 57	74472-33-6	0.3	1	5.883	3.93E-04	--	--	--	--
PCB 58	41464-49-7	0.3	1	5.883	4.25E-04	--	--	--	--
PCB 59	74472-33-6	0.2	0.5	5.663	3.26E-04	--	--	--	--
PCB 60	33025-41-1	0.3	1	5.823	4.89E-04	--	--	--	--
PCB 61	33284-53-6	0.4	1	5.753	7.51E-04	--	--	--	--
PCB 62	54230-22-7	0.2	0.5	5.603	3.74E-04	--	--	--	--
PCB 63	74472-34-7	0.4	1	5.883	4.58E-04	--	--	--	--
PCB 64	52663-58-8	0.2	0.5	5.663	3.80E-04	--	--	--	--
PCB 65	33284-54-7	0.5	1	5.573	1.27E-03	--	--	--	--
PCB 66	32598-10-0	0.4	1	5.913	4.89E-04	--	--	--	--
PCB 67	73575-53-8	0.4	1	5.913	4.58E-04	--	--	--	--
PCB 68	73575-52-7	0.4	1	5.973	3.99E-04	--	--	--	--
PCB 69	60233-24-1	0.3	1	5.753	4.86E-04	--	--	--	--
PCB 70	32598-11-1	0.4	1	5.913	5.19E-04	--	--	--	--
PCB 71	41464-46-4	0.3	1	5.693	6.08E-04	--	--	--	--
PCB 72	41464-42-0	0.4	1	5.973	4.26E-04	--	--	--	--

Table 5-2. Data Quality Goals for PCB Congeners in Polyethylene

Analyte	CAS No.	Reporting Limits in Polyethylene		Method Detection Limit (MDL) in Porewater		LCS	MS/MSD	Precision	Labeled Compound
		MDL	MRL	Log K_{pe-w}	C_w (ng/L)	% Recovery	% Recovery	Relative Percent Difference	% Recovery
		(ng/g PE)							
PCB 73	74338-23-1	0.2	1	5.753	3.97E-04	--	--	--	--
PCB 74	32690-93-0	0.4	1	5.913	5.19E-04	--	--	--	--
PCB 75	32598-12-2	0.2	0.5	5.763	2.59E-04	--	--	--	--
PCB 76	70362-48-0	0.4	1	5.843	6.10E-04	--	--	--	--
PCB 77	32598-13-3	0.4	1	6.073	3.59E-04	50-150	50-150	50	--
PCB 78	70362-49-1	0.4	1	6.063	3.68E-04	--	--	--	--
PCB 79	41464-48-6	0.4	1	6.133	3.13E-04	--	--	--	--
PCB 80	33284-52-5	0.5	1	6.193	2.89E-04	--	--	--	--
PCB 81	70362-50-4	0.5	1	6.073	3.80E-04	50-150	50-150	50	--
PCB 82	52663-62-4	0.3	1	5.913	3.97E-04	--	--	--	--
PCB 83	60145-20-2	0.6	1	5.973	5.85E-04	--	--	--	--
PCB 84	52663-60-2	0.3	1	5.753	5.30E-04	--	--	--	--
PCB 85	65510-45-4	0.3	0.5	6.013	2.43E-04	--	--	--	--
PCB 86	55312-69-1	0.4	1	5.943	4.28E-04	--	--	--	--
PCB 87	38380-02-8	0.4	1	6.003	3.72E-04	--	--	--	--
PCB 88	55215-17-3	0.3	1	5.783	4.94E-04	--	--	--	--
PCB 89	73575-57-2	0.5	1	5.783	7.83E-04	--	--	--	--
PCB 90	68194-07-0	0.6	3	6.073	5.07E-04	--	--	--	--
PCB 91	68194-05-8	0.3	1	5.843	4.31E-04	--	--	--	--
PCB 92	52663-61-3	0.3	1	6.063	2.59E-04	--	--	--	--
PCB 93	73575-56-1	0.6	1	5.753	9.71E-04	--	--	--	--
PCB 94	73575-55-0	0.3	1	5.843	4.31E-04	--	--	--	--
PCB 95	38379-99-6	0.6	1	5.843	7.90E-04	--	--	--	--
PCB 96	73575-54-9	0.5	1	5.423	1.98E-03	--	--	--	--

Table 5-2. Data Quality Goals for PCB Congeners in Polyethylene

Analyte	CAS No.	Reporting Limits in Polyethylene		Method Detection Limit (MDL) in Porewater		LCS	MS/MSD	Precision	Labeled Compound
		MDL	MRL	Log K_{pe-w}	C_w (ng/L)	% Recovery	% Recovery	Relative Percent Difference	% Recovery
		(ng/g PE)							
PCB 97	41464-51-1	0.4	1	6.003	3.72E-04	--	--	--	--
PCB 98	60233-25-2	0.6	1	5.843	7.90E-04	--	--	--	--
PCB 99	38380-01-7	0.6	1	6.103	4.34E-04	--	--	--	--
PCB 100	39485-83-1	0.6	1	5.943	6.27E-04	--	--	--	--
PCB 101	37680-73-2	0.6	3	6.093	4.84E-04	--	--	--	--
PCB 102	68194-06-9	0.6	1	5.873	7.37E-04	--	--	--	--
PCB 103	60145-21-3	0.6	1	5.933	6.71E-04	--	--	--	--
PCB 104	56558-16-8	0.6	1	5.523	1.72E-03	50-150	50-150	50	--
PCB 105	32598-14-4	0.3	0.5	6.363	1.19E-04	50-150	50-150	50	--
PCB 106	70424-69-0	0.4	1	6.353	1.55E-04	--	--	--	--
PCB 107	70424-68-9	0.3	0.5	6.423	9.44E-05	--	--	--	--
PCB 108	70362-41-3	0.7	3	6.423	2.55E-04	--	--	--	--
PCB 109	74472-35-8	0.4	1	6.193	2.40E-04	--	--	--	--
PCB 110	38380-03-9	0.6	3	6.193	3.85E-04	--	--	--	--
PCB 111	39635-32-0	0.6	3	6.473	2.02E-04	--	--	--	--
PCB 112	74472-36-9	0.6	3	6.163	4.29E-04	--	--	--	--
PCB 113	68194-10-5	0.6	3	6.253	3.35E-04	--	--	--	--
PCB 114	74472-37-0	0.3	1	6.363	1.30E-04	50-150	50-150	50	--
PCB 115	74472-38-1	0.6	3	6.203	3.76E-04	--	--	--	--
PCB 116	18259-05-7	0.3	0.5	6.043	2.26E-04	--	--	--	--
PCB 117	68194-11-6	0.3	0.5	6.173	1.68E-04	--	--	--	--
PCB 118	31508-00-6	0.5	1	6.453	1.67E-04	50-150	50-150	50	--
PCB 119	56558-17-9	0.4	1	6.293	1.91E-04	--	--	--	--
PCB 120	68194-12-7	0.4	1	6.503	1.18E-04	--	--	--	--

Table 5-2. Data Quality Goals for PCB Congeners in Polyethylene

Analyte	CAS No.	Reporting Limits in Polyethylene		Method Detection Limit (MDL) in Porewater		LCS	MS/MSD	Precision	Labeled Compound
		MDL	MRL	Log K_{pe-w}	C_w (ng/L)	% Recovery	% Recovery	Relative Percent Difference	% Recovery
		(ng/g PE)							
PCB 121	56558-18-0	0.5	1	6.353	2.33E-04	--	--	--	--
PCB 122	76842-07-4	0.3	1	6.353	1.33E-04	--	--	--	--
PCB 123	65510-44-3	0.4	1	6.453	1.32E-04	50-150	50-150	50	--
PCB 124	70424-70-3	0.7	3	6.443	2.43E-04	--	--	--	--
PCB 125	74472-39-2	0.4	1	6.223	2.24E-04	--	--	--	--
PCB 126	57465-28-8	0.4	1	6.603	8.73E-05	50-150	50-150	50	--
PCB 127	39635-33-1	0.7	3	6.663	1.52E-04	--	--	--	--
PCB 128	38380-07-3	0.3	1	6.453	1.06E-04	--	--	--	--
PCB 129	55215-18-4	0.5	1	6.443	1.89E-04	--	--	--	--
PCB 130	52663-66-8	0.4	1	6.513	1.07E-04	--	--	--	--
PCB 131	61798-70-7	0.3	1	6.293	1.53E-04	--	--	--	--
PCB 132	38380-05-1	0.3	1	6.293	1.53E-04	--	--	--	--
PCB 133	35694-04-3	0.4	1	6.573	1.14E-04	--	--	--	--
PCB 134	52704-70-8	0.3	1	6.263	1.77E-04	--	--	--	--
PCB 135	52744-13-5	0.3	1	6.353	1.22E-04	--	--	--	--
PCB 136	38411-22-2	0.2	0.5	5.933	2.63E-04	--	--	--	--
PCB 137	35694-06-5	0.8	3	6.543	2.15E-04	--	--	--	--
PCB 138	35065-28-2	0.5	1	6.543	1.50E-04	--	--	--	--
PCB 139	56030-56-9	0.5	1	6.383	2.07E-04	--	--	--	--
PCB 140	59291-64-4	0.5	1	6.383	2.07E-04	--	--	--	--
PCB 141	52712-04-6	0.2	0.5	6.533	6.59E-05	--	--	--	--
PCB 142	41411-61-4	0.8	3	6.223	4.64E-04	--	--	--	--
PCB 143	68194-15-0	0.3	1	6.313	1.58E-04	--	--	--	--
PCB 144	68194-14-9	0.4	1	6.383	1.76E-04	--	--	--	--

Table 5-2. Data Quality Goals for PCB Congeners in Polyethylene

Analyte	CAS No.	Reporting Limits in Polyethylene		Method Detection Limit (MDL) in Porewater		LCS	MS/MSD	Precision	Labeled Compound
		MDL	MRL	Log K_{pe-w}	C_w (ng/L)	% Recovery	% Recovery	Relative Percent Difference	% Recovery
		(ng/g PE)							
PCB 145	74472-40-5	0.8	3	5.963	8.71E-04	--	--	--	--
PCB 146	51908-16-8	0.5	1	6.603	1.12E-04	--	--	--	--
PCB 147	68194-13-8	0.5	1	6.353	2.00E-04	--	--	--	--
PCB 148	74472-41-6	0.8	3	6.443	2.88E-04	--	--	--	--
PCB 149	38380-04-0	0.5	1	6.383	1.86E-04	--	--	--	--
PCB 150	68194-08-1	0.8	3	6.033	7.65E-04	--	--	--	--
PCB 151	52663-63-5	0.3	1	6.353	1.22E-04	--	--	--	--
PCB 152	68194-09-2	0.6	3	5.933	7.00E-04	--	--	--	--
PCB 153	35065-27-1	0.3	1	6.633	7.57E-05	--	--	--	--
PCB 154	60145-22-4	0.3	1	6.473	9.25E-05	--	--	--	--
PCB 155	33979-03-2	0.9	3	6.123	6.40E-04	50-150	50-150	50	--
PCB 156	38380-08-4	0.3	1	6.893	4.16E-05	50-150	50-150	50	--
PCB 157	69782-90-7	0.3	1	6.893	4.16E-05	50-150	50-150	50	--
PCB 158	74472-42-7	0.3	0.5	6.733	4.62E-05	--	--	--	--
PCB 159	39635-35-3	0.9	3	6.953	9.75E-05	--	--	--	--
PCB 160	41411-62-5	0.5	1	6.643	1.19E-04	--	--	--	--
PCB 161	74472-43-8	0.9	3	6.793	1.41E-04	--	--	--	--
PCB 162	39635-34-2	0.9	3	6.953	9.75E-05	--	--	--	--
PCB 163	74472-44-9	0.5	1	6.703	1.04E-04	--	--	--	--
PCB 164	74472-45-0	0.4	3	6.733	6.47E-05	--	--	--	--
PCB 165	74472-46-1	0.9	3	6.763	1.55E-04	--	--	--	--
PCB 166	41411-63-6	0.3	1	6.643	6.83E-05	--	--	--	--
PCB 167	52663-72-6	0.3	1	6.983	2.86E-05	50-150	50-150	50	--
PCB 168	59291-65-5	0.3	1	6.823	4.89E-05	--	--	--	--

Table 5-2. Data Quality Goals for PCB Congeners in Polyethylene

Analyte	CAS No.	Reporting Limits in Polyethylene		Method Detection Limit (MDL) in Porewater		LCS	MS/MSD	Precision	Labeled Compound
		MDL	MRL	Log K_{pe-w}	C_w (ng/L)	% Recovery	% Recovery	Relative Percent Difference	% Recovery
		(ng/g PE)							
PCB 169	32774-16-6	0.4	1	7.133	2.94E-05	50-150	50-150	50	--
PCB 170	35065-30-6	0.4	1	6.983	4.16E-05	--	--	--	--
PCB 171	52663-71-5	0.9	3	6.823	1.39E-04	--	--	--	--
PCB 172	52663-74-8	1.0	3	7.043	8.60E-05	--	--	--	--
PCB 173	68194-16-1	0.9	3	6.733	1.71E-04	--	--	--	--
PCB 174	38411-25-5	0.5	1	6.823	7.14E-05	--	--	--	--
PCB 175	40186-70-7	1	3	6.883	1.24E-04	--	--	--	--
PCB 176	52663-65-7	1	3	6.473	3.28E-04	--	--	--	--
PCB 177	52663-70-4	0.4	1	6.793	5.64E-05	--	--	--	--
PCB 178	52663-67-9	0.6	1	6.853	7.72E-05	--	--	--	--
PCB 179	52663-64-6	0.6	1	6.443	2.07E-04	--	--	--	--
PCB 180	35065-29-3	0.4	1	7.073	2.96E-05	--	--	--	--
PCB 181	74472-47-2	1	3	6.823	1.50E-04	--	--	--	--
PCB 182	60145-23-5	1	3	6.913	1.22E-04	--	--	--	--
PCB 183	52663-69-1	1	3	6.913	1.22E-04	--	--	--	--
PCB 184	74472-48-3	1	3	6.563	2.74E-04	--	--	--	--
PCB 185	52712-05-7	1	3	6.823	1.50E-04	--	--	--	--
PCB 186	74472-49-4	1	3	6.403	4.05E-04	--	--	--	--
PCB 187	52663-68-0	0.5	1	6.883	6.22E-05	--	--	--	--
PCB 188	74487-85-7	0.6	1	6.533	1.69E-04	50-150	50-150	50	--
PCB 189	39635-31-9	0.5	1	7.423	1.70E-05	50-150	50-150	50	--
PCB 190	41411-64-7	0.6	1	7.173	3.86E-05	--	--	--	--
PCB 191	74472-50-7	1	3	7.263	5.73E-05	--	--	--	--
PCB 192	74472-51-8	1	3	7.233	6.14E-05	--	--	--	--

Table 5-2. Data Quality Goals for PCB Congeners in Polyethylene

Analyte	CAS No.	Reporting Limits in Polyethylene		Method Detection Limit (MDL) in Porewater		LCS	MS/MSD	Precision	Labeled Compound
		MDL	MRL	Log K_{pe-w}	C_w (ng/L)	% Recovery	% Recovery	Relative Percent Difference	% Recovery
		(ng/g PE)							
PCB 193	69782-91-8	0.4	1	7.233	2.05E-05	--	--	--	--
PCB 194	35694-08-7	0.4	1	7.513	1.30E-05	--	--	--	--
PCB 195	52663-78-2	1	3	7.273	5.73E-05	--	--	--	--
PCB 196	42740-50-1	1	3	7.363	4.66E-05	--	--	--	--
PCB 197	33091-17-7	0.6	3	7.013	6.07E-05	--	--	--	--
PCB 198	68194-17-2	0.5	1	7.333	2.32E-05	--	--	--	--
PCB 199	52663-75-9	0.5	1	6.913	6.11E-05	--	--	--	--
PCB 200	52663-73-7	0.6	3	6.983	6.50E-05	--	--	--	--
PCB 201	40186-71-8	1	3	7.333	5.11E-05	--	--	--	--
PCB 202	2136-99-4	1	3	6.953	1.23E-04	50-150	50-150	50	--
PCB 203	52663-76-0	1	3	7.363	4.77E-05	--	--	--	--
PCB 204	74472-52-9	1	3	7.013	1.09E-04	--	--	--	--
PCB 205	74472-53-0	1	3	7.713	2.18E-05	50-150	50-150	50	--
PCB 206	40186-72-9	1	3	7.803	1.77E-05	50-150	50-150	50	--
PCB 207	52663-79-3	1	3	7.453	3.96E-05	--	--	--	--
PCB 208	52663-77-1	1	3	7.423	4.34E-05	50-150	50-150	50	--
PCB 209	2051-24-3	0.4	1	7.893	4.80E-06	50-150	50-150	50	--
PCB 1L	---	---	---			15-140	15-140	--	15-150
PCB 3L	---	---	---			15-140	15-140	--	15-150
PCB 4L	---	---	---			30-140	30-140	--	25-150
PCB 15L	---	---	---			30-140	30-140	--	25-150
PCB 19L	---	---	---			30-140	30-140	--	25-150
PCB 37L	---	---	---			30-140	30-140	--	25-150
PCB 54L	---	---	---			30-140	30-140	--	25-150
PCB 77L	---	---	---			30-140	30-140	--	25-150
PCB 81L	---	---	---			30-140	30-140	--	25-150

Table 5-2. Data Quality Goals for PCB Congeners in Polyethylene

Analyte	CAS No.	Reporting Limits in Polyethylene		Method Detection Limit (MDL) in Porewater		LCS	MS/MSD	Precision	Labeled Compound
		MDL	MRL	Log K_{pe-w}	C_w (ng/L)	% Recovery	% Recovery	Relative Percent Difference	% Recovery
		(ng/g PE)							
PCB 104L	---	---	---			30-140	30-140	--	25-150
PCB 105L	---	---	---			30-140	30-140	--	25-150
PCB 114L	---	---	---			30-140	30-140	--	25-150
PCB 118L	---	---	---			30-140	30-140	--	25-150
PCB 123L	---	---	---			30-140	30-140	--	25-150
PCB 126L	---	---	---			30-140	30-140	--	25-150
PCB 155L	---	---	---			30-140	30-140	--	25-150
PCB 156L	---	---	---			30-140	30-140	--	25-150
PCB 157L	---	---	---			30-140	30-140	--	25-150
PCB 167L	---	---	---			30-140	30-140	--	25-150
PCB 169L	---	---	---			30-140	30-140	--	25-150
PCB 188L	---	---	---			30-140	30-140	--	25-150
PCB 189L	---	---	---			30-140	30-140	--	25-150
PCB 202L	---	---	---			30-140	30-140	--	25-150
PCB 205L	---	---	---			30-140	30-140	--	25-150
PCB 206L	---	---	---			30-140	30-140	--	25-150
PCB 208L	---	---	---			30-140	30-140	--	25-150
PCB 209L	---	---	---			30-140	30-140	--	25-150
PCB 28L	---	---	---			40-125	40-125	--	30-135
PCB 111L	---	---	---			40-125	40-125	--	30-135
PCB 178L	---	---	---			40-125	40-125	--	30-135

Acronyms:

Cw = Concentration in Water
L = Labeled
LCS = Laboratory Control Sample
Log Kpe-w = polyethylene-water partitioning coeff
MDL = Method Detection Limit
MRL = Method Reporting Limit
MS/MSD = Martix Spike/Matrix Spike Duplicate
PCB = Polychlorinated biphenyl
PE = polyethylene

Table 5-3. Sample Preparation and Analysis Method Summary for Sediment Samples

Analyte	Preparation Method	Procedure	Analytical Method	Procedure
Total Solids	SW160.3	Oven dry	SW160.3	Gravimetric
Total Organic Carbon	Plumb 1981	Acid pretreatment	Plumb *et al* ., 1981	Combustion; colormetric titration
Black Carbons	Gustafsson 1997	Dry & Grind to 60 mesh	Gustafsson 1997	Combustion; IR
Grain Size	PSEP	Oven dry	PSEP 1986	Sieves and pipette
PCB Aroclors	SW3546	Microwave extraction	SW8082A	GC/ECD
	SW3665A	Sulfuric acid cleanup		
	SW3630C	Silica gel cleanup		
	SW3660B	Sulfur cleanup		
PCB Congeners	SW3541	Automated Soxhlet Extraction	EPA1668C	HRGC/HRMS
	SW3640A	Gel permeation chromatography		

Notes:

ALS SOP No. GEN- GEN- BLACK C SOOT PREP, September 20, 2010

EPA 1668C - Method 1668C: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, USEPA, April 2010.

GC/ECD - Gas chromatography/electron capture detector

HRGC - High resolution gas chromatography

HRMS - High resolution mass spectrometer

PSEP 1986 - Puget Sound Estuary Program (PSEP) Recommended Protocols for Measuring Conventional Sediment Variables in Puget Sound, Puget Sound Water Quality Authority, March 1986.

SIM - Selective ion monitoring

SW Methods - *USEPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition* , 1998.

Table 5-4. Parameters Used to Evaluate Data Quality

Data Quality Indicators	QC Parameters
Precision	RPD values of:
	(1) LCS/LCS Duplicate
	(2) MS/MSD
	(3) Field Duplicates
Accuracy/Bias	Percent Recovery (%R) or Percent Difference (%D) values of:
	(1) Initial Calibration and Calibration Verification
	(2) LCS
	(3) MS
	(4) Surrogate Spikes
	Results of:
	(1) Instrument and Calibration Blank
	(2) Method (Preparation) Blank
	(3) Trip Blank
	(4) Equipment Rinsate Blank
Representativeness	Results of All Blanks
	Sample Integrity (CoC and Sample Receipt Forms)
	Holding Times
Comparability	Sample-specific reporting limits
	Sample Collection Methods
	Laboratory Analytical Methods
Completeness	Data qualifiers
	Laboratory deliverables
	Requested/Reported valid results
Sensitivity	MDLs and MRLs

Notes:
LCS – Laboratory Control Sample
MS/MSD - Matrix spike/matrix spike duplicate

Table 5-5. Laboratory Method Detections, Method Reporting Limits for Sediment Analyses

Analytes	CAS #	Lab Limits	
		MDLs	MRLs
Conventional Inorganic Parameters (mg/kg)			
Total Solids	--	--	0.10%
Total Organic Carbon	--	0.02%	0.05%
Black Carbon	--	0.01%	0.01%
Grain Size	--	0.10%	0.10%
PCBs Aroclors (µg/kg)			
Aroclor 1016	12674-11-2	2.1	10
Aroclor 1221	11104-28-2	2.1	10
Aroclor 1232	11141-16-5	2.1	10
Aroclor 1242	53469-21-9	2.1	10
Aroclor 1248	12672-29-6	2.1	10
Aroclor 1254	11097-69-1	2.1	10
Aroclor 1260	11096-82-5	2.1	10
Aroclor 1262	37324-23-5	2.1	10
Aroclor 1268	11100-14-4	2.1	10

Notes:

MDL = method detection limit

mg/kg=milligram per kilogram

MRL = method reporting limit

µg/kg=microgram per kilogram.

MRLs and MDLs are based on 2011 performance audits conducted by ALS.

PCB = Polychlorinated biphenyl

Table 5-6. Accuracy and Precision Control Criteria for Chemicals in Sediments

Analyte	Analytical Method	Surrogate Spike Accuracy [1] (% Rec.) [2]	LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD or %RSD)
Conventional Inorganic Parameters					
Solids, Total	SW160.3	NA	NA	NA	≤20
Total Volatile Solids	PSEP	NA	NA	NA	≤20
Total Organic Carbon	PSEP	NA	80-120	75-125	%RSD≤20
Grain Size	PSEP	NA	NA	NA	%RSD≤20
Black Carbon	Gustafsson	NA	80-120	75-125	≤20
PCB Aroclors					
Aroclor 1016	SW8082	NA	53-100	53-100	≤40
Aroclor 1260	SW8082	NA	58-112	58-112	≤40
Decachlorobiphenyl	SW8082	35-119	NA	NA	NA
Tetrachloro-m-xylene	SW8082	33-143	NA	NA	NA

Notes:

1. Listed surrogate spike, precision, and accuracy control limits based on in-house performance statistics of ALS Inc. The values are subject to change as the laboratory is updating the control limits per EPA requirements.
2. % Rec. = Percent recovery

LCS = Laboratory Control Sample

PCB = Polychlorinated biphenyl

PCB = Polychlorinated biphenyl

Table 5-7. Data Quality Goals for PCB Congeners in Sediments

Analyte	CAS No.	MDL	MRL	LCS	MS/MSD	Precision	Labeled Compound
		(ng/Kg)		% Recovery	% Recovery	Relative % Difference	% Recovery
PCB 1	2051-60-7	8	20	50-150	50-150	50	--
PCB 2	2051-61-8	0.4	1	--	--	--	--
PCB 3	2051-62-9	9	20	50-150	50-150	50	--
PCB 4	13029-08-8	17	50	50-150	50-150	50	--
PCB 5	16605-91-7	1	5	--	--	--	--
PCB 6	25569-80-6	1	5	--	--	--	--
PCB 7	33284-50-3	2	5	--	--	--	--
PCB 8	34883-43-7	12	50	--	--	--	--
PCB 9	34883-39-1	2	5	--	--	--	--
PCB 10	33146-45-1	2	5	--	--	--	--
PCB 11	2050-67-1	10	100	--	--	--	--
PCB 12	2974-92-7	3	10	--	--	--	--
PCB 13	2974-90-5	3	10	--	--	--	--
PCB 14	34883-41-5	3	10	--	--	--	--
PCB 15	2050-68-2	18	50	50-150	50-150	50	--
PCB 16	38444-78-9	4	10	--	--	--	--
PCB 17	37680-66-3	9	20	--	--	--	--
PCB 18	37680-65-2	20	50	--	--	--	--
PCB 19	38444-73-4	4	10	50-150	50-150	50	--
PCB 20	38444-84-7	19	50	--	--	--	--
PCB 21	55702-46-0	5	20	--	--	--	--
PCB 22	38444-85-8	9	20	--	--	--	--
PCB 23	55720-44-0	5	20	--	--	--	--
PCB 24	55702-45-9	5	20	--	--	--	--
PCB 25	55712-37-3	5	20	--	--	--	--
PCB 26	38444-81-4	8	20	--	--	--	--
PCB 27	38444-76-7	6	20	--	--	--	--
PCB 28	7012-37-5	19	50	--	--	--	--
PCB 29	15862-07-4	8	20	--	--	--	--
PCB 30	35693-92-6	20	50	--	--	--	--
PCB 31	16606-02-3	15	50	--	--	--	--

Table 5-7. Data Quality Goals for PCB Congeners in Sediments

Analyte	CAS No.	MDL	MRL	LCS	MS/MSD	Precision	Labeled Compound
		(ng/Kg)		% Recovery	% Recovery	Relative % Difference	% Recovery
PCB 32	38444-77-8	8	20	--	--	--	--
PCB 33	38444-86-9	5	20	--	--	--	--
PCB 34	37680-68-5	7	20	--	--	--	--
PCB 35	37680-69-6	8	20	--	--	--	--
PCB 36	38444-87-0	8	20	--	--	--	--
PCB 37	38444-90-5	13	50	50-150	50-150	50	--
PCB 38	53555-66-1	8	20	--	--	--	--
PCB 39	38444-88-1	9	20	--	--	--	--
PCB 40	38444-93-8	12	50	--	--	--	--
PCB 41	52663-59-9	12	50	--	--	--	--
PCB 42	36559-22-5	6	20	--	--	--	--
PCB 43	70362-46-8	9	50	--	--	--	--
PCB 44	41464-39-5	19	50	--	--	--	--
PCB 45	70362-45-7	5	20	--	--	--	--
PCB 46	41464-47-5	10	20	--	--	--	--
PCB 47	2437-79-8	19	50	--	--	--	--
PCB 48	70362-47-9	8	20	--	--	--	--
PCB 49	41464-40-8	11	50	--	--	--	--
PCB 50	62796-65-0	6	20	--	--	--	--
PCB 51	68194-04-7	5	20	--	--	--	--
PCB 52	35693-99-3	19	50	--	--	--	--
PCB 53	41464-41-9	6	20	--	--	--	--
PCB 54	15968-05-5	12	50	50-150	50-150	50	--
PCB 55	74338-24-2	12	50	--	--	--	--
PCB 56	41464-43-1	10	20	--	--	--	--
PCB 57	74472-33-6	12	50	--	--	--	--
PCB 58	41464-49-7	13	50	--	--	--	--
PCB 59	74472-33-6	6	20	--	--	--	--
PCB 60	33025-41-1	13	50	--	--	--	--
PCB 61	33284-53-6	17	50	--	--	--	--
PCB 62	54230-22-7	6	20	--	--	--	--

Table 5-7. Data Quality Goals for PCB Congeners in Sediments

Analyte	CAS No.	MDL	MRL	LCS	MS/MSD	Precision	Labeled Compound
		(ng/Kg)		% Recovery	% Recovery	Relative % Difference	% Recovery
PCB 63	74472-34-7	14	50	--	--	--	--
PCB 64	52663-58-8	7	20	--	--	--	--
PCB 65	33284-54-7	19	50	--	--	--	--
PCB 66	32598-10-0	16	50	--	--	--	--
PCB 67	73575-53-8	15	50	--	--	--	--
PCB 68	73575-52-7	15	50	--	--	--	--
PCB 69	60233-24-1	11	50	--	--	--	--
PCB 70	32598-11-1	17	50	--	--	--	--
PCB 71	41464-46-4	12	50	--	--	--	--
PCB 72	41464-42-0	16	50	--	--	--	--
PCB 73	74338-23-1	9	50	--	--	--	--
PCB 74	32690-93-0	17	50	--	--	--	--
PCB 75	32598-12-2	6	20	--	--	--	--
PCB 76	70362-48-0	17	50	--	--	--	--
PCB 77	32598-13-3	17	50	50-150	50-150	50	--
PCB 78	70362-49-1	17	50	--	--	--	--
PCB 79	41464-48-6	17	50	--	--	--	--
PCB 80	33284-52-5	18	50	--	--	--	--
PCB 81	70362-50-4	18	50	50-150	50-150	50	--
PCB 82	52663-62-4	13	50	--	--	--	--
PCB 83	60145-20-2	22	50	--	--	--	--
PCB 84	52663-60-2	12	50	--	--	--	--
PCB 85	65510-45-4	10	20	--	--	--	--
PCB 86	55312-69-1	15	50	--	--	--	--
PCB 87	38380-02-8	15	50	--	--	--	--
PCB 88	55215-17-3	12	50	--	--	--	--
PCB 89	73575-57-2	19	50	--	--	--	--
PCB 90	68194-07-0	24	100	--	--	--	--
PCB 91	68194-05-8	12	50	--	--	--	--
PCB 92	52663-61-3	12	50	--	--	--	--
PCB 93	73575-56-1	22	50	--	--	--	--

Table 5-7. Data Quality Goals for PCB Congeners in Sediments

Analyte	CAS No.	MDL	MRL	LCS	MS/MSD	Precision	Labeled Compound
		(ng/Kg)		% Recovery	% Recovery	Relative % Difference	% Recovery
PCB 94	73575-55-0	12	50	--	--	--	--
PCB 95	38379-99-6	22	50	--	--	--	--
PCB 96	73575-54-9	21	50	--	--	--	--
PCB 97	41464-51-1	15	50	--	--	--	--
PCB 98	60233-25-2	22	50	--	--	--	--
PCB 99	38380-01-7	22	50	--	--	--	--
PCB 100	39485-83-1	22	50	--	--	--	--
PCB 101	37680-73-2	24	100	--	--	--	--
PCB 102	68194-06-9	22	50	--	--	--	--
PCB 103	60145-21-3	23	50	--	--	--	--
PCB 104	56558-16-8	23	50	50-150	50-150	50	--
PCB 105	32598-14-4	11	20	50-150	50-150	50	--
PCB 106	70424-69-0	14	50	--	--	--	--
PCB 107	70424-68-9	10	20	--	--	--	--
PCB 108	70362-41-3	27	100	--	--	--	--
PCB 109	74472-35-8	15	50	--	--	--	--
PCB 110	38380-03-9	24	100	--	--	--	--
PCB 111	39635-32-0	24	100	--	--	--	--
PCB 112	74472-36-9	25	100	--	--	--	--
PCB 113	68194-10-5	24	100	--	--	--	--
PCB 114	74472-37-0	12	50	50-150	50-150	50	--
PCB 115	74472-38-1	24	100	--	--	--	--
PCB 116	18259-05-7	10	20	--	--	--	--
PCB 117	68194-11-6	10	20	--	--	--	--
PCB 118	31508-00-6	19	50	50-150	50-150	50	--
PCB 119	56558-17-9	15	50	--	--	--	--
PCB 120	68194-12-7	15	50	--	--	--	--
PCB 121	56558-18-0	21	50	--	--	--	--
PCB 122	76842-07-4	12	50	--	--	--	--
PCB 123	65510-44-3	15	50	50-150	50-150	50	--
PCB 124	70424-70-3	27	100	--	--	--	--

Table 5-7. Data Quality Goals for PCB Congeners in Sediments

Analyte	CAS No.	MDL	MRL	LCS	MS/MSD	Precision	Labeled Compound
		(ng/Kg)		% Recovery	% Recovery	Relative % Difference	% Recovery
PCB 125	74472-39-2	15	50	--	--	--	--
PCB 126	57465-28-8	14	50	50-150	50-150	50	--
PCB 127	39635-33-1	28	100	--	--	--	--
PCB 128	38380-07-3	12	50	--	--	--	--
PCB 129	55215-18-4	21	50	--	--	--	--
PCB 130	52663-66-8	14	50	--	--	--	--
PCB 131	61798-70-7	12	50	--	--	--	--
PCB 132	38380-05-1	12	50	--	--	--	--
PCB 133	35694-04-3	17	50	--	--	--	--
PCB 134	52704-70-8	13	50	--	--	--	--
PCB 135	52744-13-5	11	50	--	--	--	--
PCB 136	38411-22-2	9	20	--	--	--	--
PCB 137	35694-06-5	30	100	--	--	--	--
PCB 138	35065-28-2	21	50	--	--	--	--
PCB 139	56030-56-9	20	50	--	--	--	--
PCB 140	59291-64-4	20	50	--	--	--	--
PCB 141	52712-04-6	9	20	--	--	--	--
PCB 142	41411-61-4	31	100	--	--	--	--
PCB 143	68194-15-0	13	50	--	--	--	--
PCB 144	68194-14-9	17	50	--	--	--	--
PCB 145	74472-40-5	32	100	--	--	--	--
PCB 146	51908-16-8	18	50	--	--	--	--
PCB 147	68194-13-8	18	50	--	--	--	--
PCB 148	74472-41-6	32	100	--	--	--	--
PCB 149	38380-04-0	18	50	--	--	--	--
PCB 150	68194-08-1	33	100	--	--	--	--
PCB 151	52663-63-5	11	50	--	--	--	--
PCB 152	68194-09-2	24	100	--	--	--	--
PCB 153	35065-27-1	13	50	--	--	--	--
PCB 154	60145-22-4	11	50	--	--	--	--
PCB 155	33979-03-2	34	100	50-150	50-150	50	--

Table 5-7. Data Quality Goals for PCB Congeners in Sediments

Analyte	CAS No.	MDL	MRL	LCS	MS/MSD	Precision	Labeled Compound
		(ng/Kg)		% Recovery	% Recovery	Relative % Difference	% Recovery
PCB 156	38380-08-4	13	50	50-150	50-150	50	--
PCB 157	69782-90-7	13	50	50-150	50-150	50	--
PCB 158	74472-42-7	10	20	--	--	--	--
PCB 159	39635-35-3	35	100	--	--	--	--
PCB 160	41411-62-5	21	50	--	--	--	--
PCB 161	74472-43-8	35	100	--	--	--	--
PCB 162	39635-34-2	35	100	--	--	--	--
PCB 163	74472-44-9	21	50	--	--	--	--
PCB 164	74472-45-0	14	100	--	--	--	--
PCB 165	74472-46-1	36	100	--	--	--	--
PCB 166	41411-63-6	12	50	--	--	--	--
PCB 167	52663-72-6	11	50	50-150	50-150	50	--
PCB 168	59291-65-5	13	50	--	--	--	--
PCB 169	32774-16-6	16	50	50-150	50-150	50	--
PCB 170	35065-30-6	16	50	--	--	--	--
PCB 171	52663-71-5	37	100	--	--	--	--
PCB 172	52663-74-8	38	100	--	--	--	--
PCB 173	68194-16-1	37	100	--	--	--	--
PCB 174	38411-25-5	19	50	--	--	--	--
PCB 175	40186-70-7	38	100	--	--	--	--
PCB 176	52663-65-7	39	100	--	--	--	--
PCB 177	52663-70-4	14	50	--	--	--	--
PCB 178	52663-67-9	22	50	--	--	--	--
PCB 179	52663-64-6	23	50	--	--	--	--
PCB 180	35065-29-3	14	50	--	--	--	--
PCB 181	74472-47-2	40	100	--	--	--	--
PCB 182	60145-23-5	40	100	--	--	--	--
PCB 183	52663-69-1	40	100	--	--	--	--
PCB 184	74472-48-3	40	100	--	--	--	--
PCB 185	52712-05-7	40	100	--	--	--	--
PCB 186	74472-49-4	41	100	--	--	--	--

Table 5-7. Data Quality Goals for PCB Congeners in Sediments

Analyte	CAS No.	MDL	MRL	LCS	MS/MSD	Precision	Labeled Compound
		(ng/Kg)		% Recovery	% Recovery	Relative % Difference	% Recovery
PCB 187	52663-68-0	19	50	--	--	--	--
PCB 188	74487-85-7	23	50	50-150	50-150	50	--
PCB 189	39635-31-9	18	50	50-150	50-150	50	--
PCB 190	41411-64-7	23	50	--	--	--	--
PCB 191	74472-50-7	42	100	--	--	--	--
PCB 192	74472-51-8	42	100	--	--	--	--
PCB 193	69782-91-8	14	50	--	--	--	--
PCB 194	35694-08-7	17	50	--	--	--	--
PCB 195	52663-78-2	43	100	--	--	--	--
PCB 196	42740-50-1	43	100	--	--	--	--
PCB 197	33091-17-7	25	100	--	--	--	--
PCB 198	68194-17-2	20	50	--	--	--	--
PCB 199	52663-75-9	20	50	--	--	--	--
PCB 200	52663-73-7	25	100	--	--	--	--
PCB 201	40186-71-8	44	100	--	--	--	--
PCB 202	2136-99-4	44	100	50-150	50-150	50	--
PCB 203	52663-76-0	44	100	--	--	--	--
PCB 204	74472-52-9	45	100	--	--	--	--
PCB 205	74472-53-0	45	100	50-150	50-150	50	--
PCB 206	40186-72-9	45	100	50-150	50-150	50	--
PCB 207	52663-79-3	45	100	--	--	--	--
PCB 208	52663-77-1	46	100	50-150	50-150	50	--
PCB 209	2051-24-3	15	50	50-150	50-150	50	--
PCB 1L	--	--	--	15-140	15-140	--	15-150
PCB 3L	--	--	--	15-140	15-140	--	15-150
PCB 4L	--	--	--	30-140	30-140	--	25-150
PCB 15L	--	--	--	30-140	30-140	--	25-150
PCB 19L	--	--	--	30-140	30-140	--	25-150
PCB 37L	--	--	--	30-140	30-140	--	25-150
PCB 54L	--	--	--	30-140	30-140	--	25-150
PCB 77L	--	--	--	30-140	30-140	--	25-150

Table 5-7. Data Quality Goals for PCB Congeners in Sediments

Analyte	CAS No.	MDL	MRL	LCS	MS/MSD	Precision	Labeled Compound
		(ng/Kg)		% Recovery	% Recovery	Relative % Difference	% Recovery
PCB 81L	--	--	--	30-140	30-140	--	25-150
PCB 104L	--	--	--	30-140	30-140	--	25-150
PCB 105L	--	--	--	30-140	30-140	--	25-150
PCB 114L	--	--	--	30-140	30-140	--	25-150
PCB 118L	--	--	--	30-140	30-140	--	25-150
PCB 123L	--	--	--	30-140	30-140	--	25-150
PCB 126L	--	--	--	30-140	30-140	--	25-150
PCB 155L	--	--	--	30-140	30-140	--	25-150
PCB 156L	--	--	--	30-140	30-140	--	25-150
PCB 157L	--	--	--	30-140	30-140	--	25-150
PCB 167L	--	--	--	30-140	30-140	--	25-150
PCB 169L	--	--	--	30-140	30-140	--	25-150
PCB 188L	--	--	--	30-140	30-140	--	25-150
PCB 189L	--	--	--	30-140	30-140	--	25-150
PCB 202L	--	--	--	30-140	30-140	--	25-150
PCB 205L	--	--	--	30-140	30-140	--	25-150
PCB 206L	--	--	--	30-140	30-140	--	25-150
PCB 208L	--	--	--	30-140	30-140	--	25-150
PCB 209L	--	--	--	30-140	30-140	--	25-150
PCB 28L	--	--	--	40-125	40-125	--	30-135
PCB 111L	--	--	--	40-125	40-125	--	30-135
PCB 178L	--	--	--	40-125	40-125	--	30-135

Acronyms:

L = Labeled
LCS = Laboratory Control Sample
MDL = Method Detection Limit
MRL = Method Reporting Limit
MS/MSD = Martix Spike/Matrix Spike Duplicate
ng/Kg = nanogram/kilogram
PCB = Polychlorinated biphenyl

Table 7-1. Schedule for Project Deliverables

Activity	Work Product	Schedule
Porewater Sampling and Analysis Plan	Draft Porewater SAP	March 4, 2014
	Final Porewater SAP	Due to EPA Team: May 23, 2014
Porewater SAP HASP	Draft HASP	June 22, 2014 - 6 weeks ahead of expected passive sampler placement
	Final HASP	Due to EPA Team: 30 days after receipt of EPA Team comments on the draft
Passive Sampler Placement	None	Targeted for early August 2014[1]
Passive Sampler Retrieval	None	Targeted for mid-October[1]
Porewater Field Sampling and Data Report	Draft Porewater Report	Due to EPA Team: 90 days after PED Retrieval
	Final Porewater Report	Due to EPA Team: 30 days after receipt of EPA Team comments on the draft
Implementability Study	Draft Implementability Report	Due to EPA Team: 60 days after submission of the Draft Porewater Report to EPA
	Final Implementability Report	Due to EPA Team: 30 days after receipt of EPA Team comments on the draft
Recontamination Evaluation	Draft Recontamination Assessment Report	Due to EPA Team: 60 days after submission of the Draft Porewater Report to EPA
	Final Recontamination Assessment Report	Due to EPA Team: 30 days after receipt of EPA Team comments on the draft

Notes:

1. Passive sampler placement will target a period when the Willamette River stage is at its lowest and is currently scheduled to occur in early August 2014. Passive sampler retrieval will occur approximately 60 days after placement. The target passive sampler placement date is targeted for late August 2014 through mid-October but the actual installation and retrieval schedule is contingent upon vessel traffic considerations at the Glacier NW and Cargill Docks.

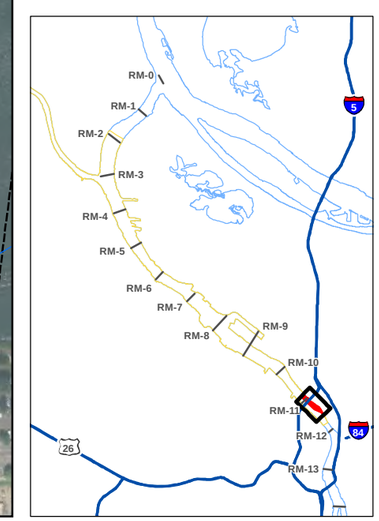
FIGURE 1-1
Project Area Map

River Mile 11 East Porewater Sampling and Analysis Plan

Willamette River
Portland, Oregon

LEGEND

- RM11E Project Area (dashed line indicates inferred top of bank)
- Shoreline Properties
- U.S. Army Corps of Engineers Navigation Channel
- River Mile (RM) Tenth



MAP NOTES:
Date: February 27, 2014
RM = River Mile
1. The locations of all features shown are approximate.
2. RM11E Project Area includes AOPC 25 and the adjacent riverbank area to the top of bank.
3. Air photo taken fall 2012 by METRO.

N

0 150 300
Feet

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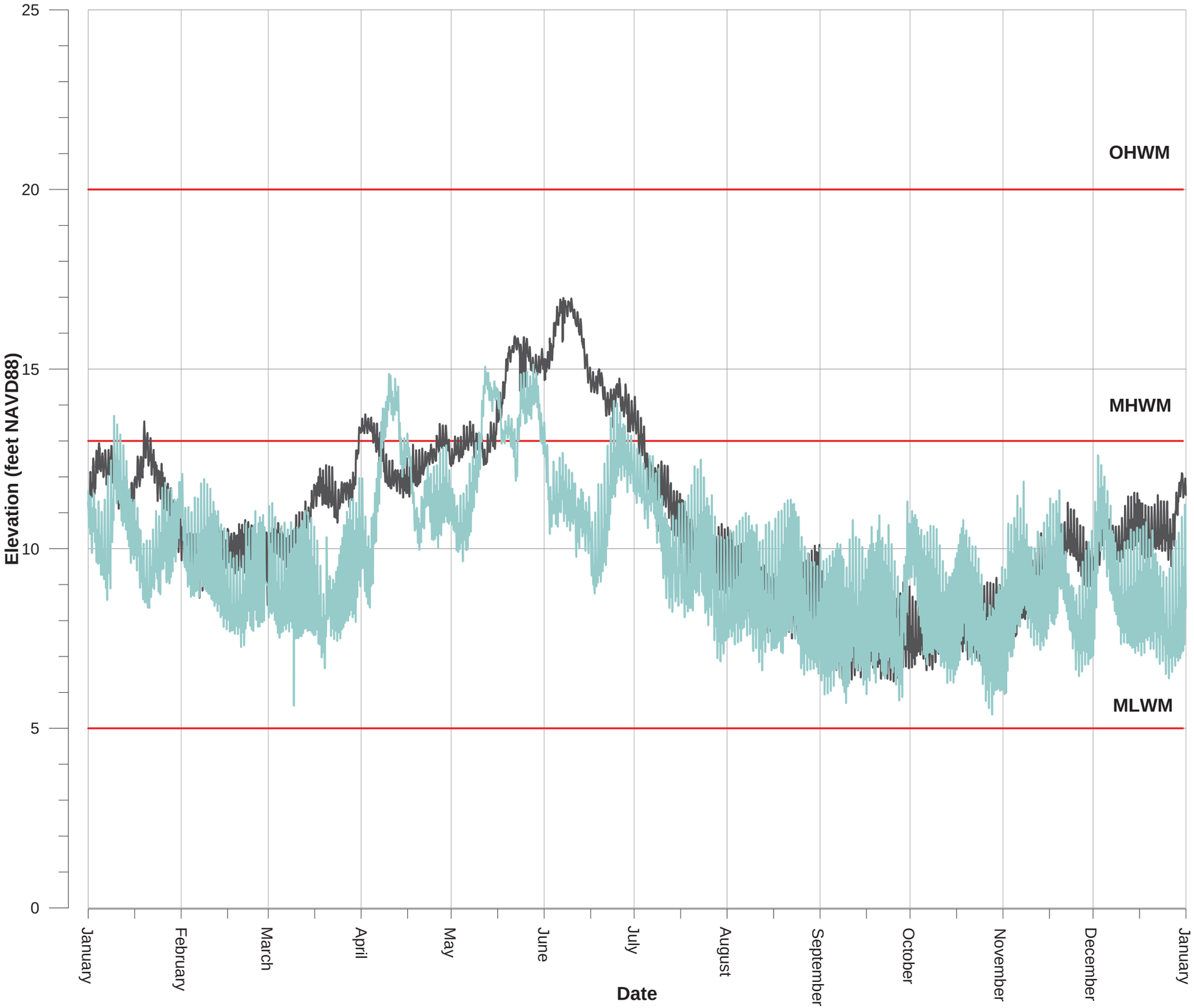

Figure 2-1
Willamette River Stage

Porewater Sampling Analysis Plan
River Mile 11 East
Portland, OR

LEGEND

- 2013 Willamette River Stage
- Average Willamette River Stage (2007-2012)
- Portland Harbor Water Level Datums

Notes:

OHWM = Ordinary High Water Mark, refers to upper edge of the riverbank and defines the elevation beyond which inundation by the river is limited to extreme flow events, which occur approximately every 5 years.

MHWM = Mean High Water Mark, refers to the elevation defining the shoreline boundary of the Portland Harbor Superfund Site and is based on DEQ memorandum dated July 9, 2003 to EPA regarding the upland/in-water boundary for the Superfund Site (DEQ 2003b).

MLWM = Mean Low Water Mark, refers to the average approximate average low water height.

Sampling Period = approximate time of the year to collect groundwater samples during a lower river stage height.

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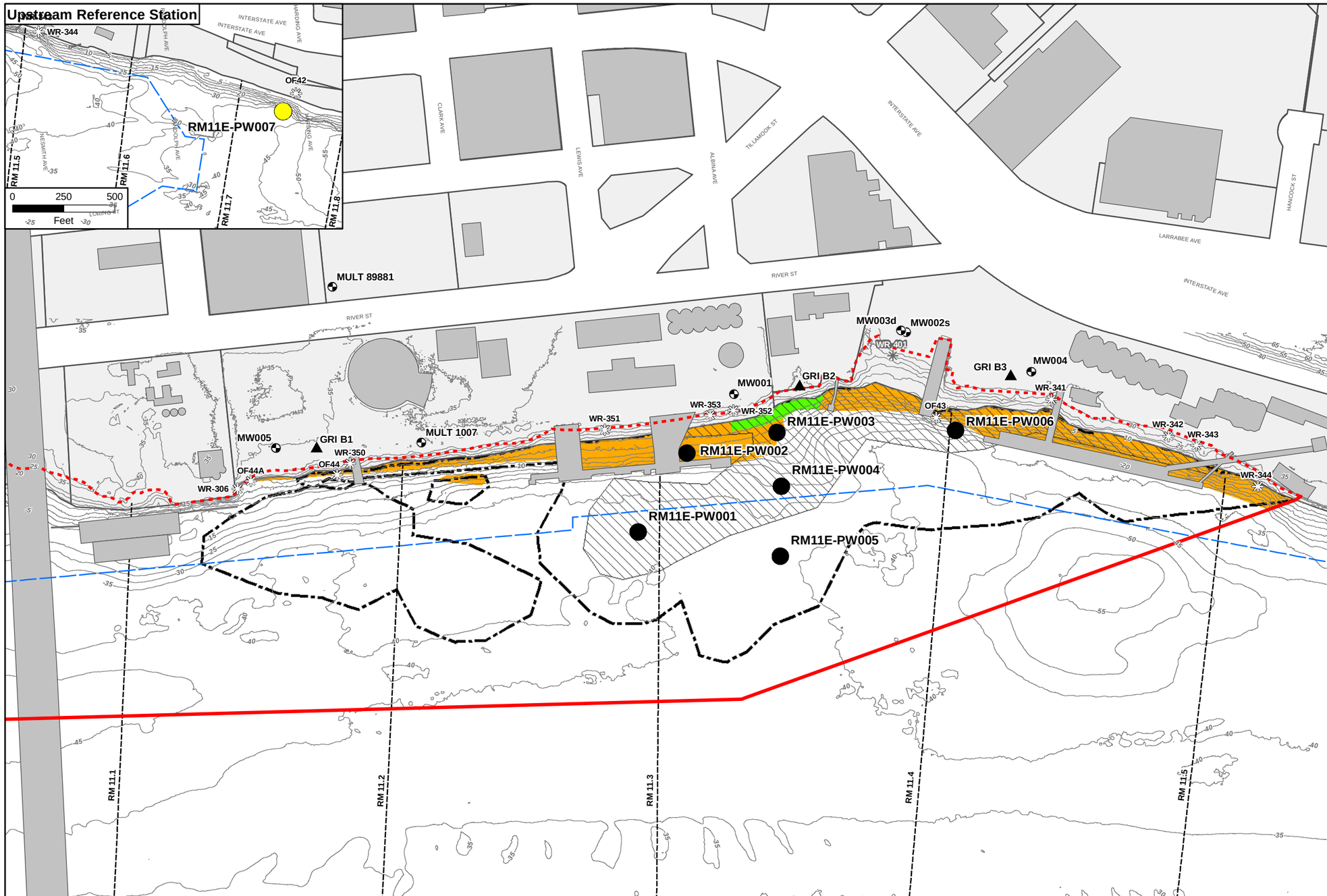

FIGURE 2-2
Proposed Porewater Sampling Locations

River Mile 11 East Porewater Sampling and Analysis Plan

Willamette River
Portland, Oregon

LEGEND

Proposed Porewater Sample Locations

- Porewater Sample
- Upstream Reference Station

All Other Features

- Engineered Cap Zone
- In-Situ Treatment Area
- FS 75 ug/kg RAL boundary
- Comprehensive Benthic Risk Area
- Geotechnical Boring
- Monitoring Well
- RM11E Project Area (dashed line indicates inferred top of bank)
- U.S. Army Corps of Engineers Navigation Channel
- Building/Structure
- Tax Lot
- Active Outfall
- Inactive Outfall
- 5 foot Bathymetric Contour
- River Mile (RM) Tenth

MAP NOTES:

Date: February 26, 2014
FS = Feasibility Study
RAL = Remedial Action Level
RM = River Mile
1. The locations of all features shown are approximate.
2. The comprehensive benthic risk areas and FS RAL footprint is consistent with the information presented in the Draft FS report for the Portland Harbor (Anchor QEA et al., 2012).
3. Engineered Cap Zone and In-Situ Treatment Areas digitized from Figure 2.6-2K of the FS.

0 100 200
Feet

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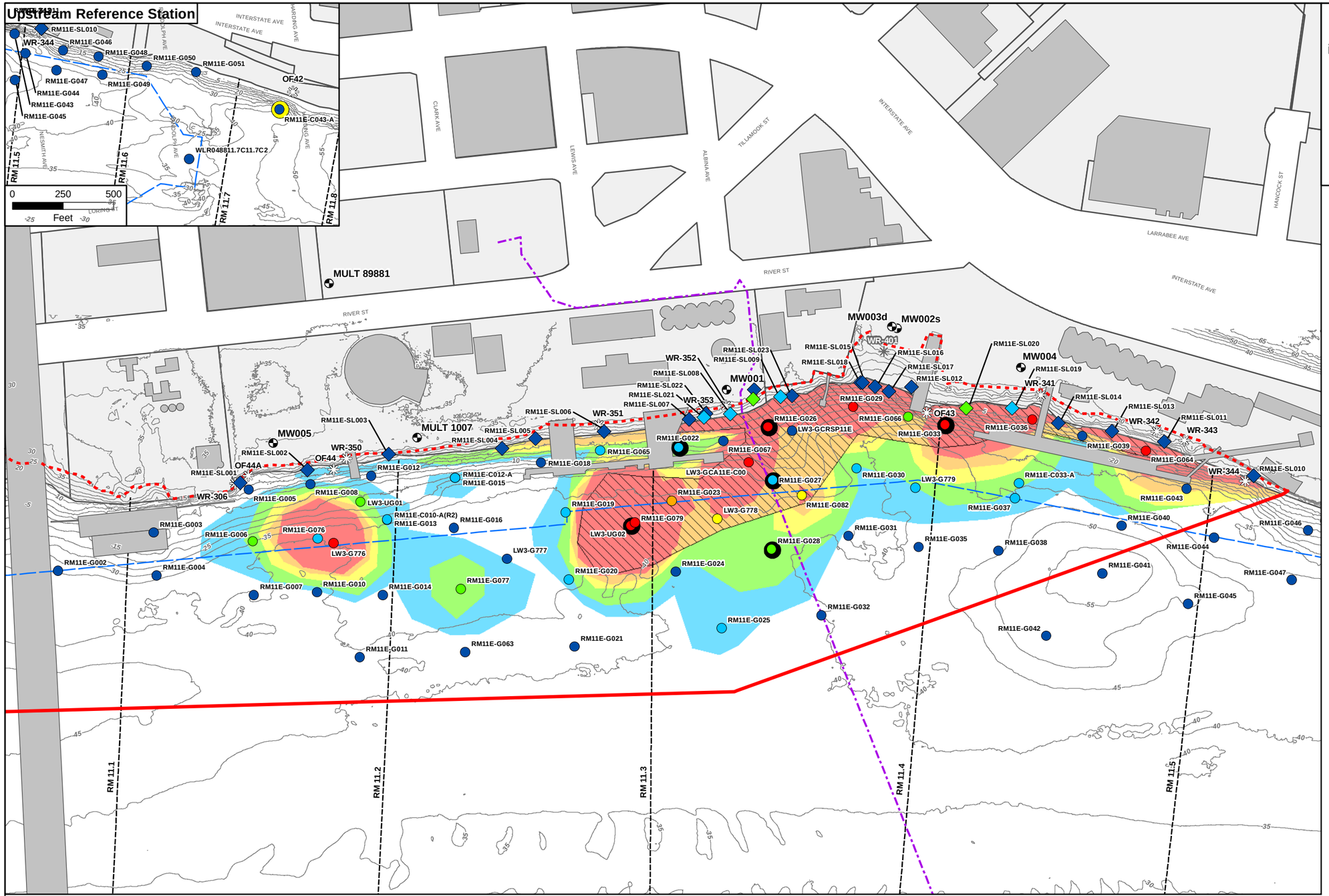

FIGURE 2-3
Total PCB Concentrations in Bank Soils and Surface Sediment

River Mile 11 East Porewater Sampling and Analysis Plan

Willamette River
Portland, Oregon

LEGEND

- Bank Soil Sample Location
- Surface Sediment Sample Location
- Proposed Porewater Sample Location
- Proposed Upstream Reference Station Location

Total PCBs in Surface Sediment (FS RAL Footprints)

- 75 - 200 ug/kg
- >200 - 500 ug/kg
- >500 - 750 ug/kg
- >750 - 1,000 ug/kg
- >1,000 ug/kg
- Comprehensive Benthic Risk Area

All Other Features

- Monitoring Well
- RM11E Project Area (dashed line indicates inferred top of bank)
- U.S. Army Corps of Engineers Navigation Channel
- Building/Structure
- Tax Lot
- Active Outfall
- Inactive Outfall
- Cable Crossing (approximate)
- 5 foot Bathymetric Contour
- River Mile (RM) Tenth

MAP NOTES:

Date: February 27, 2014
FS = Feasibility Study
PCB = Polychlorinated Biphenyl
RAL = Remedial Action Level
RM = River Mile

1. The locations of all features shown are approximate.
2. The comprehensive benthic risk areas are consistent with the information presented in the Draft FS report for the Portland Harbor (Anchor QEA et al., 2012).
3. Source of existing sediment sample locations is LWG SCRA Database (Integral et al., 2011) and RM11E Field and Data Reports (GSI, 2010).
4. Total PCBs were calculated using the Portland Harbor Remedial Investigation (RI) data rules and calculated totals are the sum of all detected concentrations; non-detected concentrations are treated as zero.
5. The breakdown of total PCB concentrations is consistent with each of the remedial alternatives (B through F) presented in the Draft FS report for the Portland Harbor (Anchor QEA et al., 2012).
6.Two multi-point composite samples, collected from the upper and lower riverbank in the southwest corner of the Glacier NW property are included in this figure and the data are presented in Glacier NW's Riverbank Soil Source Control Screening Evaluation (ERM, 2013).
7. New surface sediment data (RM11E-G076, -G077, -G079) collected as part of the RM11E Supplemental RI/FS are included in this figure.

Total PCBs in Bank Soils and Surface Sediment

- <75 ug/kg
- >75 - 200 ug/kg
- >200 - 500 ug/kg
- >500 - 750 ug/kg
- >750 - 1,000 ug/kg
- >1,000 ug/kg

N

0 100 200
Feet

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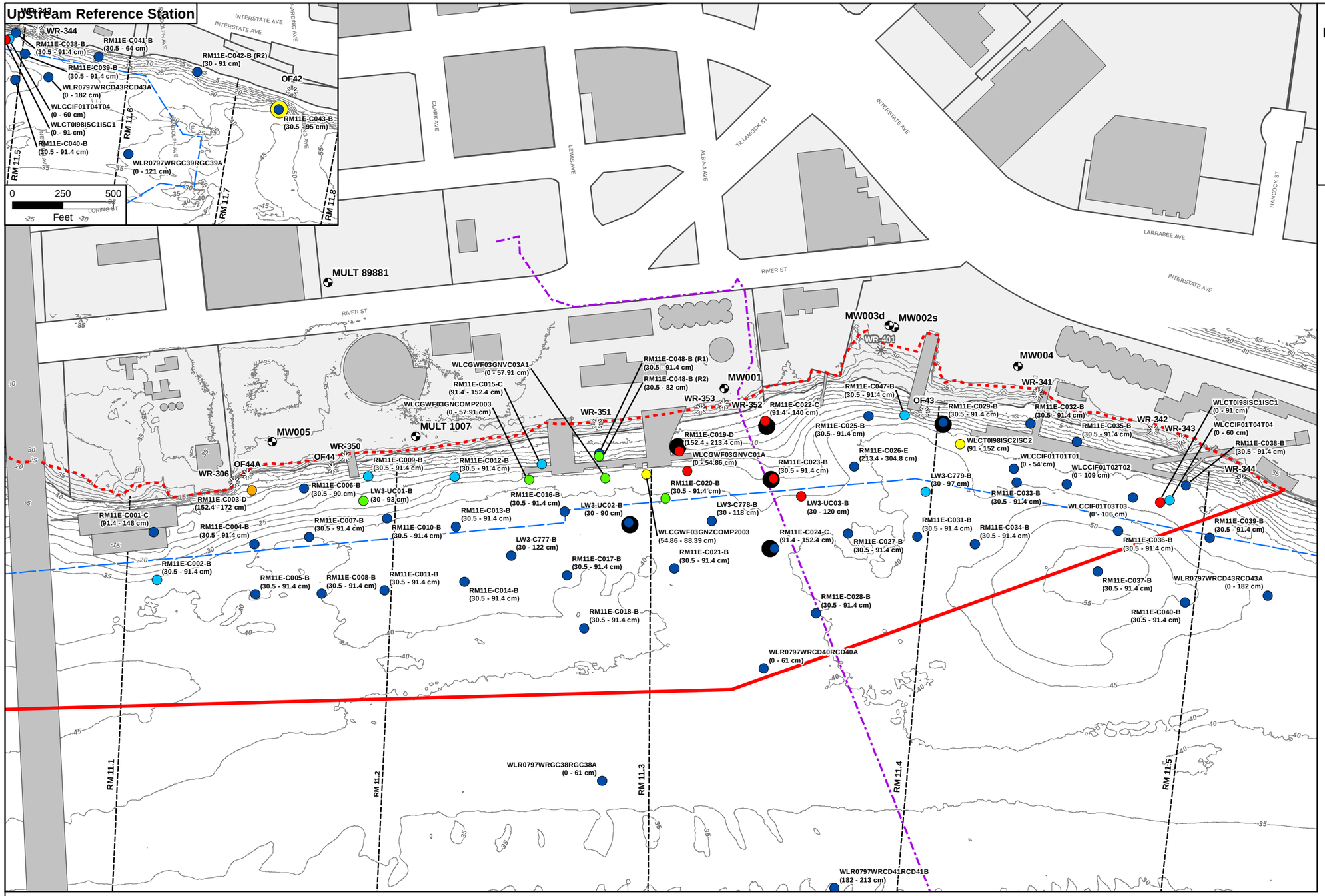

FIGURE 2-4
Maximum Total PCB Concentrations in Subsurface Sediment

River Mile 11 East Porewater Sampling and Analysis Plan

Willamette River
Portland, Oregon

LEGEND

- Subsurface Sediment Sample Location
- Proposed Porewater Sample Location
- Proposed Upstream Reference Station Location

All Other Features

- Monitoring Well
- RM11E Project Area (dashed line indicates inferred top of bank)
- U.S. Army Corps of Engineers Navigation Channel
- Building/Structure
- Tax Lot
- Active Outfall
- Inactive Outfall
- Cable Crossing (approximate)
- 5 foot Bathymetric Contour
- River Mile (RM) Tenth

MAP NOTES:

Date: February 27, 2014
FS = Feasibility Study
PCB = Polychlorinated Biphenyl
RM = River Mile
1. The locations of all features shown are approximate.
2. The proposed porewater sampling station is co-located with previously collected surface sediment grab sampling locations, which differs slightly from the paired subsurface sediment core locations.
3. Source of existing sediment sample locations is LWG SCRA Database (Integral et al., 2011) and RM11E Field and Data Reports (GSI, 2010).
4. Total PCBs were calculated using the Portland Harbor Remedial Investigation (RI) data rules and calculated totals are the sum of all detected concentrations; non-detected concentrations are treated as zero.
5. The breakdown of total PCB concentrations is consistent with each of the remedial alternatives (B through F) presented in the Draft FS report for the Portland Harbor (Anchor QEA et al., 2012).

Maximum Total PCBs in Subsurface Sediment

- <75 ug/kg
- >75 - 200 ug/kg
- >200 - 500 ug/kg
- >500 - 750 ug/kg
- >750 - 1,000 ug/kg
- >1,000 ug/kg

N

0 100 200
Feet

GSI Water Solutions, Inc.

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Attachment 1

RM11E Field Forms

FIELD CHANGE REQUEST (FCR) FORM

Project Name: River Mile 11 East, Supplemental RI/FS Investigation

FCR Number: _____ **FCR Date:_____**

Prepared for: Sean Sheldrake, EPA

Summary:

_____

_____

Description of Proposed Modification:

_____

_____

Reasons for Modification:

_____

_____

_____

_____

_____

Schedule Impacts:

_____

_____

Submitted by:

Project Manager: _____ Date:_____

Approved by:

EPA Representative: _____ Date:_____

Sediment Core Drive Log

Job:

Job No:

Field Reps:

Contractor:

Core Location:

Date: Time:

Attempt #: Accept/Reject

Sample Method:

Proposed Coordinates	Actual Coordinates
N: E:	N: E:
Mudline:	Mudline:
Core Drive:	Core Drive: Core Recovery:

DTS Boat:

DTS Lead Line:

Mudline Elevation:

Tide Measurements (Datum:)

Time/Height:

Time/Height:

Description:

(free fall, fingers inverted, vibration needed to drive/extract, estimation of density, debris encountered, slopes, refusal, mudline conditions, drive action, etc.)

Measurement (to nearest 0.1 foot):

Core Tube Length:

Avg. % Recovery:

Avg. % Compaction:

Section:	Length:	Description at Cuts:
A =		
B =		
C =		
D =		

Total Drive: Length Recovered:

Notes:

f:/fieldforms/sedimentcoredrivelog

CORE LOG

Project: RM11E Porewater Sampling	Station ID:	Location: Portland, OR	Page ____ of ____

Latitude:	Longitude:	Name of Driller:	Drilling Firm:
Time:	Date:	Core Type :	Core Size: 4" OD Aluminum; 3.75" ID
Mudline:	Elevation Datum:	Penetration:	Acquisition:
Tide Time/Height	Tide Time/Height	Percent Recovery:	Accept/Reject: Accept Reject
Date Logged:	Time Logged:	Name of Core Logger:	

Depth (unit)	Sampling					USCS group name, color, grain size range, minor constituents, plasticity, odor, sheen, moisture content, texture, wethering, cementation, geologic interpretation, etc
	USCS	Munsel Color	PID	Sample Depth	Sample Number	

Chain of Custody Record & Laboratory Analysis Request

Science and Engineering for the Environment LLC
4401 Latona NE
Seattle, WA 98105
206-418-6173

Lab Assigned Number:	Turn-around Requested: Normal	Date:
Client Company:	Phone:	Page: of
Client Contact: Tim Thompson 206-418-6173		No. of Coolers: Cooler Temps:
Client Project Name:		Analysis Requested
Client Project #:	Samplers: SEE, GIS	

Sample ID	Date	Time	Matrix	No. Containers	Grain Size	TOC	Total Solids	PCB Aroclors	PCB Congeners	Black Carbon	Chem Archive	MS/MSD								Notes/Comments

Comments/Special Instructions	Relinqushed by: (Signature)	Received by: (Signature)	Relinquished by: (Signature)		Received by: (Signature)
	Printed Name: Tim Thompson	Printed Name:	Printed Name:		Printed Name:
	Company: SEE	Company:	Company:		Company:
	Date & Time:	Date & Time:	Date & Time:		Date & Time:

Attachment 2

Inadvertent Discovery Plan

Attachment 2

Plan and Procedures for the Inadvertent Discovery of Cultural Resources and Human Skeletal Remains

RM11E Project Area, Portland, Oregon

The RM11E Participation Group plans to conduct surface sediment sampling concurrent with porewater sampling efforts as part of the Supplemental Remedial Investigation and Feasibility Study (RI/FS). This work is being conducted pursuant to the Statement of Work (SOW) contained within the Administrative Settlement Agreement and Order on Consent (Settlement Agreement) (U.S. Environmental Protection Agency [EPA] Region 10, CERCLA Docket No. 10-2013-0087). These investigations are supplementary to the RI/FS for the Portland Harbor Superfund Site and are targeted to facilitate selection and design of a final remedy at the RM11E Project Area.

The following Inadvertent Discovery Plan (IDP) outlines procedures to follow, in accordance with state and federal laws, if archaeological materials or human remains are discovered.

Introduction

Cultural resources are extremely important to our Tribal nations and to our state's sense of identity and history. Tribal cultural resources can include ceremonial artifacts and objects at burial sites. The locations of burials and their associated human remains are also of great importance to the traditions and identity of Tribes. Properties that contain cultural resources are of critical significance to Tribal nations. Therefore, it is extremely important that identification and protection of cultural resources be considered carefully in planning for any ground-disturbing activities at a site.

The *Cultural Resource Monitoring River Mile 11 East Focused Sediment Characterization, Willamette River, Portland, Multnomah County, Oregon* identified a moderate to high likelihood that archaeologically sensitive artifacts exist onsite (SWCA, 2010). Previous RM11E sediment investigations found numerous historic debris, which consisted of a mix of fragments including brick, nails, unknown metal, glass, ceramics, wood and plastic as well as recent anthropogenic debris mixed in with river sediments of sand, gravel, and silt. The debris appeared to be the result of a large amount of land-derived debris and fill pushed into the Willamette River channel during shoreline demolition events and historic structures at sites. No prehistoric archaeological materials or evidence of intact subsurface prehistoric cultural deposits were observed during the course of previous work.

This document provides guidelines should someone discover a cultural or archaeological resource onsite. It is important that workers onsite comply with applicable state and federal laws protecting these cultural resources.

Recognizing Cultural Resources

A cultural resource discovery could be prehistoric or historic. Examples include:

- An accumulation of shell, burned rocks, or other food related materials,
- Bones or small pieces of bone,
- An area of charcoal or very dark stained soil with artifacts,
- Stone tools or waste flakes (i.e. an arrowhead, or stone chips),
- Clusters of tin cans or bottles, logging or agricultural equipment that appear to be older than 50 years,
- Buried railroad tracks, decking, or other industrial materials.

When in doubt, assume the material is a cultural resource.

Inadvertent Discovery Plan Procedures

Due to the potential to encounter archaeologically sensitive artifacts in the RM11E Project Area, David Ellis of Willamette Cultural Resources Associates, LTD. (Willamette CRA) and his team will be retained to oversee potential cultural resources found on the Site during surface sediment sampling activities. Mr. Ellis has provided training to GSI field staff to define what kind of artifacts and deposits require examination and documentation by an archaeologist. A Willamette CRA archaeologist was present during the initial Supplemental RI/FS sampling conducted in November 2013 to inspect surface sediment grab samples. No sensitive prehistoric or historic artifacts were found during this sampling or previous RM11E sediment sampling conducted in this area. This archaeologist will be on-call during logging and processing of diver-collected surface sediment samples at the laboratory.

If archaeologically sensitive prehistoric or historic artifacts are discovered when the archaeologist is not on site (i.e., on-call), the following steps will be taken:

STEP 1: STOP WORK. If any ALS, SEE, or GSI employee, contractor or subcontractor believes that he or she has uncovered a cultural resource that requires examination by an archaeologist at any point in the project, processing of the sample in question must stop.

STEP 2: NOTIFY MONITOR. The Field Director will notify the project archeologist to examine the material in question. If the archeologist determines that the artifact(s) and/or deposits require formal documentation, they will follow the IDP procedures listed below and inform ALS, SEE or GSI staff when/if sampling activities may resume.

STEP 3: NOTIFY GSI PROJECT MANAGEMENT. If the project archeologist initiates the IDP procedures listed below, the Field Director should notify the senior Project Manager who will subsequently inform the RM11E Participation Group of the status of such activities. If deemed appropriate by the project archeologist and the Project Manager, sample processing activities may recommence on other samples while awaiting the results of the applicable IDP consultations.

The following presents specific IDP procedures for the RM11E site.

- If any previously unidentified archaeological materials are encountered, processing of the sample in question shall stop immediately, and the Field Director will notify the project archaeologist. The project archeologist will follow State Historic Preservation Office (SHPO) guidelines for known sites and isolated finds (ORS 358.905-385.955 or sites along scenic waterways (ORS 390.805-390.925).
- If isolated artifacts (designated by SHPO as less than 10 artifacts of non-diagnostic quality) are recovered, sample processing may continue.
- If human remains or funerary objects are encountered the Oregon State Police, SHPO, and the appropriate tribes will be notified in accordance with Oregon state laws and regulations (ORS 97.740-97.760; State Executive Order Number 96-30). The Multnomah County Medical Examiner will also be notified if human remains are encountered.
    - If human remains are encountered, they should be treated with dignity and respect at all times. Cover the remains with a tarp or other materials (not soil or rocks) for temporary protection in place and to shield them from being photographed. Do not call 911 or speak with the media.
- If faunal (bone) material is observed in a sample and is clearly not fish or bird bone, the project archeologist immediately will contact a supervisory archaeologist with osteological training to determine if the bone is human or animal. Work will be halted until the osteological determination has been made.

Documentation of Archaeological Materials

All artifacts collected from samples will be analyzed, catalogued and temporarily curated. Ultimate disposition of cultural materials will be determined in consultation with SHPO, or any other applicable tribes and agencies.

All archaeologically sensitive prehistoric or historic cultural material discovered during project activities will be recorded by a professional archaeologist on a form using standard techniques. Site overviews, features, and artifacts will be photographed; stratigraphic profiles and soil/sediment descriptions will be prepared for subsurface exposures. Discovery locations will be documented on scaled site plans and site location maps.

Archaeological discoveries will be documented and reported to SHPO, other potential federal agencies, and any potentially affected tribe(s). If no cultural resources are encountered, the

project archeologist will sign a compliance certification and summarize all observations for inclusion in the Field Sampling and Data Report.

Finds of incidental debris that do not warrant formal documentation will be provided to Willamette CRA for review.

References

SWCA Environmental Consultants, 2010. *Cultural Resource Monitoring Rive mile 11 East Focused Sediment Characterization Willamette River, Portland, Multnomah County, Oregon.* April 2010. Prepared for GSI Water Solutions, Inc.

Attachment 3

SOP for Diver-Placement and Retrieval of Passive Samplers and Co-located Sediment Samples

Standard Operating Procedures for Diver-Placement and Retrieval of Passive Samplers and Co-located Sediment Samples

1. SCOPE AND APPLICATION

This standard operating procedures (SOP) document presents detailed descriptions of methodologies for divers to follow during the placement and retrieval of passive samplers and collection of co-located sediment samples for the River Mile 11E (RM11E) supplemental remedial investigation/feasibility study (RI/FS). The purpose of this document is to provide a clear set of protocols for the field and dive team to review and follow in order to ensure data of sufficient quality and consistency are collected to meet project objectives described in the Porewater Sampling and Analysis Plan (SAP). This SOP includes procedures for a reconnaissance survey to test and confirm the sampling methodology will be successful in the field conditions at the site.

2. METHOD SUMMARY

Porewater samplers consist of pre-fitted 10 x 50 cm polyethylene (PE) strips mounted into aluminum sampling frames. A photo showing the passive sampling device (PSD) assembly is shown in Figure 1. Divers will install the samplers at the selected sampling locations, to a target depth of 30 cm below mud line (bml), leaving 20 cm of the PE extending above the mudline in the overlying surface water. The sampler will be left to interrogate the sediment and overlying water for approximately 60 days. At the time of retrieval, the divers will first insert a 40 x 10 cm diameter core approximately 2 inches away from the PSD location, and then remove the PSDs from the sediment bed. The core will then be withdrawn with a cap placed over the bottom of the tube. The PSD, complete with the polyethylene strip still inside, will be brought to the surface, initially cleaned of mud, labeled, wrapped in aluminum foil, and stored on ice for transport to the laboratory. The collected cores will be capped and labeled in the field. The PE and collected sediment will be transported in separate ice filled coolers to ALS's laboratory in Kelso, Washington for further processing.

SEE LLC	RM11 East Project Portland, OR	Figure-1: Porewater Sampling Device. (A) Hammer assembly; (B) PE Plate Cover; (C) Depth Stop

3. PRE-DIVE BRIEFING

3-1 Review the Ballard Marine Construction (BMC) Dive, and Health and Safety Plans (HSP) with the dive team.

3-2 Review the current day activities and associated portions of the RM11E passive sampling SOP with the dive team at the start of each day's dive operations.

3-3 Inspect the sampling equipment to ensure it will function properly.

3-4 Verify that the diver has the required gear.

3-5 Verify that the underwater video equipment and communication equipment is operating.

3-6 Verify that the dGPS is operating correctly, and reporting at least three satellite signals.

3-7 Review the method to establish the sample location (below) and water depth on the river bed below the dock (such as measurement from pilings).

4. NAVIGATION

4-1 All sample locations will be established using a differential global positioning system (dGPS). Navigation and recording of site coordinates will be in Oregon State Plan North coordinates. A check of the dGPS will occur by taking a reading at either the inside corner upstream (approximately SE) of the Glacier Dock, or the inside downstream corner (approximately NE) of the Cargill Dock. The dGPS reading will be taken at the same location at the beginning and end of each sampling day, and recorded in the field log book. Navigation accuracy will be ± 3 ft. If multiple dGPS are onboard, the units will be checked against one another and have an accuracy of ± 3 ft. If the dGPS readings are not consistent, the Field Director will make a determination as to which unit will be used to locate stations. Justification for that decision will be recorded in the Field Logbook.

4-2 Target sampling stations are identified in Table 2-3 of the SAP. The boat will navigate to the fixed coordinates of each location, and lower an anchor attached to line onto the position immediately below the dGPS antenna. To the extent practical in the moving river current, the line will be drawn vertical and the position again confirmed with the dGPS. The position will be recorded in the field log book and on the PSD Log.

4-3 It is preferable that the boat remain on station using an anchor or a shoreline tie off. Live-boat operations are permitted, but only within the safety parameters defined in the BMC HSP.

4-4 The diver will enter the water and slowly proceed down the anchor line to the river bed, taking care to minimize disturbance of the sediment.

4-5 The Field Director (FD) onboard the boat will view the general sample location on video, look for areas of soft sediment and minimal debris, and coordinate the selection of the actual sampling location with the diver. Observations on the target sampling location will be recorded in the field sampling log.

4-6 If the diver is required to move the location, the line anchor will be moved to the actual sampling location, and a new coordinate fix will be taken. The sample location may be moved within 10 meters (approximately 30 feet) of the location identified in Table 1. The rationale for moving the location, along with the new coordinates is recorded in the field notebook and onto the PSD Log.

4-7 If sample locations must be moved more than 10 meters from the initial target, the FD will contact the EPA Remedial Program Manager (RPM) to discuss proposed field changes. If the RPM cannot be reached, the FD will call and/or e-mail the Project Manager for EPA's oversight contractor CDM, to discuss proposed field changes. A Field Change Request form will be submitted via e-mail as a follow up to any sampling location changes (see Attachment 1).

5. RECONNAISSANCE SURVEY PROCEDURES

5-1 The scope and objective for the reconnaissance survey is presented in Section 4.4 of the Porewater SAP. The reconnaissance survey was completed on May 2, 2014. The methods used during that survey are documented in this SOP because subsequent sections reference these procedures.

5-2 Navigation and underwater confirmation of the target location are as described in Section 4 of this SOP.

5-3 A PSD fitted with blank polyethylene (PE) sheeting will be lowered to the diver on a clipped line after confirmation of the sampling location.

5-4 The diver's helmet cam will be operational throughout dive-portion of the reconnaissance survey. The video from the procedure will become part of the record for the reconnaissance survey.

5-5 At the confirmed target location, the diver will report the general bottom conditions (e.g., sediment type, presence of debris) to the surface team. The diver will install the PSD parallel to the flow of the river, by pushing or hammering the device into the sediments up to the fixed stop legs on the sampler, which are preset to allow 30 cm of PE exposure to surface sediment and 20 cm exposure to the overlying water. The PSD will be driven at a slow, steady rate; the process of insertion should take 0.5 - 1 minute. The diver may gently rock the sample device if resistance is encountered. During the PSD placement, the diver will communicate with the boat to describe the degree of ease/difficulty associated with the push, and whether refusal is encountered prior to completion. This information is recorded into the field log notebook.

5-6 Should refusal be encountered prior to full sampler insertion, the diver will gently withdraw the sampler and inspect the PE for any rips or tears. If a rip or tear is present, a new sampler will be lowered to the diver for a second attempt. Results of the initial insertion attempt are recorded in the field log and onto the PSD Log.

5-7 In the event of refusal, the diver, in communication with the FD, will visually examine the area again in an approximately 1 m arc, targeting a location that at the surface appears to be free of debris and rubble. The diver will then repeat Steps 5-3 and 5-4 for a total of three attempts at a target location. If all three attempts are unsuccessful, this will be noted in the field log, and the reconnaissance team will proceed to the next location.

5-8 Upon successful placement, the diver will carefully remove the stainless steel covers from both sides of the PSD, and clip them to the carabineer connected to the boat line. After removal of the covers, one minute is allowed to elapse to allow for the sediment to consolidate around the device. If possible, a photograph or video will be taken. The sampling device will then be withdrawn by slowly pulling the frame vertically from the

sediment. If necessary, a gentle rocking motion may be used to assist in the release of the frame. Upon withdrawal, the diver will note any tears or rips in the PE to the surface team, and then signal the surface team to bring the PSD assembly to the surface. The diver will remain at the sampling location until confirmation by the surface team that there are no rips or tears to the PE.

5-9 Once the diver and sampling device are safely on-board, the FD will again inspect the PE for rips or tears, note those in the log book, and take a photograph of the sampler.

5-10 The reconnaissance team can then weigh anchor, and proceed to the next location and repeat Steps 5-1 through 5-8. A sampler with intact PE can be used at the next test station.

6. FIELD PLACEMENT OF PASSIVE SAMPLING DEVICES

6-1 The scope and objective for the field deployment of the passive sampling devices is presented in Section 4.5 of the Porewater SAP.

6-2 The diver's helmet cam will be operational throughout the dive-portion of the field placement. The video from the procedure will become part of the Porewater Characterization Report.

6-3 Porewater samplers fitted with PE that have been impregnated with PCB performance reference compounds (PRC) will arrive from the laboratory wrapped in aluminum foil and ready for deployment. All personnel handling the PE samplers must be attired in Level D personal protective gear and wear new nitrile gloves for each sampler at all times. Care should be taken to avoid any contact with the polyethylene film in the frame.

6-4 Navigation and underwater confirmation of the target location are as described in Section 4 of this SOP.

6-5 The FD will take the sampler and fit it into the hammer assembly shown in Figure 1. Each sampler should arrive at the station fitted with the PE, and with the stainless steel PE-protective plates in place. Should for any reason those plates come off, or need to be put onto another sampler, Figure 2 shows generally how those are placed onto the sampler. The FD is responsible for ensuring that the plate covers, and the entire passive samplers are correctly assembled and ready for deployment.

6-6 The diver will enter the water and occupy the location on the river bed indicated by the anchor line as the sampling site. Once on the anchor, the passive sampling assembly will be carefully lowered down to the diver. Until the sampler is secure in the sediment, the PE sampler will be attached via a carabineer to a weighted tag line secured to the sample boat.

6-7 At the confirmed target location, the diver will report the general bottom conditions (e.g., sediment type, presence of debris) to the surface team. The diver will insert the PSD assembly into the sediment, parallel to the flow of the river, and push or hammer the sampler into the sediments up to the fixed stop legs on the hammer assembly, which are preset to allow 30 cm of PE exposure to surface sediment and 20 cm exposure to the overlying water. The sampler should be driven at a slow, steady rate; the process of insertion should take 0.5 - 1 minute. The diver may gently rock the sample device if/when resistance is encountered. During the insertion, the diver will communicate with the boat to describe the degree of ease/difficulty associated with the push, and whether refusal is encountered prior to completion. This information is recorded into the field log notebook. After successful insertion, the diver will pull the two (2) protective plates, and pull the pin to release the hammer assembly from the sampler. It may be necessary for the diver to hold the sampler in place while removing the hammer assembly.

6-8 Should refusal be encountered prior to full sampler insertion, the PSD will be gently withdrawn and inspected to determine if the protective plates are still in place. If the plates are in place, the diver may proceed with a second attempt at sample deployment. If the plates are not in place, the diver will inspect the PE for any rips or tears. If the diver observes any rips or tears in the PE membrane, sample deployment with that device will be aborted and a new PSD lowered to the diver in order to proceed. Results of the initial insertion attempt are recorded in the field log and onto the PSD Log.

6-9 If it is necessary to locate an alternate location to place the samplers, the diver, in communication with the FD, will visually inspect the area again in an approximately 1 m arc, targeting a location that at the surface appears to be free of debris and rubble. The diver will then repeat Steps 6-6 through 6-8 for a total of three attempts at a target location. The maximum radius from the target location that can be used without confirmation with EPA is 10 m.

6-10 If all three attempts fail, and the sample locations must be moved more than 10 meters from the initial target, the FD will contact the EPA Remedial Program Manager following the procedures listed in Step 4-7.

6-11 Upon successful insertion of the sampler, the FD will record the date, the time, and the coordinates of the sample location in the field log and on the PSD Log. Individual pictures from the diver helmet cam of each location will become part of the Porewater Characterization Report.

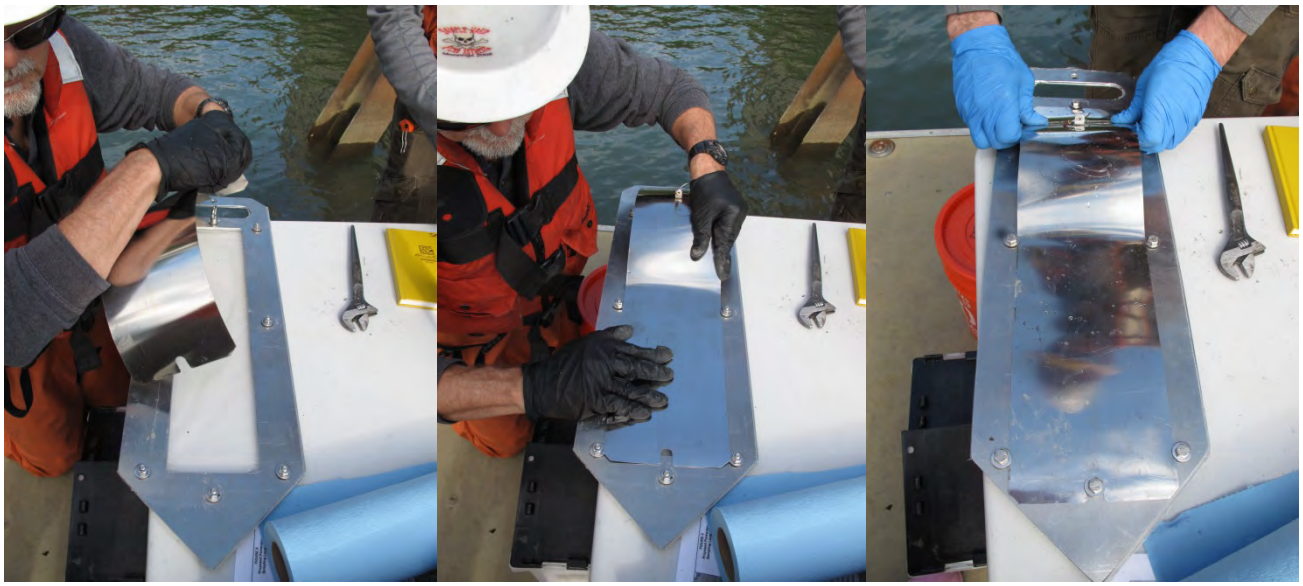
6-12 After successful deployment of the PSD, a second sampling assembly will be lowered to the diver. The second sampler will be inserted approximately 8 inches (two fist-widths) from the first sampler following the procedures in Step 6-7.

6-13 Once a successful sampler-insertion has been achieved, the weighted tag line secured to the boat will be released to the diver. Two tag lines will be attached to each sampler and

each tag line will be attached to an anchor point. At the inshore stations (PW002, PW003, and PW006), the tag lines will be secured to the nearest piling or other suitable tie-point. For the offshore stations (PW001, PW004, and PW005) the diver will secure the tag lines parallel to the flow of the river into the sediment using a shore anchor, or similar-constructed anchor. The tag lines serve two purposes: to provide anchorage to secure the sampler frame to the river bed during deployment, and to help locate the sampler again at the end of the deployment period. For all the offshore stations, a Benthos ALP365 Pinger will be attached to one of the samplers to assist in the location and retrieval of samplers. For the up-river station (PW007), the preferred method will be to secure the sampler via lines to a fixed upland or in-river structure. If a secure structure is not available, that station will also be secured with anchored tag lines.

6-14 Upon successful insertion of the sampler, the FD will record the date, time, measured water depth, any issues related to sampler placement, and sample coordinates in the field log and on the PSD Log.

6-15 Upon completion, the dive/field team may then proceed to the next sampling location.



SEE LLC	RM11 East Project Portland, OR	Figure-2: Installation of PE Plate Cover

7. RETRIEVAL OF PASSIVE SAMPLING DEVICE

7-1 The scope and objectives for the field retrieval of the passive sampling devices is presented in Section 4.5 of the Porewater SAP.

7-2 The diver's helmet cam will be operational throughout the dive-portion of the passive sampler retrieval. The video will be become part of the Porewater Characterization Report.

7-3 Porewater samplers are fitted with PE that has been impregnated with PCB PRCs. All personnel handling the PE samplers must be attired in Level D personal protective gear and wear new nitrile gloves for each sampler. Care must be taken to (1) use only one pair of gloves per passive sampling unit, (2) to minimize contact with the polyethylene strip in the sampler, and (3) to prevent any contact between the sampled sediment and the polyethylene sheets.

7-4 The procedures for navigation and target station location are as described in Section 4 of this SOP.

7-5 The dive boat will return to the station coordinates recorded at the time of sampler placement. Navigating with the dGPS, an anchored line will be dropped onto the station coordinates as close as practicable to the station location.

7-6 For the inshore stations (PW002, PW003, and PW006), and the upriver station (PW007), the tag line previously secured to a piling or other tie-point will be located, and the diver will follow the line to the samplers, and proceed with Steps 7-10 through 7-13, and then the sediment sampling described in Section 8 of this SOP.

7-7 For the offshore stations (PW001, PW004 and PW005), the passive samplers will be located using a hand-held pinger locator (Benthos DPL-275). Prior to entering the water the FD will check to confirm the locator is (1) working, and (2) that a signal from the sampler is being received

7-8 For the offshore stations the diver will enter the water with the hand-held pinger locator (Benthos DPL-275), and descend down the anchor line to the approximate sediment location. It is expected that the diver will be able to locate the sampler by sound. In the event the diver is able to quickly locate the sampler, they can proceed with sampler retrieval, as described below.

7-9 In the event the diver cannot locate the tied tag line for the inshore stations, or the sampler by sound for the offshore stations, the diver will proceed to conduct a search arc from the estimated location to locate the sampler tag line. The diver will swim in 180^0 search arcs in approximately 3 ft intervals out from the station location anchor line. Visibility is expected to be low during this search; the diver has the option of using a common garden hand rake to pull through the sediment to snag the tag line.

7-10 Upon location of the tag line and sampler, the diver will first proceed to pull out the tag line anchors from the sediment (or release the tag line from the tie-point), and float the line to the surface. It may be necessary to use a small lift bag to bring the tag line to the surface. The diver must confirm that the tag line is still attached to the sampler, and the boat crew must confirm that the tag line is securely fixed on the deck before the sampler may be pulled from the sediment. This is to ensure the sampler will not accidentally float away during retrieval.

7-11 The diver will measure the distance from the mudline to the top of the exposed PE frame, and report that distance to the FD, who will record that measure in the field notebook.

7-12 Prior to extracting the PSD, the sediment core sampler will be driven in between the two PSDs following the procedures defined in Steps 8-5 through 8-7. Care must be taken by the diver to ensure that (1) the core is driven between the two PSD samplers approximately two inches (2 diver-gloved fingers) from each sampler, and (2) that the core drive is strictly vertical in order to not contact and potentially damage the PE sheet,

and (3) that the integrity of the sediment stratigraphy for core logging at the laboratory is preserved.

7-13 Once the core sampler is in place, the passive sampling frames may then be withdrawn (one at a time) by slowly pulling the frame vertically from the sediment. If necessary, a gentle rocking motion may be used to assist in the release of the frame. Upon withdrawal, the diver will note any tears or rips in the PE to the surface team, and then notify the support team that the sampler may be lifted to the surface via the tag line. The diver will remain at the sampler location to complete collection of the sediment core, as described in Section 8 of this SOP.

7-14 Once the passive porewater sampler is onboard, it will be processed according to the procedures described in Section 9 of this SOP.

7-15 All samples from a site (PE and sediment) must be labeled, secured, properly packed, and placed into an ice cooler according to the procedures in Section 9 of this SOP, before the field crew may move to the next location.

8. SEDIMENT CORE COLLECTION

8-1 Retrieved sediment cores may have contaminated mud on the exterior of the sample tube when they reach the surface. All boat personnel handling sediment core samplers must wear new nitrile gloves at all times.

8-2 The procedures for navigation and target station location are as described in Section 4 of this SOP.

8-3 The diver's helmet cam will be operational throughout the dive-portion of the field placement. The video from the procedure will become part of the Porewater Characterization Report.

8-4 The sediment core sample will be collected by the diver from between the passive porewater sampler location as described previously in Step 7-12.

8-5 A tag line will be clipped to the sediment core sampler, and carefully lowered to the diver. The hand-core samplers will be constructed of pre-cleaned 10 cm (4-inch) diameter aluminum barrels approximately 45 cm in length, and fitted with a bottom core-catcher. The tag line will remain clipped to the sampler throughout coring and retrieval.

8-6 The diver will push the sample tube into the sediment between the two PE samplers as described previously in Step 7-12. The desired sample depth is 30 cm (12 inches) below the mudline; the diver should attempt to push the core barrel 40 cm into the sediment. As required, the diver may gently rock the sampler back and forth, and if necessary, hammer the tube into the sediment if significant resistance is encountered.

8-7 Once the core has been advanced to the minimum acceptable depth of 30 cm below mudline, the diver will so indicate and the FD will record the time of sample collection in the field notebook and on the sample collection log.

8-8 Prior to proceeding with the core withdrawal, the passive samplers will be removed and secured as described previously in Step 7-13.

8-9 The diver will then withdraw the core sampler, placing caps over the open core barrel on the top and bottom of the tube, and signal the support boat that the core may be pulled to the surface. The diver will make careful note of where the core collection hole in the sediment is, and wait at the location until the FD indicates that a successful sample has been collected.

8-10 Once the core is on board, any excess water in the core barrel is decanted, and the amount of material retained in the core tube is measured and recorded in the field log. Recovery is defined as the amount of sediment retained (acquisition) in the core tube divided by the amount the core tube penetrated into the sediment column (penetration). The minimum acceptable recovery is 30 cm.

8-11 In the event that insufficient material is retained, the contents of the core barrel will be discarded, the core barrel cleaned, and lowered back to diver for a second attempt. The second drive will be two inches (two diver fingers) upstream of the original location. If a third attempt is needed, the third drive will be made two inches downstream of the original location. Up to three attempts will be made, at which time the EPA RPM will be contacted to discuss further options for sediment collection.

8-12 For each core attempt, the station name, latitude/longitude, time of collection, depth to mudline, the river elevation at the time of collection and depth of penetration are noted in the field log.

8-13 Once the core barrel is onboard, it will be handled according to the procedures described in Section 9 of this SOP.

8-14 All samples from a site (PE and sediment) must be labeled, secured, properly packed, and placed into an ice filled cooler according to the procedures in Section 9 of this SOP before the field crew may move to the next location.

9. FIELD PROCESSING OF PE SAMPLERS AND SEDIMENT CORES

9-1 Once onboard the boat, the PE in the samplers will be immediately rinsed with laboratory reagent-grade water and rubbed with a clean Kimwipe™, and at least one additional rinse

to remove as much adhering sediment as possible1. All personnel handling the PE samplers must be attired in Level D personal protective gear and wear new nitrile gloves for each sampler.

9-2 The PE sampler will be photographed, and the general condition of the PE will be noted in the field log (e.g., intact, tears in the PE, biofouling, presence of oil, etc.).

9-3 The sample's identification number will be recorded onto the sampler sleeve, and then the entire sampler will be wrapped in aluminum foil. The station identification number will be recorded with an indelible marker on the aluminum foil, along with the date and time.

9-4 The PE sampler will then be placed into the ice filled cooler for transport to ALS in Kelso, WA for processing and analysis.

9-5 Sediment core samples collected will also have the station ID recorded onto the sample tube; this can be scratched directly onto the tube. The core lids will be secured with duct tape, and the sample ID, date and time will be written using an indelible marker onto the taped lids with an arrow indicating the direction to the surface of the sediment. "TOP" will be written onto the taped lid indicating the sediment surface.

9-6 These secured cores will then be placed upright into an ice filled cooler for transport to ALS.

9-7 The samples must remain in the custody of the FD or SAC at all times during the transport to, and during processing at ALS.

[1] Additional cleaning of the PE will occur at the laboratory to remove all remaining sediment, prior to analysis.

Attachment 4

RM11E Porewater Sampling Reconnaissance Activity Report

DAILY ACTIVITY REPORT

Report # 1 **DATE** May 2, 2014 S M T W TH ***F*** S

Field Investigation Manager: Tim Thompson

WEATHER Bright Sun Partly Cloudy ***Overcast*** Rain Snow

TEMPERATURE °F <32° 32-45° ***45-60°*** 60-70° 70-85° >85°

WIND Still **Mod.** High

HUMIDITY Dry **Mod.** Humid

RIVER STAGE:

Time	15:00	15:15	15:30	15:45	16:00	16:15	16:30	16:45	17:00	17:15	17:30	17:45	18:00
USGS Morrison Bridge (ft)	8.01	7.98	7.94	7.9	7.86	7.82	7.78	7.76	7.72	7.73	7.71	7.67	7.67
NAVD 88 (ft)	13.06	13.03	12.99	12.95	12.91	12.87	12.83	12.81	12.77	12.78	12.76	12.72	12.72

TASK: ☐ Industrial Area Soils ☐ Industrial Area Groundwater ☐ Surface Water ☑ Sediment ☐ MIS

SUBCONTRACTORS/VISITORS ON SITE: Erin Carroll Hughes, GSI; Justin Siewert, BMC; Mike Wright, BMC; Steven Crouchley, BMC (Diver); Shane Veentjer, BMC; Shawn Oliveira, CDM; Tim Thompson, SEE.

EQUIPMENT ON SITE: Ballard Marine Construction vessel

WORK PERFORMED (INCLUDE ANY SAMPLES COLLECTED):

- **On Board at 1530 hours**. Conducted the pre-dive briefing per the Standard Operating Procedures and a Health and Safety briefing.
- **Diver-Placement and Retrieval of Passive Samplers and Co-located Sediment Samples (SOP)**. This included an overview of sampling locations and methods for the BMC crew plus GSI and CDM, as well as focused training for the diver on the operation of the modified passive sampling device (PSD) per the changes listed in the April 17, 2014 EPA Response to the SAP. These changes, and photographs of the sampling equipment as modified, are presented in Attachment 1 and Figure 1.
- **Navigation.** Navigation was conducted according to the procedures listed in the SOP, with the exception that the check of the dGPS at the inside corner upstream (approximately SE) of the Glacier Dock, or the inside downstream corner (approximately NE) of the Cargill Dock did not occur. As noted below under Quality Control Activities, there were three identical dGPS units onboard, all reporting within 1 ft. of each other. Therefore, the additional navigation check was not thought necessary for this reconnaissance survey. All reconnaissance stations were on location; the diver did not deviate from the targeted location.
- **PW003** Successful drive of the PSD with no tears or other visible stress in the PE sheets. Diver reported shallow silt with ¼" gravel. Hammer required for sampler placement, with some bowing of the stainless steel PE covers reported by diver, but remained principally in place. Insert time was 30 seconds; allowed 60 seconds to elapse after pulling the PE covers before removing the sampler from the sediment. See additional information on sample description log.
- **PW006** Successful drive of the PSD with no tears or other visible stress in the PE sheets. Prior to sampling additional care was taken with the PE covers to ensure they were installed correctly. Diver reported bottom conditions of silt, rip rap, twigs and sticks. PSD pushed in manually approximately 2", and then hammered into the sediment the remaining length. PE plate covers remained in place with no bowing. Insert time was 45 seconds, allowed 60 seconds to elapse before removing the sampler from the sediment. Cycle time for diver (away, installation, return) was 8 minutes. See additional information on sample description log.
- **PW002** Successful drive with no tears or other visible stress in the PE sheets. Diver reported principally woody debris and sediment. Insert time not noted; will plan on getting those later from diver video. Photo taken and shown as Figure 3. Diver Cycle Time 7 minutes. See additional information on sample description log.
- **PW001** Had planned to add this fourth station. Anchored barges and worked toward locating dive location. Location is offshore in the river channel, and between the current and then the increasing winds, along with rapidly approaching darkness, elected not to make this additional attempt.

QUALITY CONTROL ACTIVITIES (INCLUDING FIELD CALIBRATIONS)

- Three Trimble GeoXH – GeoExplorer 2008 Series - dGPS units onboard. All units reported the same location within an accuracy of 1 ft.
- Lead line depth taken at each station, Water depths corrected to USGS Willamette River Gage depths, and then converted to NAVID88.

HEALTH AND SAFETY LEVELS AND ACTIVITIES: **Tailgate Meeting Held ☑**

- H&S Briefing conducted prior to undertaking PSD reconnaissance. This was the second briefing of the day for the field crew: the additional meeting was necessitated by the addition of T. Thompson

PROBLEMS ENCOUNTERED/CORRECTION ACTION TAKEN:

- No problems of significance encountered – all went well.

SPECIAL NOTES:

- No significant changes to the PSD SOPs or Porewater SAP, other than those listed here, are necessary. Revisions to the SAP will be incorporated into the Final SAP

TOMORROW'S EXPECTATIONS:

- File report with the RM11E Group and with EPA

ATTACHMENTS:

Attachment 1

Site Description Logs and Photos

PREPARED BY: Tim Thompson

SIGNATURE: Filed electronically.

ATTACHMENT 1: Changes to the March 3, 2014 Reconnaissance Survey Procedures

1. **Changes to the Passive Sampling Device** - Figure 1 of the SOP showed a general schematic of the Passive Sampling Device (PSD). The basic design has been modified to accommodate a polyethylene (PE) sheet that is 10 cm wide by 50 cm in length. In addition, the PSD was modified to be able to be coupled to a detachable diver-operated hammer, a depth-stop built into the diver-operated hammer, as well as to have removable stainless steel protective covers over the PE sheeting.

   Figure 1 below on the left shows the assembled PSD with hammer (A), covers (B) and depth stop (C). Figure 1 on the right shows the PSD post-retrieval with the PE intact. The depth guide (C) is more clearly seen in this photograph. Figure 2 shows the placement of the stainless-steel cover over the PE prior to sampling deployment.

2. **Changes to Placement and Retrieval of the Passive Sampling Device** - Procedures in the field generally followed those listed in Section 5 of the SOP: Reconnaissance Survey Procedures. The changes to the PSD necessitated a complete re-write for Procedures 5-5 and 5-8. The revised procedures, which were implemented in the field, are as follows:

   5-5 At the confirmed target location, the diver will report the general bottom conditions (e.g., sediment type, presence of debris) to the surface team. The PSD assembly is lowered to the diver on a clipped line. The diver will insert the PSD Assembly into the sediment, parallel to the flow of the river, and push or hammer the PSD into the sediments up to the fixed stop legs on the sampler, which are preset to allow 30 cm of PE exposure to surface sediment and 20 cm exposure to the overlying water. The sampler should be driven at a slow, steady rate; the process of insertion should take 0.5 - 1 minute. The diver may gently rock the sample device if/when resistance is encountered. During the insertion, the diver will communicate with the boat to describe the degree of ease/difficulty associated with the push, and whether refusal is encountered prior to completion. This information is recorded into the field log notebook.

   5-8 Upon successful insertion, the diver will first remove the stainless steel covers from both sides of the PSD, and clip those to the carabineer connected to the boat line. After removal of the covers, one minute is allowed to elapse to allow for the sediment to consolidate around the device. If possible, a photograph or video will be taken. The sampling device will then be withdrawn by slowly pulling the frame vertically from the sediment. If necessary, a gentle rocking motion may be used to assist in the release of the frame. Upon withdrawal, the diver will note any tears or rips in the PE to the surface team, and then signal the surface team to bring the PSD assembly to the surface. The diver will remain at the sampling location until confirmation by the surface team that there are no rips or tears to the PE.

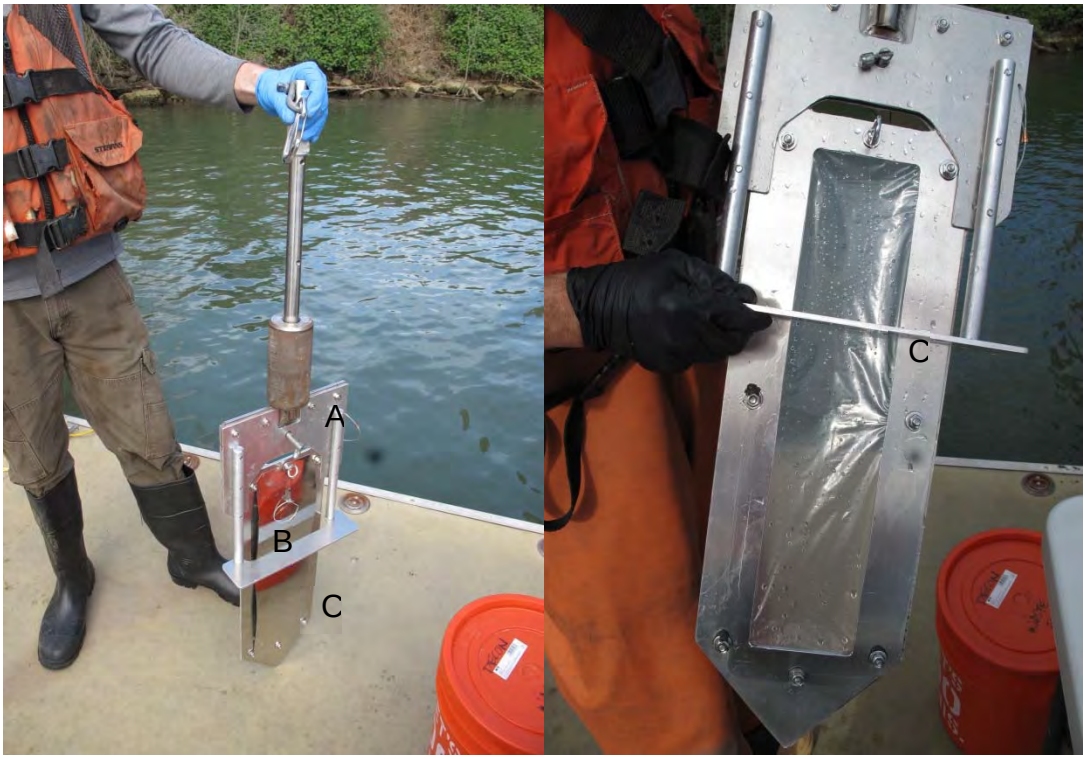


Figure 1: Passive Sampling Device Assembly

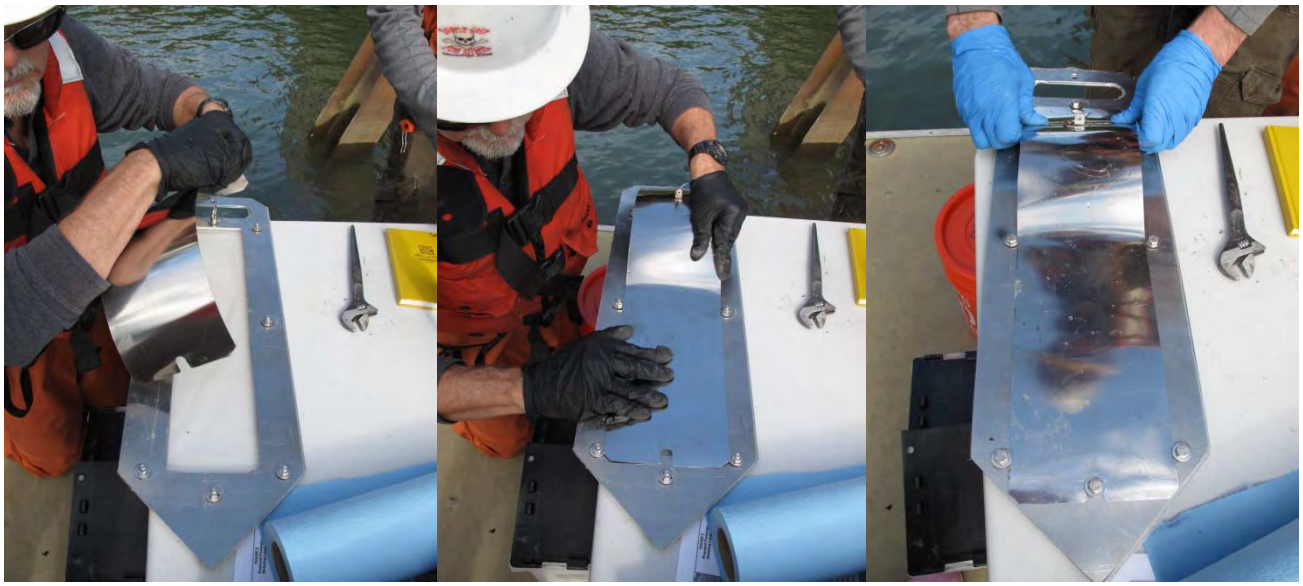


Figure 2: Stainless Steel PE Cover

PW003 Sample Description Log

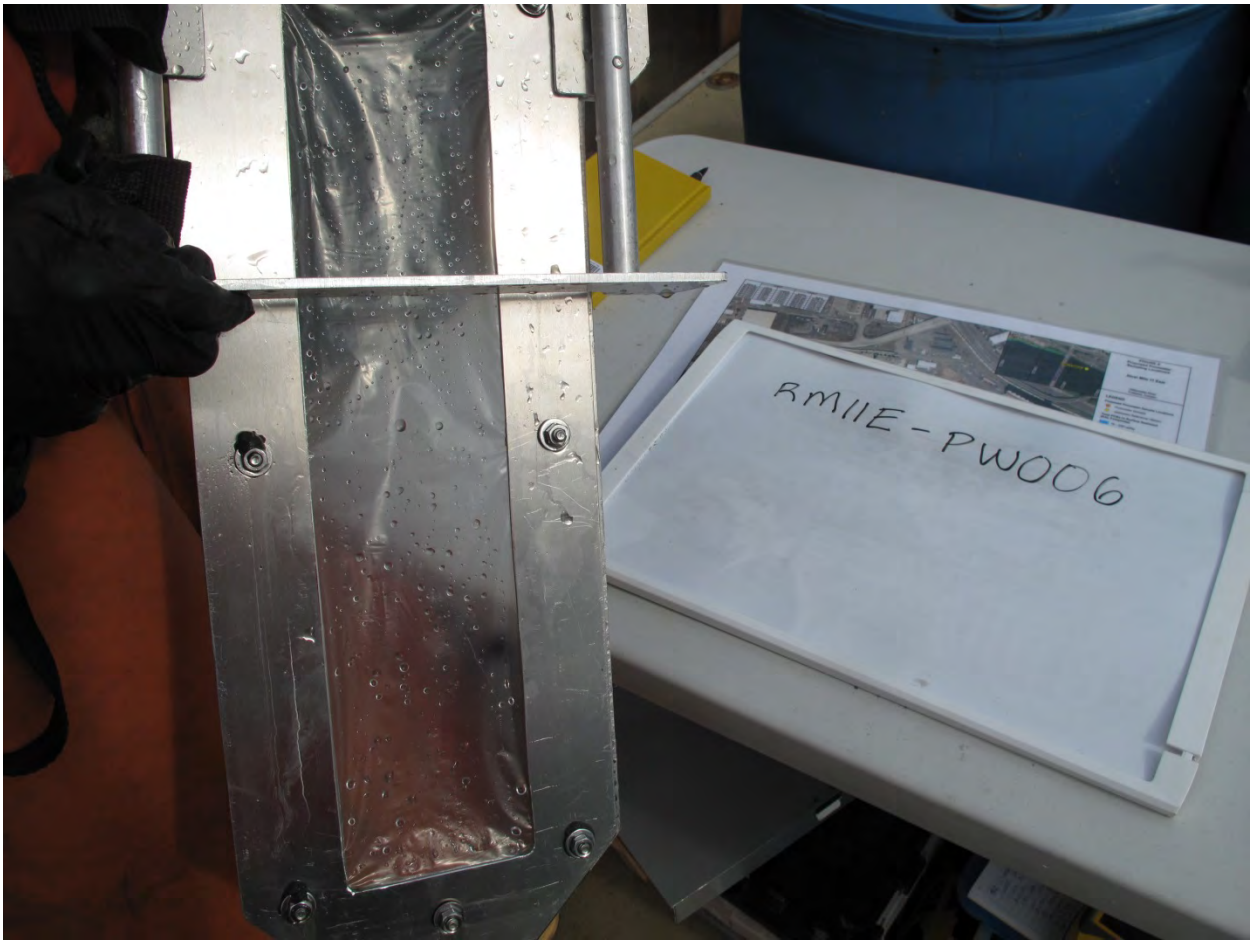
Proposed Coordinates		Actual Location	
N: 689248.6	E: 7644329.2	N: 689250.69	E: 7644325.54
		Measured Depth = -13.4 ft	River Height = 12.3 ft
		Corrected Elevation (NAVD88 ft) = -0.57	

Description: Shallow silt sitting on 1/4" gravel. Required hammer to push in PSD – approximately 30 seconds till PSD inserted to stops. Steel cover plates bowed out during drive, but readily removed. Inspection at removal by diver did not indicate any tears or rips. Confirmed once sampler retrieved on-board.

PW006 Sample Description Log

Proposed Coordinates		Actual Location	
N: 689004.6	E: 7644579.4	N: 688997.94	E: 7644586.21
		Diver In Water: 16:51	Diver On Board: 16:59
		Measured Depth = -36.9 ft	River Height = 12.77 ft
		Corrected Elevation (NAVD88 ft) = -24.7	

Description: Silt, rip rap, river rock, twigs and sticks. Approximately 2" push by hand, and then approximately 45 seconds of hammer till PSD inserted to stops. Steel cover plates remained intact during placement. Inspection at removal by diver did not indicate any tears or rips. Confirmed once sampler retrieved on-board.



PW002 Sample Description Log

Proposed Coordinates		Actual Location	
N: 689344.3	E: 7644176.0	N: 45.53686 Lat N: 689344.1 OSP	E: 122.67765 Lon E: 7644177.6 OSP
		Diver In Water: 17:45	Diver On Board: 17:52
		Measured Depth = -17.3 ft	River Height = 12.72 ft
		Corrected Elevation (NAVD88 ft) - 4.58	

Description: Woody debris, sediment. Inspection at removal by diver did not indicate any tears or rips. Confirmed once sampler retrieved on-board.

Note: GSI was recording in Latitude/Longitude, while SEE was using Oregon State Plane Coordinates (OSP). The Latitude/Longitude for this station was entered into the log book, and then converted to OSP North coordinates

Attachment 5

SOP for Preparing Polyethylene (PE) Passive Samplers and PCB Congener Performance Reference Compounds Loading. ALS SOP Code: SOC-PE/PRC

STANDARD OPERATING PROCEDURE

DOCUMENT TITLE:	***PREPARATION OF POLYETHYLENE (PE) PASSIVE SAMPLERS WITH PERFORMANCE REFERENCE COMPOUND (PRC) LOADING***
REFERENCED METHOD:	***Gschwend & MacFarland – Massachusetts Institute of Technology***
SOP ID:	***SOC – PE/PRC***
REV. NUMBER:	***0***
EFFECTIVE DATE:	***5/12/2014***

PREPARATION OF POLYETHYLENE (PE) PASSIVE SAMPLERS WITH PERFORMANCE REFERENCE COMPOUND (PRC) LOADING

SOC – PE/PRC

ALS-KELSO

SOP ID:	SOC – PE/PRC	Rev. Number:	0	Effective Date:	05/12/2014

Approved By: _____ Date: 5/13/14
Department Supervisor – Jon James

Approved By: _____ Date: 5-13-14
QA Manager – Lee Wolf

Approved By: _____ Date: 5/13/14
Laboratory Director – Jeff Grindstaff

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

1. SCOPE AND APPLICATION

1.1. This method describes a procedure for preparing and handling polyethylene (PE) films that will be cut into strips and used in polyethylene devices (PEDs) to passively sample hydrophobic organic compounds (HOCs) in environmental media.

1.2. This method generates PE that can be deployed within PEDs for passive sampling of HOCs in atmospheric, aqueous, or sediment-porewater systems.

1.3. PE that is prepared by this method is suitable for laboratory or *in situ* field deployment.

2. SUMMARY OF METHOD

2.1. A known mass of low density polyethylene (LDPE) sheet (usually ~1-3 g) is cleaned by sequentially extracting with methylene chloride, methanol, and ultrapure water in a closed glass vessel.

2.2. Clean PE is equilibrated with performance reference compounds (PRCs) dissolved in water or methanol-water (see Appendix 1 for possible PRCs).

2.3. Prepared PE is stored in contaminant-free, sealed, glass vessels.

2.4. Shortly before deployment, the PE is cut into strips and transported to the field wrapped in clean aluminum foil.

2.5. In the field, the PE is exposed to the environmental medium of concern. The HOCs in the medium diffuse into the PE, while PRCs diffuse out.

3. INTERFERENCES

PE is susceptible to contamination from atmospheric vapors and contact with surfaces (e.g., worker hands), so it must remain in clean sealed vessels until deployment.

4. APPARATUS AND MATERIALS

4.1. Extraction vessels: 1-L glass bottles or screw capped jars (foil-lined lids).

4.2. Storage vessels: bottles with glass stoppers or amber jars (foil-lined lids).

4.3. Bottle/jar tumbler, shaker table, bottle roller, or equivalent.

4.4. Low density polyethylene (LDPE): commercial grade, large sheet at 25μm (1 mil) or other thickness as specified by the Statement of Work (SOW). The thickness is chosen to be strong enough to withstand stresses during deployment (e.g., insertion into sediment), but thin enough to exchange a significant fraction (e.g., >20%) of its PRCs during the deployment time to be used.

4.5. Food grade aluminum foil (solvent cleaned to remove any organic residue from foil production).

4.6. Stainless steel forceps.

4.7. Teflon (or similar non-contaminating material) cutting board.

5. REAGENTS

5.1. Methylene chloride, CH2Cl2, pesticide grade or equivalent.

5.2. Methanol, CH3OH, pesticide grade or equivalent.

5.3. Organic-free reagent water (as defined in SW-846 Chapter 1).

5.4. Research grade PRCs certified 98+% pure.

Note: Specific standard materials, concentrations, solvents, and solvent purity requirements will be determined based upon the target HOCs of concern for the particular application.

6. PRESERVATION AND HANDLING

6.1. Clean PE should be stored in clean sealed glass vessels.

6.2. Until deployment, prepared PE (PE loaded with PRCs) is stored in sealed glass containers with a few mL of organic-free reagent water added to maintain 100% relative humidity within the storage vessels (minimizing sorptive losses of PRCs to glass vessel walls).

6.3. Laboratory and field personnel should wear nitrile or latex gloves whenever handling clean PE.

6.4. Methylene chloride-rinsed, stainless steel forceps and scissors are used when manipulation of clean PE is required.

6.5. Methylene chloride-rinsed, aluminum foil is used to cover any surface that clean PE may encounter.

7. PROCEDURE

7.1. Polyethylene Cleaning Procedure: LDPE is purchased from hardware/painting stores in large sheets ('dropcloth or plastic tarp' material) with thickness of 25μm (1 mil) or as specified in the SOW, depending on the user's need for strength (choose thicker) and desire to use short deployment times (use thinner). The sheet is cut into strips sized for environment and frames to be used as specified in the SOW. The dimensions of the PE strips can range significantly, depending on the organization conducting the field work and the associated SOW. An organic solvent cleaning sequence is then used to prepare the PE. This process ensures that extractable oligomers, plasticizers, and contaminating organic chemicals are

removed from the PE prior to use. All extractions are performed sequentially in the same container.

7.1.1. Methylene chloride is placed into the extraction vessel, and the PE strips are immersed in the container for 24 hours to enable time for diffusive transfers out of the PE. The initial methylene chloride extract is discarded and a second methylene chloride extraction is performed for 24 hours. The second methylene chloride extract is discarded and replaced by methanol in order to remove methylene chloride from the PE. Methanol immersion is also done for 24 hours. The initial methanol extract is discarded and followed by a second methanol soak for 24 hours. Finally, the second methanol extract is discarded and the PE undergoes three 24-hour soaks with organic-free reagent water (within the same extraction vessel) to remove residual methanol from the PE.

7.1.2. The cleaned PE is stored in organic-free reagent water in the extraction vessel until further processing.

7.1.3. Record documentation of the decontamination process using the laboratory bench sheet included in Appendix A.

7.2. Polyethylene Preparation with Performance Recovery Compounds (PRCs): PRCs are loaded into the clean PE prior to its field deployment by utilizing either aqueous (Fernandez et al. 2009) or 80:20 methanol:water equilibrations (Booij et al., 2002). Depending on the hydrophobic organic compounds of interest, PRCs should be chosen which mimic mass transfer phenomena governing exchanges during field deployments. It is important to avoid adding PRCs that the analytical laboratory already uses as surrogate or injection standards. PRC loading is performed by placing the PE in pre-cleaned glass vessels containing known PRC solutions made up in organic-free reagent water with or without pesticide-grade methanol, depending on whether aqueous or methanol:water loading is done. The PE user should estimate the expected accumulation of target compounds in the passive sampler and seek to load with similar levels of PRCs to facilitate the eventual chemical analyses. Sufficient PRC equilibration time during this PE preparation step is necessary to ensure uniform PE loading across the entire PE thickness. Hence, a thicker PE sheet is more robust for field use, but takes longer to load with PRCs.

7.3. Isotopically labeled compounds are useful internal standards when Gas Chromatography-Mass Spectrometry (GCMS) is the method of separation and detection. For example, C13-labeled PCBs are effective methodological standards for PE passive sampling. One subset of compounds distributed across the range of PCB Congeners to be assessed should be used as PRCs, while another set is used as surrogate (recovery) compounds during later analysis of field-deployed PE. In addition, a third set is used as internal standards for instrumental analysis. Similar sets of labeled compounds should be used for other compound classes (e.g. PAHs, Dioxin/Furans, etc.). The appropriate labeled isotopes will be incorporated into the associated project SOW.

Note: if PE samples are eventually to be analyzed at a third party laboratory, PRC choices must be made so as not to conflict with recovery and injection standards used by that laboratory.

7.4. As subsequent analysis (e.g., GCMS) is best achieved with both PRCs and target HOCs present at like concentrations in the PE extracts, the optimal concentration level of the PRC loaded into the PE is dependent on the environment in which the PE is to be deployed. For example, if a target HOC is expected to occur in the water or pore water near 1 ng/L levels, one can use that compound's LDPE-water partition coefficient (e.g., Fernandez et al., 2009; Lohmann, 2012) to estimate the expected levels in the PE after deployment:

7.5. The appropriate concentrations for the loading solution will be incorporated in the project SOW.

7.6. Aqueous PRC Loading: A solvent-cleaned and dried glass container is filled with ultrapure water that has been spiked with known concentrations of PRCs. A known mass of pre-cleaned PE is then added and weighted to insure complete PE submersion. The vessel is agitated to remove any air pockets adhering to the submerged PE. Equilibration times vary for different PRC/PE thickness combinations and the PE-water phase ratio. For PCBs, use at least 30 days to insure homogeneous distributions of the PRCs throughout the entire thickness of the PE film unless faster equilibration has been confirmed. Confirmation can be done by time course measures of PRC concentrations in the PE or by showing that concentrations of PRCs are the same for films of different thicknesses, but the same masses. Generally, PE is stored in the PRC solution until it is to be deployed.

7.7. Methanol-Aided PRC Loading: A solvent-cleaned and dried glass container is filled with an 80:20 mixture of pesticide grade methanol and ultrapure water that has been spiked with known concentrations of PRCs. A known mass of pre-cleaned PE is then added and weighted to insure complete submersion. The vessel should be agitated to remove any air pockets adhering to the submerged PE. Equilibration times vary for different PRC/PE thickness combinations and the PE-solvent phase ratio, but typically this step is completed within 7 days since methanol swells the PE and thereby speeds PRC diffusion into the polymer sheet (Booij et al., 2002). Generally, the PE is stored in the PRC solution until shortly before it is to be deployed. Before deployment, the PRC-loaded PE is rinsed with ultrapure water, and then it is soaked in ultrapure water for 24 h to remove methanol from the PE. This methanol leaching step is repeated twice to insure complete methanol removal.

7.8. Record information related to the PRC loading to document the entire process using the bench sheet included in Appendix B.

7.9. PE and PED Storage and Shipment

7.9.1. Prepared PEDs in their foil envelops may be stored a few days at ambient temperature prior to deployment. Freezing or excessive heat should be avoided to minimize the likelihood of changing the polymer crystallinity. It is recommended that PEDs be hand carried or shipped in a timely fashion (Overnight or Next Day if possible) to minimize chances sampler contamination or damage.

7.9.2. If PE is to be shipped to another location for PED assembly, the PE strips are individually sealed in pre-cleaned glass vials that contain a little water (~1 mL). Freezing during shipping should be avoided, but PE strips should be shipped to maintain a temperature of 4±2˚C.

8. QUALITY CONTROL

8.1. PRC Loading Validation: Representative samples of prepared PE should be collected, extracted, and analyzed prior to field deployment to validate that the PRC concentrations are consistent with their intended loadings, and these standards have uniform concentrations in a batch of PE.

8.2. Target HOC Blanks: Subsamples of prepared PE commensurate in size with the planned environmental PE samples as defined in the project SOW should be collected, extracted, and analyzed prior to field deployment to demonstrate that other substances have not contaminated the PE which would contribute to interfering background for the target HOCs.

9. METHOD PERFORMANCE

9.1. PRC data obtained from PE check samples collected from representative parts of the prepared PE should be consistent within about 10% (i.e. 100 x standard deviation / mean).

9.2. Target HOC concentrations should be undetectable in the prepared PE (e.g., < 1 ng/g PE assuming 100 mg PE subsamples).

10. REFERENCES

10.1. Adams, R.G., Lohmann, R., Fernandez L.A., MacFarlane, J.K., and Gschwend, P.M., Environmental Science and Technology; 2007, 41, 1317-1323.

10.2. Booij, K, Smedes, F., van Weerlee, E.M., Chemosphere 2002, 46, 1157-1161.

10.3. Fernandez, LA, MacFarlane, J.K., Tcaciuc, A.P., and Gschwend, P.M., Environmental Science and Technology; 2009, 43, 1430-1436.

10.4. Hawker DW and Connell DW., Environmental Science and Technology; 1988, 22, 382-387.

10.5. Lohmann, R. MacFarlane, J.K., and Gschwend, P.M.; Environmental Science and Technology; 2005, 39, 141-148.

10.6. Lohmann, R. Environmental Science and Technology; 2012, 46, 606–618.

Appendix A – Bench Sheet for PE Decontamination

ALS Environmental, Inc.

Polyethylene (PE) Cleaning sheet

Analyst: _____ Date: _____

PE dimensions _____

	Start time/Date	End time/Date
Initial Methylene Chloride soak	_____	_____
Second Methylene Chloride soak	_____	_____
Initial Methanol soak	_____	_____
Second Methanol soak	_____	_____
Initial Water soak	_____	_____
Second Water soak	_____	_____
Third Water soak	_____	_____

Comments: _____

_____

_____

_____

Appendix B – Bench Sheet for PRC Loading

ALS Environmental, Inc.

PRC Loading Sheet

Analyst: _____ Date: _____

Aqueous or Methanol-Aided PRC Loading: _____

Batch ID : _____

PE Dimensions: _____

Number of PE Strips per Liter: _____

PRC Loading Solution Information

Stock Standard ID: _____

Stock Standard Concentration (ug/L): _____

Stock Standard Expiration: _____

Aliquot of Stock Standard (mL): _____

Final Volume (mL): _____

Concentration in PRC Loading Solution (ug/L) _____

PE I.D.	Start Time	Start Date	End Time	End Date

Witness: _____

Comments: _____

_____

_____

_____

Attachment 6

SOP Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Bio solids and Tissue by Isotope Dilution HRGC/HRMS. ALS SOP Code: HMS-1668

ALS Environmental

DOCUMENT TITLE:	CHLORINATED BYPHENYL CONGENERS IN WATER, SOIL, SEDIMENT, BIOSOLIDS, AND TISSUE BY ISOTOPE DILUTION
REFERENCED METHOD:	EPA 1668A
SOP ID:	HMS-1668A
REV. NUMBER:	04.0
EFFECTIVE DATE:	6/21/2013

CHLORINATED BYPHENYL CONGENERS IN WATER, SOIL, SEDIMENT, BIOSOLIDS, AND TISSUE BY ISOTOPE DILUTION

SOPID:	HMS-1668A	Rev. Number:	04.0	Effective Date:	6/21/2013

Approved By: _____ Date: 06/21/13
Technical Director - Lan Le

Approved By: _____ Date: 6/21/13
QA Manager – Rebecca Pierrot

Approved By: _____ Date: 06/21/13
Laboratory Director – Jim Plassard

Archival Date: _____ Doc Control ID#: _____ Editor: _____

CHLORINATED BYPHENYL CONGENERS IN WATER, SOIL, SEDIMENT, BIOSOLIDS, AND TISSUE BY ISOTOPE DILUTION

1 Forward

1.1 This procedure is currently undergoing a major revision. Until such time that the revision is complete, the analytical procedures outlined in the attached document reflect current laboratory practice, with the exception that the laboratory is currently monitoring the primary ion for Dichlorobyphenyls, which greatly reduces contribution of PFK interference. This change will be reflected in the new revision.

1.2 The sample preparation, extraction, and purification techniques are currently addressed in individual extraction and cleanup Standard Operating Procedures, which supersede this document.

1.3 All references to Columbia Analytical Services, Inc. (CAS, CAS/Houston) refer to ALS Environmental - Houston HRMS.

Columbia Analytical Services

SOP CHANGE FORM

SOP Title: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by Isotope Dilution HRGC/HRMS
SOP Code: HMS-1668B
SOP Revision No.: 1
SOP Date: 08/13/10 (changes effective 09/06/11)
SOP Section(s) Affected by Change: Sections 11.1.2.2, 11.2.1.3

Description of Change:

11.1.2.2 Prior to any sample manipulation, the sample container is allowed to reach ambient temperature; this is monitored by the cessation of condensation on the sample container. Following the sample warming, mark the sample meniscus on the sample container using the Diamond Scribe tool or black permanent marker; chlorine measurements may be done at this time.

11.2.1.3 Transfer the sample to the ½ gallon extraction vessel.Add 60 mL of dichloromethane to the spiked sample in the ½ gallon extraction vessel. Vent the vapors under a fume hood by shaking for a few seconds then unsealing the cap. Repeat this process until pressure no longer builds in the vessel. Extract the sample by shaking the jar manually or on an automatic shaker for 3 minutes. Allow the layers to separate for a minimum of 10 minutes and transfer the dichloromethane layer (bottom) into an appropriately labeled 250mL jar. If an emulsion layer forms, employ mechanical techniques (ultrasonic bath, centrifugation) to complete the phase separation.

Reason(s) for Change(s):
These changes reflect technically-sound practice, and are required by the method as minimum sample processing steps.

Change(s) Submitted by: EB	Date: 9/6/11

Approvals:

Technical Reviewer Signature: [illegible]	Date: 9/6/11
QA PM Signature: R Pierrot	Date: 9/6/11
Department Supervisor/Manager Signature:	Date:

Change(s) Effective Date: 09/06/11

Distribution: Original filed with original SOP Photocopy attached to each controlled copy

Columbia Analytical Services

SOP CHANGE FORM

SOP Title: Method 1668: Analysis of Polychlorinated Biphenyl Congeners by High Resolution Gas Chromatography/ High Resolution Mass Spectrometry (HRGC/HRMS)

SOP Code: HMS-1668A

SOP Revision No.: 4

SOP Date: 08/20/10 (effective 08/19/11)

SOP Section(s) Affected by Change: Sections 11.1.2.2, 11.2.1.2, 11.2.1.3, 11.2.1.4

Description of Change:

11.1.2.2 Prior to any sample manipulation, the sample container is allowed to reach ambient temperature; this is monitored by the cessation of condensation on the sample container. Transfer the sample to the ½ gal. extraction vessel. Mark the volume on the ½ gal. extraction vessel with a permanent marker at the sample meniscus.

11.2.1.2 Spike 1.0mL of the Matrix standard spiking solution at 5ng/mL (Table 3) into the LCS/DLCS aliquots. Record this addition on the bench sheet. These will serve as both the precision and accuracy for the batch. Following addition of both Labeled and Matrix standard spiking solutions, allow the samples to equilibrate for one hour.

11.2.1.3 Add 60 mL of dichloromethane to the spiked sample in the ½ gal. extraction vessel. Vent the vapors under a fume hood by shaking for a few seconds then unsealing the cap. Repeat this process until pressure no longer builds in the vessel. Extract the sample by shaking the jar manually or on an automatic shaker for 3 minutes. Allow the layers to separate for a minimum of 10 minutes and transfer the dichloromethane layer (bottom) into an appropriately labeled 250mL jar. If an emulsion layer forms, employ mechanical techniques (ultrasonic bath, centrifugation) to complete the phase separation.

11.2.1.4 Repeat Section 11.2.1.3 twice to give a total extraction volume of 180 mL of dichloromethane. Following extraction, remove any excess water from the dichloromethane portion by swirling the sample extract and removing the water that collects on top.

Reason(s) for Change(s):
These changes reflect technically-sound practice, and are required by the method as minimum sample processing steps.

Change(s) Submitted by: EB	Date: 8/19/11

Distribution: Original filed with original SOP Photocopy attached to each controlled copy

Approvals:

Technical Reviewer Signature:	Date: 8/19/11
QA PM Signature: Rebecca Prent	Date: 8/19/11
Department Supervisor/Manager Signature:	Date: 8-19-11

Change(s) Effective Date: 8/23/11

Distribution: Original filed with original SOP Photocopy attached to each controlled copy

Columbia Analytical Services

SOP CHANGE FORM

SOP Title: Method 1668A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment Biosolids, and Tissues by Isotope Dilution HRMS

SOP Code: HMS-1668a

SOP Revision No.: 4

SOP Date: 8/13/10

SOP Section(s) Affected by Change: 12.3

Description of Change:
At a minimum, initial calibration must be performed every one year. Initial calibrations are not used if over one year has passed since the analysis of the initial calibration standards making up the calibration used for sample/QC analyses. Calibration may be required sooner if instrument performance dictates. Also to clarify, initial calibrations are required following any installation of a new GC column or when MS source maintenance is performed (e.g. source cleaning).

Reason(s) for Change(s):
Recent evaluation of historical data indicates that instrument calibrations often do not remain stable for longer periods of time such as 2-4 years. It is acknowledged that dioxin, PCB, and similar compounds exhibit greater stability that most other environmental target compounds, but other factors can impact *calibration* stability in addition to *compound* stability.

Change(s) Submitted by: Jeff Christian	Date: 8/10/11
Approvals:	
Technical Reviewer Signature: Jeff Christian	Date: 8/10/11
QA PM Signature: Rebecca [illegible]	Date: 8/19/11
Department Supervisor/Manager Signature: [illegible]	Date: 8/19/11
Change(s) Effective Date: 8/23/11	

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Columbia Analytical Services

SOP CHANGE FORM

SOP Title: Method 1668 Analysis of Polychlorinated Biphenyl Congers by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS)

SOP Code: HMS-1668A

SOP Revision No.: 4

SOP Date: 8/20/2010

SOP Section(s) Affected by Change: 12.4, 12.4.2, 12.4.3-12.4.6

Description of Change:

12.4 –

System and Laboratory Performance – At the beginning of each 12-hour shift during which analyses are performed, GC/MS system performance and calibration are verified for all native PCBs and labeled compounds. For these tests, analysis of the CS-3 calibration verification (VER) standard (Table 5) and the diluted combined 209 congener solution (Table 3) must be used to verify all performance criteria. *The beginning and end of the 12-hour period shift are defined by the beginning and ending mass spectrometer resolving power check. The mass spectrometer resolving power check is followed by the diluted 209 congener solution and the routine calibration check (CS-3), respectively.* Adjustment and/or recalibration must be performed until all performance criteria are met. Only after all performance criteria are met may samples, blanks, IPRs, and OPRs (LCSs) be analyzed.

12.4.2 becomes 12.4.2.1, and 12.4.2 reads ***Calibration Verification***
Sections 12.4.3 through 12.4.6 become 12.4.2.2 through 12.4.2.5 respectively.

Insert Figures to Figure 3a and 3b, renumber subsequent figures respectively

Reason(s) for Change(s):
Change is in response to NCAR20110412, requiring a more definitive discussion of the 12-hour period.

Change(s) Submitted by: R. Pierrot	Date: 4/26/2011

Approvals:

Technical Reviewer Signature: Virginia Lining	Date: 4/26/11
QA PM Signature: Rebecca Pierrot	Date: 4/26/11
Department Supervisor/Manager Signature:	Date:

Change(s) Effective Date: 4/28/2011

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FIGURE 3a

ANALYTICAL SEQUENCE FOR A TYPICAL 12-HOUR SHIFT

Time	Analysis
Beginning of 12-hr shift	PFK HRMS Tune
	Diluted 209 Congener Solution
	Beginning CS3
	Method Blank
	Samples and Batch QC
End of 12-hr shift	PFK HRMS Tune

FIGURE 3b

ANALYTICAL SEQUENCE FOR A TYPICAL 12-HOUR SHIFT

Time	Analysis
Beginning of 1st 12-hr shift	PFK HRMS Tune
	Diluted 209 Congener Solution
	Beginning CS3
	Method Blank
	Samples and Batch QC
	Closing CS3
End of 1st 12-hr shift Beginning of 2nd 12-hr shift	PFK HRMS Tune
	Diluted 209 Congener Solution
	Method Blank
	Samples and Batch QC
End of 2nd 12-hour shift	PFK HRMS Tune

Columbia Analytical Services

SOP CHANGE FORM

SOP Title: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by Isotope Dilution HRGC/HRMS

SOP Code: HMS-1668A

SOP Revision No.: 4

SOP Date: 08/13/20 (effective 08/20/10)

SOP Section(s) Affected by Change: 11.2.2.1-11.2.2.3, 11.2.3.2, 11.2.4.1-11.2.4.3

Description of Change:
Spiking will now occur prior to mixing solid and tissue samples with sodium sulfate.

11.2.2.1 Add 10-20g anhydrous sodium sulfate to each thimble containing the sample and mix thoroughly to evenly distribute the sodium sulfate. If the mixture is not free-flowing, continue to mix in sodium sulfate until a free-flowing consistency can be obtained.

11.2.2.2 Spike 1.0mL of the Labeled standard spiking solution at 10-20ng/mL (Table 3) into each sample and each QC aliquot. Record this addition on the bench sheet.

11.2.2.3 Spike 1.0mL of the Matrix standard spiking solution at 5ng/mL (Table 3) into the LCS/DLCS aliquots. Record this addition on the bench sheet. These will serve as both the precision and accuracy for the batch.

11.2.3.2 Rather than adding sample/spiking solution to a clean thimble, instead add sample to a clean beaker to record weight. Add 10-20g sodium sulfate and mix thoroughly to evenly distribute the sodium sulfate. If the mixture is not free-flowing, continue to mix in sodium sulfate until a free-flowing consistency can be obtained. Spike 1.0mL of the Labeled standard spiking solution at 10-20ng/mL (Table 3). Record the standard addition on the bench sheet. Transfer sample to ASE cell. Top with sodium sulfate.

11.2.4.1 Add 20-30g anhydrous sodium sulfate to each thimble containing the sample and mix thoroughly to evenly distribute the sodium sulfate. If the mixture is not free-flowing, continue to mix in sodium sulfate until a free-flowing consistency can be obtained.

11.2.4.2 Spike 1.0mL of the Labeled standard spiking solution at 10-20ng/mL (Table 3) into each sample and each QC aliquot. Record this addition on the bench sheet. If lipid determination is to be performed on a sample,

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spike 2.0mL of the Labeled standard spiking solution, instead.

11.2.4.3 Spike 1.0mL of the Matrix standard spiking solution at 5ng/mL (Table 3) into the LCS/DLCS aliquots. Record this addition on the bench sheet. These will serve as both the precision and accuracy for the batch.

Reason(s) for Change(s):
After some recent investigation, the EPA ORCR released a memo recommending that the recently updated language in some EPA 3500 series methods regarding standard addition should not be followed and that addition of labeled standards to solid samples should occur following mixture with sodium sulfate to minimize potential low percent recovery issues.

Change(s) Submitted by: Andrew Biddle	Date: 08/17/10

Approvals:

Technical Reviewer Signature:	Date:
QA PM Signature:	Date:
Department Supervisor/Manager Signature:	Date: 8/17/10

Change(s) Effective Date: 08/17/10

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STANDARD OPERATING PROCEDURE

CHLORINATED BIPHENYL CONGENERS IN WATER, SOIL, SEDIMENT, BIOSOLIDS AND TISSUE BY ISOTOPE DILUTION HRGC/HRMS

SOP Code: HMS-1668A

Revision: 4

Effective: 08/20/10

Approved by: _____ _____
Xiangqin 'Sam' Liang, Laboratory Director Date

_____ _____
Dr. Lan Le, Technical Director Date

_____ _____
Andrew Biddle, Quality Assurance Program Manager Date

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Annual review of this SOP has been performed; this SOP reflects current practice.
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER: _____
Initials: _____ Date: _____

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Standard Operating Procedure for

Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids and Tissue by Isotope Dilution HRGC/HRMS

1. SCOPE AND APPLICATION

1.1 This SOP is for determination of polychlorinated biphenyl congeners (PCBs) in water, soil, sediment, biosolids, tissue, and other sample matrices by high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) utilizing EPA Method 1668 Revision A.

1.2 The PCBs that can be determined by this SOP are the 12 polychlorinated biphenyls (PCBs) designated as toxic by the World Health Organization (WHO) plus the remaining 197 PCBs, approximately 125 of which are resolved adequately on an SPB-Octyl gas chromatographic column to be determined as individual congeners. The remaining approximately 70 congeners are determined as mixtures of isomers (co-elutions). The list of 209 PCBs is given in Table 1.

1.3 The 12 PCBs designated as toxic by WHO and the earliest and latest eluted congener at each chlorination level are determined by the isotope dilution quantitation technique; the remaining congeners are determined by the internal standard quantitation technique.

1.4 This SOP allows determination of the PCB toxicity equivalent (TEQ) for the toxics in a sample using toxicity equivalence factors (TEFs). This SOP also allows estimation of homolog totals by level of chlorination and estimation of total PCBs in a sample by summation of the concentrations of the PCB congeners and congener groups.

1.5 This SOP is for use in data gathering and monitoring associated with the Clean Water Act, the Resource Conservation and Recovery Act, the Comprehensive Environmental Response, Compensation and Liability Act, and the Safe Drinking Water Act. EPA Method 1668A is based on a compilation of methods from the technical literature and on EPA Method 1613.

1.6 The detection limits and quantitation levels in this procedure are usually dependent on the level of interferences and laboratory background levels rather than instrumental limitations. The estimated method detection limits are the levels at which the PCBs can be determined with laboratory contamination present.

1.7 EPA Method 1668A is "performance based." CAS Houston is permitted to modify the procedure to overcome interferences or lower the cost of measurements, provided that all performance criteria are met. The requirements for establishing procedural equivalency are given in Section 13.1 and CAS Houston's modifications are detailed in Section 21. Any modification of EPA Method 1668A, beyond those expressly permitted, shall be considered a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

2. METHOD SUMMARY

Flow charts that summarize procedures for sample preparation, extraction, and analysis are given in Figure 1 for aqueous and solid samples and Figure 2 for tissue samples, with additional modifications as mentioned in Section 21.

2.1 Extraction

2.1.1 Aqueous samples (samples containing less than one percent solids) – Stable isotopically labeled analogs of the toxics and labeled earliest and latest level of chlorination (LOC) PCBs are spiked into the sample aliquot. The sample is extracted using a modified separatory funnel extraction and concentrated for clean up.

2.1.2 Solid, semi-solid, and multi-phase samples (excluding tissue) – The labeled compounds are spiked into sample material that corresponds to approximately 5g on a dry weight basis. For samples with high moisture content, the amount of wet weight material to add the labeled compounds to would necessarily be greater than 5g. All samples are homogenized properly and extracted in a Soxhlet extraction apparatus or Accelerated Solvent Extractor (ASE). The extract is concentrated for clean up.

2.1.3 Fish and other tissue – An aliquot of tissue sufficient enough to provide sample for PCB and lipid analysis is homogenized and spiked with labeled compounds. The sample is mixed with anhydrous sodium sulfate and extracted for 16-24 hours using a 1:1 dichloromethane/hexane solution in a Soxhlet extraction apparatus. The extract is split; with one aliquot concentrated for clean up and the other concentrated for lipid determination (if applicable).

2.2 After extraction, a labeled clean up standard is spiked into the extract which is then cleaned up using back-extraction with sulfuric acid and/or base, and silica gel or Florisil chromatography.

2.3 After clean up, the extract is concentrated to 20µL. Immediately prior to injection, labeled injection internal standards are added to each extract and an aliquot of the extract is injected into the gas chromatograph (GC). The analytes are separated by the GC and detected by a high resolution (≥10,000) mass spectrometer. Two exact m/z's are monitored at each level of chlorination throughout a pre-determined retention time window.

2.4 An individual PCB congener is identified by comparing the GC retention time and ion-abundance ratio of two exact m/z's with the corresponding retention time of an authentic standard and the theoretical or acquired ion-abundance ratio of the two exact m/z's.

2.5 Quantitative analysis is performed in one of two ways using selected ion current profile (SICP) areas:

2.5.1 For the toxics and the earliest and latest level of chlorination CBs (Toxics 1,06) the GC/MS is multi-point calibrated and the concentration is determined using the isotope dilution technique.

2.5.2 For all other congeners, the GC/MS is calibrated at a single concentration and the concentrations are determined using the internal standard technique.

2.5.3 For the labeled congeners, the GC/MS is calibrated using replicates at a single concentration and the concentrations of these labeled compounds are determined using the internal standard technique.

2.6 The quality of analysis is assured through reproducible calibration and testing of the extraction, clean up and GC/MS systems.

3. DEFINITIONS

3.1 Abbreviations

MoCB	=	Monochlorinatedbiphenyl
DiCB	=	Dichlorinatedbiphenyl
TrCB	=	Trichlorinatedbiphenyl
TeCB	=	Tetrachlorinatedbiphenyl
PeCB	=	Pentachlorinatedbiphenyl
HxCB	=	Hexachlorinatedbiphenyl
HpCB	=	Heptachlorinatedbiphenyl
OcCB	=	Octachlorinatedbiphenyl
NoCB	=	Nonachlorinatedbiphenyl
DeCB	=	Decachlorinatedbiphenyl
PCB	=	Polychlorinatedbiphenyl
PFK	=	Perfluorokerosene
CS	=	Clean up standard
LS	=	Labeled standard
IS	=	Internal injection standard
HRGC	=	High-resolution gas chromatography
HRMS	=	High-resolution mass spectrometry
TEF	=	Toxicity equivalence factor
TEQ	=	Toxicity equivalent

3.2 **Polychlorinatedbiphenyls (PCBs)**– compounds that contain from 1 to 10 chlorine atoms, resulting in a total of 209 compounds determined by this procedure. The 209 PCBs are listed in Table 1.

3.3 **Isomer** – compounds having the same number and type of chlorine atoms, but substituted in different positions.

3.4 **Labeled Standard** – an isotopically labeled analog that is added to all samples, including method blanks and quality control samples, before extraction. They are used along with response factors to measure the concentrations of the congeners. The labeled Toxics/LOC standard concentrations are listed in Table 3.

3.5 **Calibration Solutions** – solutions containing known amounts of unlabeled PCBs and labeled standards, listed in Table 5. The set of 5 solutions is used to determine the instrument response of the unlabeled analytes relative to the C13-labeled standards. A sixth calibration solution contains all 209 congeners and is used for calibration of all compounds not contained in the 5 point curve.

3.6 **Internal Standard Solution** – a solution containing the internal injection standards that are added to the extract after final concentration for HRGC/HRMS analysis to determine the recovery efficiencies achieved for the C13-labeled standards. Concentrations are listed in Table 3.

3.7 **Matrix Spike Solution** – a solution of native (unlabeled) Toxics/LOC PCBs that are spiked into the laboratory control samples prior to extraction to monitor method performance. Concentrations are listed in Table 3.

3.8 **Clean Up Standard Solution** – a solution containing C13-labeled compounds that is spiked to the extract prior to clean up to measure the efficiency of the clean up procedure. Concentrations are listed in Table 3.

3.9 **Method Blank (MB)** – represents the background contributions from glassware, extraction and clean up solvents. An MB is spiked with a solution of C13-labeled standards, extracted, cleaned up, and analyzed by HRGC/HRMS in exactly the same manner as the test samples.

3.10 **Calibration Verification Standard (VER)** – The mid-point calibration standard (CS-3) that is used to verify calibration (Table 3).

3.11 **Estimated Detection Limit (EDL)** – The sample specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level.

4. INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts, elevated baselines, and/or lock-mass suppression causing misinterpretation of chromatograms. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Where possible, reagents are cleaned by extraction or solvent rinse. Environmentally abundant PCBs have been shown to be very difficult to completely eliminate from the laboratory at levels lower than the EDLs.

4.2 The use of high purity reagents and solvents can be used to minimize interference problems. Solvent lots are tested for the absence of PCBs prior to use by the laboratory. Disposable glassware is used whenever possible. Reusable glassware must be cleaned according to the procedures in the SOP for *Washing Glassware*, ***SMO-WASH***

4.3 Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the PCBs. The most frequently encountered interferences are chlorinated dioxins and dibenzofurans, methoxy biphenyls, hydroxydiphenyl ethers, benzylphenyl ethers, brominated diphenyl ethers, polynuclear aromatics, polychlorinated naphthalenes, and pesticides. Because very low levels of PCBs are measured by this procedure, the elimination of interferences is essential. The clean up steps given in Section 11.3 can be used to reduce or eliminate these interferences and thereby permit reliable determination of the PCBs at the levels indicated in Table 2.

4.4 A high resolution capillary column (SPB-Octyl) is used for this procedure; however, no known single column can resolve all isomers. Many compounds coelute with other compounds of similar m/z's. Determination of which compounds coelute is performed by the injection of the calibration standard containing all 209 congeners. Confirmation can be performed on an alternate column (DB-1); however, this process is not undertaken commonly by CAS/Houston and is only available to be performed by request from the client.

5. SAFETY

5.1 The toxicity or carcinogenicity of each chemical used in this procedure has not been precisely determined; however, each compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level.

5.1.1 PCBs have been tentatively classified as known or suspected human or mammalian carcinogens. On the basis of the available toxicological and physical properties of the PCBs, pure standards should be handled only by highly trained personnel thoroughly familiar with handling and cautionary procedures and the associated risks.

5.1.2 It is recommended that the laboratory purchase dilute standard solutions of the analytes in this procedure. However, if primary solutions are prepared, they must be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator must be worn when high concentrations are handled.

5.2 The laboratory will ensure that all analysts receive adequate safety training prior to working with the chemicals and compounds associated with this method.

5.3 The solvents and acids in use in the laboratory may be hazardous and should be treated as such. See the *Environmental Health and Safety Manual*, ***CHP*** (Chemical Hygiene Plan), Section 5, for more information.

5.4 Disposable gloves, apron or lab coat, safety glasses or mask, and a fume hood must be used. During analytical operations that may give rise to aerosols or dusts, personnel should wear respirators equipped with activated carbon filters. Eye protection must be worn while working with exposed samples or pure analytical standards. Latex gloves are commonly used to reduce exposure of the hands.

5.5 **Waste Handling** – Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans. Janitors and other personnel should be trained in the safe handling of waste.

5.6 Low-level contamination is always a possibility for HRGC/HRMS analysis due to the chemical properties of dioxins/furans and the low resolution of the instruments. To eliminate this contamination, it may be necessary to install activated carbon filters and perform hexane wipes on counters and hood surfaces used for sample processing.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1 The client shall collect aqueous samples in amber glass containers and collect solid samples in wide mouth containers following conventional sampling practices.

6.2 Aqueous Samples

6.2.1 To remove any residual chlorine in aqueous samples, add approximately 80mg sodium thiosulfate per liter of water. To verify its absence, measure the chlorine presence with an indicator strip accurate down to 0.05ppm, or 0.05mg/L. Repeat process until chlorine is completely removed.

6.2.2 The client shall ensure aqueous samples are maintained in the dark at <6°C from the time of collection until receipt at the laboratory. If the sample will be frozen, allow room for expansion. Store in the dark at <6°C.

6.3 **Solid, Mixed-phase, Semi-solid, and Oil Samples, Excluding Tissue** – The client shall ensure solid, semi-solid, oily, and mixed-phase samples are maintained in the dark at <6°C from the time of collection until receipt at the laboratory. Store solid, semi-solid, oily, and mixed-phase samples in the dark at <-10°C.

6.4 Fish and Other Tissue Samples

6.4.1 Fish may be cleaned, filleted, or processed in other ways in the field, such that the laboratory may expect to receive whole fish, fish fillets, or other tissues for analysis.

6.4.2 Collect fish, wrap in aluminum foil, and maintain at <0°C from the time of collection until receipt at the laboratory, to a maximum time of 24 hours. If a longer transport time is necessary, freeze the sample. Ideally, fish should be frozen upon collection and shipped to the laboratory under dry ice.

6.4.3 Freeze tissue samples upon receipt at the laboratory and maintain in the dark at -20 to -10°C until prepared.

6.5 Holding Times

6.5.1 There are no demonstrated maximum holding times associated with the PCBs in aqueous, solid, semi-solid, tissue, or other sample matrices. If stored in the dark at 0-6°C, aqueous samples may be stored for up to one year. Similarly, if stored in the dark at -20 to -10°C, solid, semi-solid, multi-phase, and tissue samples may be stored for up to one year.

6.5.2 Store sample extracts in the dark at 0-6°C until analyzed. If extracts are stored at 0-6°C, sample extracts may be stored for up to one year

6.6 Samples are usually shipped from the client to the lab by a standard shipping service such as UPS, Federal Express, DHL, or USPS.

6.7 Samples are logged in to CAS LIMS and labeled for tracking through the laboratory. A cooler receipt form is filled out, cataloging any non-conformances associated with the receipt of the sample containers. See the SOP for *Sample Receiving, **SMO-WET,*** for a more detailed explanation.

6.8 Before extraction, a visual inspection of a sample is done. Aqueous samples deemed to have >1% solids are filtered unless otherwise specified by the client. Solid samples are homogenized thoroughly prior to aliquotting. See the SOP for *Subsampling and Compositing Aqueous and Soil Samples, **WET-SSMP***, for more information.

7. APPARATUS AND EQUIPMENT

7.1 Gas Chromatograph/High-Resolution Mass Spectrometer/Data System (GC/HRMS/DS) - The GC must be equipped for temperature programming. All required accessories must be available, such as syringes, gases, and capillary columns.

7.1.1 GC Injection Port - The GC injection port must be designed for capillary columns. The use of splitless injection techniques is recommended. On column 1µL injections can be used on the 30m SPB-Octyl column. The use of a moving needle injection port is also acceptable. The use of a 2µL injection is acceptable; however, analysts must remain consistent throughout the analyses by using the same injection volume at all times.

7.1.2 Gas Chromatograph/Mass Spectrometer (GC/MS) Interface – The GC/MS interface components should withstand 350°C. Cold spots or active surfaces (adsorption sites) in the GC/MS interface can cause peak tailing and peak broadening. It is recommended that the GC column be fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam.

7.1.3 Mass Spectrometer – 28-40eV electron impact ionization, must be capable of selectively monitoring a minimum of 22 exact m/z's at high resolution (≥10,000) during a period less than 1.5 seconds, and must meet all of the performance specifications in Section 12.1.2.

7.1.4 Data System - A dedicated data system is used to control the rapid multiple-ion monitoring process and to acquire the data. Quantitation data (peak areas or peak heights) and selected ion monitoring (SIM) displays of intensities of each ion signal being monitored, including the lock-mass ion as a function of time, must be acquired during the analyses and stored. Quantitations are reported based upon computer generated peak areas. It is also recommended to have a data system capable of switching to different sets of ions (descriptors) at specified times during the GC/HRMS acquisition. The data system can provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It can also acquire mass spectral peak profiles and provide hard copies of peak profiles to demonstrate the required resolving power. The data system can permit the measurement of noise at the base line.

7.1.5 Micromass MassLynx (Version 4.1) is used to obtain all data from the HRMS system. CAS/Houston employs the following HRGC/HRMS systems:

7.1.5.1 An Agilent 6890N GC with a CTC A200S Autosampler interfaced to a Micromass Autospec Ultima HRMS.

7.1.5.2 An Agilent 6890N GC with a CTC A200S Autosampler interfaced to a Micromass Autospec Ultima HRMS.

7.1.5.3 An Agilent 7890A GC with a CTC A200S Autosampler interfaced to a Micromass Autospec Premier HRMS

7.1.5.4 An Agilent 7890A GC with a CTC A200S Autosampler interfaced to a Micromass Autospec Premier HRMS

7.2 GC Columns

7.2.1 Isomers may be unresolved so long as they have the same TEF and response factor and so long as these unresolved isomers are uniquely resolved from all other congeners. For example, the SPB-Octyl column achieves unique GC resolution of all toxics except congeners with congener numbers 156 and 157. This isomeric pair is uniquely resolved from all other congeners and these congeners have the same TEF and response factor.

7.2.2 CAS/Houston uses the SPB-Octyl column (Supelco, 24218-U).

7.2.2.1 The retention time for decachlorobiphenyl (DeCB, PCB 209) must be greater than 55 minutes.

7.2.2.2 The column must uniquely resolve congeners 34 from 23 and 187 from 182, and congeners 156 and 157 must co-elute within 2 seconds at the peak maximum. Unique resolution means a valley height less than 40% of the shorter of the two peaks that result when the diluted combined 209 congener solution is analyzed. See Figures 3 and 4.

7.2.2.3 The column must be replaced when any of the criteria in 7.2.1-7.2.2.2 are not met.

NOTE: The SPB-Octyl column is subject to rapid degradation when exposed to oxygen. The analyst should exclude oxygen from the carrier gas, should eliminate air leaks, and should cool the injector, column, and transfer line before opening the column to the atmosphere. For further information on precluding oxidation, contact the column manufacturer.

7.3 **Soxhlet/ASE Apparatus** – The laboratory will be equipped with a macro and/or micro soxhlet apparatus. CAS/Houston uses a series of four (4) Combo Mantles (Glas-Col 100D RJ30012, with Glas-Col, 104A RL3612 controllers) and a series of twenty-four (24) individual mantles (Glas-Col, TM106, with Glas-Col 104A PL120 Powr Trol and PL-312 and PL-3122 MiniTrol controllers) for the soxhlet apparatus. The combination mantles are designed for 250mL flasks (24/40, VWR, 89000-330), soxhlets (24/40 and 45/50, HGF Scientific), and condensers (45/50, HGF Scientific). The individual mantles are designed for 500mL flasks (24/40, VWR, 89000-334), soxhlets (24/40 and 55/50, HGF Scientific), and condensers (55/50, HGF Scientific). The condensers are connected to a chiller capable of maintaining a chilled temperature to ensure proper solvent reflux. Additionally, if an ASE apparatus (Dionex, ASE200) is to be employed, it must be capable of being pre-programmed to allow for automatic sample extraction at temperature and pressure settings unique to the extraction solvent. Other EPA approved extractors may be used instead.

7.4 **Miscellaneous Equipment and Materials** - The following list of items does not necessarily constitute an exhaustive compendium of the equipment needed for this analytical method.

7.4.1 Balances capable of accurately weighing to 0.001g (Denver, XE Series 300, Mettler, AJ100, and Mettler Toledo, PG603-S)
7.4.2 Centrifuge (Clay Adams Dynac, 0101)
7.4.3 Drying oven (VWR Utility Oven, Model 1305U and Blue M Electric, C4855Q)
7.4.4 250mL polypropylene beaker (Fisher, 01-291-5)
7.4.5 20mL scintillation vials (VWR, 66022-004)
7.4.6 Laboratory fume hoods
7.4.7 Pipets, disposable, serological, 10mL (Fisher, 13-676-34D)
7.4.8 Pipet tips, 2-200uL and 10-1000uL (Eppendorf, 022492039 and 022492055 respectively)
7.4.9 Commercial food preparation machine (Hobart, HCM62)
7.4.10 PTFE boiling chips (VWR, 36397-103)

NOTE: Teflon boiling chips may float in methylene chloride, may not work in the presence of any water phase, and may be penetrated by nonpolar organic compounds.

7.4.11 Glass fiber filters, Whatman filter grade (varying sizes and particle retention)
7.4.12 2-liter screw top clear jars (C&G Containers, LFW0012000)
7.4.13 Rotary evaporator with a temperature controlled water bath (Buchi, Rotavapor R-200 with Heating Bath B-490 and Rotavapor 201 with Heating Bath B-491)
7.4.14 Glass wool (EMD, EM-GX0090-2)
7.4.15 Jars, clear glass, 250mL, with Teflon lined screw caps (C&G LFA0082000)
7.4.16 Auto sampler vials with 150µL inserts (Restek, 24653)
7.4.17 Glass columns – 20mm diameter x 12in long with one tapered end (HGI Scientific specialty product)
7.4.18 Shaker (for 2L jar, Eberbach Model 6000)
7.4.19 GC Gooseneck Splitless Liners, 2mm inner diameter (Restek, 20797)
7.4.20 Thermogreen™ LB-2 11mm Septa (Supelco, 20654)
7.4.21 Autosampler Vial Caps, Seal with PTFE Liner, 11mm (Supelco, 27102-U)
7.4.22 Carrier Gas, Ultra High Purity Helium (Airgas)
7.4.23 Perfluorokerosene (PFK) Low-Boil (SynQuest, PN 1200-2-22 1g)
7.4.24 Tumbler
7.4.25 Ultrasonicator (Branson Ultrasonic Cleaner, 5510R-MT)
7.4.26 Supelex™ M-2A Ferrules 0.4mm ID (Supelco, 22474)
7.4.27 Nitrogen Evaporator with a temperature controlled water bath (Zaimtek, ZipVap #109A Custom)
7.4.28 Muffle Furnace (Blue-M/Lindberg, LGO Element Box Furnace, BF51842-P-BC)

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1 All standards, reagents, and consumable materials must be logged in and numbered when delivered. Details of all dilutions of standards must also be entered into the PCB Standards Logbook. Store standards in the dark at <6°C.

8.2 **Organic-free reagent water** – All references to water in this SOP refer to organic-free reagent water. CAS/Houston uses the water supplied from our bottled water supplier as the source of the organic-free reagent water used in the preparation of quality control samples and standards.

8.3 **Silica gel, high purity grade, type 60, 70-230 mesh (JT Baker, JT3405-05)** – Activate overnight at a minimum temperature of 120°C. Store in a glass bottle sealed with a Teflon lined screw cap.

8.4 **Silica gel impregnated with Sodium Hydroxide** – Add one part (by weight) of 1M NaOH solution to two parts (by weight) activated silica gel in a Teflon lined screw cap bottle and mix with a glass rod until free of lumps.

8.5 **Silica gel impregnated with Sulfuric Acid** – Add one part (by weight) concentrated sulfuric acid to two parts (by weight) activated silica gel in a Teflon lined screw cap bottle and mix with a glass rod until free of lumps.

8.6 **Extraction thimbles, 43mm x 123mm and 33mm x 80mm (VWR, 28320-510 and 27730-128, respectively)**

8.7 **Sulfuric Acid, concentrated, ACS grade (EMD, EM-SX1244-75)**

8.8 **Sand, quartz (VWR, BDH0274-2.5kg)**

8.9 **Sodium Sulfate, granular, anhydrous (EMD, EM-SX0760E-20)** – Activate for a minimum of one hour at a temperature of 400°C. Store in a glass bottle sealed with a Teflon lined screw cap.

8.9.1 Fill ceramic crucibles with sodium sulfate and place in the muffle furnace. Add 5-10g sand to an empty crucible and place in the muffle furnace.

8.9.2 The temperature program is set to ramp up to 400°C then hold that temperature for four hours. Start the program by holding the "**P**" button until "**Pnr 4**" appears on the controller. Press "**Run/Hold**." To cancel a program, press the up and down arrows simultaneously. At the end of the program the furnace will cool by convection.

8.9.3 When the sodium sulfate is ready to remove, transfer it to 2-liter jars and label them with the preparation date, analyst name, and lot number of the sodium sulfate. Make an entry into the Semi-Volatile Na_2SO_4 Preparation Logbook containing the preparation date, analyst name, quantity prepared, sodium sulfate lot number, and whether the run passed or failed (as indicated by a color change in the sand from gray to pink/orange).

8.9.4 If the sodium sulfate has a noticeable grayish cast, it must be discarded.

8.10 **Sodium Chloride, analytical reagent grade, 5 percent (w/v) in water (VWR, VW6430-5)**

8.11 **Sodium Hydroxide, 1N (VWR, 3222-4)**

8.12 **Florisil, 60-100 mesh (Sigma-Aldrich, 220744-250G)**

8.13 **Solvents**

8.13.1 Methylene Chloride – 99.9% minimum Assay (HPLC Grade, EMD, TXEMDX0831-1CA)
8.13.2 Hexane – 99.9% minimum Assay (HPLC Grade, EMD, TXEMH0296-1CA)
8.13.3 Nonane – 99.9% minimum Assay (HPLC Grade, Sigma, N29406-500mL)
8.13.4 Toluene – 99.9% minimum Assay (HPLC Grade, EMD, TX0737-1)

8.13.5 Acetone – 99.9% minimum Assay (HPLC Grade, EMD, AX0116-1)

8.14 Standards Suppliers

8.14.1 Cambridge Isotope Laboratories (CIL) Primary standards supplier

8.14.2 Wellington Laboratories Secondary standards supplier

8.15 Purchase, Receipt, and Handling of Standards

8.15.1 All standards purchased from CAS/Houston must have an appropriate purchase request form filled out and have approval from the Laboratory Director or other qualified individual. They must then go through the CAS/Corporate Purchasing Department. Standards are considered consumable and do not need an Expenditure Authorization Request (EAR) even if the cost exceeds the threshold. See the SOP for *Purchasing and Approval of Vendors*, ***ADM-PUR***, for more information.

8.15.2 Upon arrival, all packages containing high concentrations must be checked for damage. If any damage is present, consult with CAS/Houston Environmental Health and Safety Officer before further steps are taken. Proper safety practices should be followed when handling received standards.

8.15.3 All received standards must be assigned a PCB Standards Logbook identification number. Copy the solution description from the certificate of analysis and attach to the space provided for the next entry in the logbook. Include the received date in the logbook. The following format is applied: B(PCB)[Logbook number]-[page number]-[entry number]. An example would be B3-20-1.

8.15.4 Assign each standard an expiration date as noted by the supplier. This is usually ten years after the manufacture date. Record this in the standards logbook and on the standard vial. A standard must be disposed of after this date unless its validity can be verified (Section 8.23).

8.15.5 Record the identification number on the received vial label. Transfer the standard from the received ampoule to a 2mL amber vial, keeping the supplier label intact. Seal the vial with Teflon tape. This will ensure no deterioration will occur.

8.15.6 Record the identification number on the certificate of analysis received from the supplier and store this documentation in the "1668 Standards" binder for future reference. Certificates of analysis are also available through the Cerilliant website, CIL's manufacturer.

8.15.7 All solutions must be adequately labeled with the logbook identification code, method, short solution description, preparation date, initials of person performing preparation, expiration date, volume and concentration. Logbook entries shall include all of the above with the addition of the list of components, the

components' logbook identification code, description and concentration, plus the initials of the person witnessing the dilution steps.

8.16 **High-Resolution Concentration Calibration Solutions** – Five nonane solutions containing unlabeled and carbon-labeled PCBs at known concentrations are used to calibrate the instrument. A sixth solution containing all 209 congeners is used to calibrate the instrument for the compounds other than the toxics and earliest and latest eluting congeners in a chlorination level. The concentration ranges are homologue dependent.

8.17 **Labeled Standard Solution** – The purchased Labeled Toxics/LOC window-defining stock solution is diluted once with Nonane and again with Acetone to make the labeled standard spiking solution (Table 3). Store at ≤6 °C in the dark. All standards are assigned a one (1) year expiration date, not to exceed the latest expiration date of any parent solution. All standards in Acetone are made daily.

8.18 **Internal Standard Solution** – The purchased labeled stock solution is diluted to make a labeled internal standard stock solution. Perform an additional dilution on the stock solution to make an internal injection standard working solution (Table 3). Store at ≤6°C in the dark. All standards are assigned a one (1) year expiration date, not to exceed the latest expiration date of any parent solution.

8.19 **Clean Up Standard Solution** – The purchased labeled clean up stock solution is used to make the clean up standard stock solution. Perform an additional dilution on the stock solution to make a labeled clean up standard working solution (Table 3). Store at ≤6°C in the dark. All standards are assigned a one (1) year expiration date, not to exceed the latest expiration date of any parent solution.

8.20 **Matrix Spiking Solution (target compounds)** – The purchased Native Toxics/LOC stock solution is used to make the matrix standard stock solution. Perform an additional dilution on the stock solution to make a matrix standard working solution (Table 3). Store at ≤6°C in the dark. All standards are assigned a one (1) year expiration date, not to exceed the latest expiration date of any parent solution. All standards in Acetone are made daily.

8.21 Reference Standards are to be purchased from a supplier other than the primary standard supplier. All reference standards must be segregated from primary standards during storage. Reference standards can only be used to verify primary standards and may not be used for samples. All reference standards must be documented in the standards logbook following 8.15.3 – 8.15.7.

8.22 Research standards must be adequately labeled as such. In addition to Sec. 8.15.7, all labels for standards designated for research must also state specifically the intended research to be done, along with an appropriate qualifier, such as "For research purposes only."

8.23 Expiration dates can be extended under the conditions outlined in the CAS/Corporate *Policy for Standards and Reagents Expiration Dates*, September 2009.

9. PREVENTIVE MAINTENANCE

9.1 All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2 Inline purifiers or scrubbers should be in place for all sources of carrier gas. The purifiers are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3 Gas Chromatograph

9.3.1 Whenever GC maintenance is performed, care must be taken to minimize the introduction of air or oxygen into the column. Injection ports should be maintained by changing the injection port liner, seal, washer, o-ring, septum, column ferrule and auto sampler syringe as necessary. Liners and seals should be changed when there is a problem with chromatographic performance.

9.3.2 Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and without fragmentation by using the proper column cutting tool.

9.3.3 Over time, as contaminated samples are analyzed, the column will exhibit poorer performance. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when poor column performance occurs in conjunction with continuing calibration failures (Section 12.4)

9.4 Mass Spectrometer

9.4.1 Tune the MS as needed to result in consistent and acceptable performance.

9.4.2 The source should be cleaned, as needed, depending on the performance of the instrument.

9.5 Preventive maintenance is performed annually by the service engineer for instruments under a service contract.

10. RESPONSIBILITIES

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

11. PROCEDURE FOR SAMPLE PREPARATION, EXTRACTION, CONCENTRATION AND CLEAN UP

11.1 Sample Preparation

11.1.1 Sample preparation involves modifying the physical form of the sample so that the PCBs can be extracted efficiently. In general, the samples must be in a liquid form or in the form of finely divided solids in order for efficient extractions to take place. Table 9 lists the phases and suggested quantities for extraction of various sample matrices.

For samples known or expected to contain high levels of PCBs, the smallest sample size representative of the entire sample should be used. For all samples, the blank, Internal Precision and Recovery (IPR), and Ongoing Precision and Recovery (OPR, or LCS, Laboratory Control Sample) aliquots must be processed through the same steps as the sample to check for contamination and losses in the preparation process.

11.1.2 Aqueous Sample Preparation – Preparation of aqueous samples containing one percent suspended solids or less.

11.1.2.1 Aqueous samples containing one percent suspended solids or less are prepared using the procedure below and extracted using the extraction techniques in Section 11.2.1. This procedure is applicable for samples and QC aliquots.

11.1.2.2 Transfer the sample to the ½ gal. extraction vessel. Mark the original level of the sample on the sample container for reference.

11.1.2.3 For each batch of up to 20 samples to be extracted in the same shift place three 1L aliquots of reagent water into clean ½ gal. extraction vessels. One of these will serve as the Method Blank, and the other two will serve as the Laboratory Control Sample Duplicate Laboratory Control Sample (LCS/DLCS).

11.1.2.4 If filtration of aqueous samples is required, connect a porcelain Buchner funnel to the ½ gal. extraction vessel and affix an appropriate glass fiber filter to the funnel. Allow sample to pass through filter under a gravity flow. If the suspended solids content is too great to filter through the filter, centrifuge the sample, decant, and then filter the aqueous phase. Extract the filter following Section 11.2.4.4-5 (hexane/dichloromethane obtains higher recoveries than toluene) and combine with the aqueous extraction extract prior to clean up.

11.1.3 Solid Sample Preparation – Preparation of samples containing greater than one percent solids.

11.1.3.1 Solid samples containing one percent suspended solids or more are prepared using the procedure below and extracted using the extraction techniques in Sections 11.2.2 or 11.2.3. This procedure is applicable for samples and QC aliquots.

11.1.3.2 Weigh a well-mixed aliquot of each sample sufficient to provide 5g of dry solids into a clean thimble.

11.1.3.3 For each batch of up to 20 samples to be extracted in the same shift, weigh three 5g aliquots of the reagent grade quartz sand (or sodium sulfate, if no sand is available) into clean thimbles. One of these will serve as the Method Blank, and the other two will serve as the Laboratory Control Sample/Duplicate Laboratory Control Sample (LCS/DLCS).

11.1.3.4 Total Solids Determination. Record the weight of an empty, labeled scintillation vial on the bench sheet. Add 5-10g (wet weight) of the solid sample to the scintillation vial and record the new, combined weight. Place the vial in an oven at 110±5°C for a minimum of 24 hours. Finally, record the dry weight of the combination on the bench sheet Calculate the percent solids using the following formula:

$$\%solids = \frac{w_a}{w_b} \times 100$$

Where: w_a = combined weight after drying, in g, minus the empty vial weight.
w_b = combined weight before drying, in g, minus the empty vial weight.

11.1.4 <u>Fish and Other Tissues</u> – Most tissue samples are received previously homogenized. If a sample is received that is not, however, the portion of the sample to be used for PCB determination must be established and the following procedure used for homogenization.

11.1.4.1 Tissue samples are prepared using the procedure below and extracted using the extraction techniques in Section 11.2.4. This procedure is applicable for samples and QC aliquots

11.1.4.2 A minimum of 20g of tissue must be homogenized to ensure there is enough sample for PCB and lipid determination (with enough left over in case re-extraction is necessary). Additionally, the weight should be increased for samples requiring additional analyses.

11.1.4.3 Homogenize the sample according to the procedures in the SOP for *Tissue Preparation*, ***WET-TISP***. Transfer to a new, labeled sample

container with a Teflon-lined cap. Store at -20° to -10°C and in the dark until analysis.

11.1.4.4 Weigh approximately 5g (10g if lipids analysis is required) of the homogenized tissue into a clean thimble.

11.1.4.5 For each batch of up to 20 samples to be extracted in the same shift, weigh three 5g aliquots of contaminant free tissue or corn oil into clean thimbles. One of these will serve as the Method Blank, and the other two will serve as the Laboratory Control Sample/Duplicate Laboratory Control Sample (LCS/DLCS).

To ensure the material used for QC samples is free of target compounds, homogenize and extract the material following the steps in Sections 11.1.1.1-11.1.4.3 and 11.2.4. Analyze the extracts on the HRGC/HRMS machines to verify no PCBs are present. Once determined, the blank material can be used for QC samples. If contamination is present, however, the entire lot of material must be properly disposed of and another lot obtained.

11.2 Extraction and Concentration

NOTE: The standard solution concentrations discussed below represent the amount added to samples where the default final volume is to be 100µL. If the final volume of the extract is to be 20µL (to achieve better data quality objectives), all spiking solution concentrations must be reduced by a factor of five (5).

11.2.1 Extraction of Aqueous Samples - Aqueous samples are to be extracted using a modified separatory funnel procedure. The main difference is that the vessel is a glass jar with a Teflon-lined cap that is disposable. This greatly reduces cross-contamination.

11.2.1.1 Spike 1.0mL of the Labeled standard spiking solution at 10-20ng/mL (Table 3) into each sample and each QC aliquot. Record this addition on the bench sheet.

11.2.1.2 Spike 1.0mL of the Matrix standard spiking solution at 5ng/mL (Table 3) into the LCS/DLCS aliquots. Record this addition on the bench sheet. These will serve as both the precision and accuracy for the batch.

11.2.1.3 Add 100mL dichloromethane to the spiked sample in the ½ gal. extraction vessel. Vent the vapors under a fume hood by shaking for a few seconds then unsealing the cap. Repeat this process until pressure no longer builds in the vessel. Extract the sample by shaking the jar manually or on an automatic shaker for 3 minutes. Allow the layers to separate for a minimum of 10 minutes and pipet the dichloromethane layer (bottom) into an appropriately labeled 250mL jar. If an emulsion

layer forms, employ mechanical techniques (ultrasonic bath, centrifugation) to complete the phase separation.

11.2.1.4 Extract the sample with an additional 50mL aliquot of dichloromethane. Following extraction, remove any excess water from the dichloromethane portion by swirling the sample extract and removing the water that collects on top.

11.2.1.5 Concentrate the extract using a rotary evaporator (with heating bath at 45-50°C) or nitrogen evaporator to a volume of approximately 15mL. A toluene keeper may be added at the option of the analyst. Concentration techniques must be precise and the analyst must not to let the solvent level get too low or else risk the loss of the lighter CB compounds.

11.2.1.6 Sample Volume Determination. Following extraction, using tap water, refill the sample container to the line marked in Section 11.1.2.2. Measure the volume of this water using a calibrated graduated cylinder and record on the extraction bench sheet.

11.2.2 Soxhlet Extraction of Solid Samples - Solid samples are to be extracted using either a soxhlet extraction apparatus or an Accelerated Solvent Extractor (ASE). Choosing which apparatus to use is at the discretion of the analyst; however, the ASE should only be used for low percent solids samples.

11.2.2.1 Spike 1.0mL of the Labeled standard spiking solution at 10-20ng/mL (Table 3) into each sample and each QC aliquot. Record this addition on the bench sheet.

11.2.2.2 Spike 1.0mL of the Matrix standard spiking solution at 5ng/mL (Table 3) into the LCS/DLCS aliquots. Record this addition on the bench sheet. These will serve as both the precision and accuracy for the batch.

11.2.2.3 Add 10-20g anhydrous sodium sulfate to each thimble containing the sample and mix thoroughly to evenly distribute the sodium sulfate. If the mixture is not free-flowing, continue to mix in sodium sulfate until a free-flowing consistency can be obtained.

11.2.2.4 Transfer thimble to soxhlet extractor fitted with a 500mL boiling flask containing 240mL Toluene and boiling chips. Alternatively, the micro-soxhlet apparatus utilizes a smaller set up, thus requiring only 125mL Toluene and boiling chips to be added to a 250mL boiling flask.

11.2.2.5 Place the soxhlet apparatus onto a boiling mantle/condenser combination and reflux overnight (16-24 hours). Solvent must cycle 5 times per hour minimum (controller set at "6" or "60%").

11.2.2.6 Cool the extraction apparatus. Concentrate the extract using a rotary evaporator (with heating bath at 45-50°C), nitrogen evaporator, or by air drying under the fume hood to a volume of approximately 15mL. No keeper solvent is to be added. Concentration techniques must be precise and the analyst should be sure not to let the solvent level get too low or else risk the loss of the lighter CB compounds.

11.2.3 ASE Extraction of Solid Samples – Solid samples are to be extracted using either a soxhlet extraction apparatus or an Accelerated Solvent Extractor (ASE). Choosing which apparatus to use is at the discretion of the analyst; however, the ASE should only be used for low percent solids samples.

CAS/Houston uses a Dionex ASE200 Extractor. Other EPA approved extractors may be used instead.

11.2.3.1 Label stainless steel ASE cells accordingly. Add a frit to each cell and cover with a small amount of sodium sulfate.

11.2.3.2 Rather than adding sample/spiking solution to a clean thimble, instead add sample to a clean beaker to record weight. Add 10-20g sodium sulfate, spike 1.0mL of the Labeled standard spiking solution at 10-20ng/mL (Table 3), and mix thoroughly to evenly distribute the sodium sulfate. Record the standard addition on the bench sheet. If the mixture is not free-flowing, continue to mix in sodium sulfate until a free-flowing consistency can be obtained. Transfer sample to ASE cell. Top with sodium sulfate.

11.2.3.3 Label 60mL collection vials and position both the cell and 60mL collection vial in corresponding locations on the trays. Double check to ensure all cell positions match their respective vial positions.

11.2.3.4 Check Toluene reservoir and fill if low. Check Nitrogen level. If low, replace tank. Use safety precautions when handling the gas tanks.

11.2.3.5 To start the ASE: Press "Menu," "Load Method," "Number 1," and "Start" on the screen. All methods and schedules are pre-programmed based on Dionex recommendations for the solvents used, and can be found in the appropriate maintenance logbook.

11.2.3.6 Concentrate the extract using a rotary evaporator (with heating bath at 45-50°C) or nitrogen evaporator to a volume of approximately 15mL. No keeper solvent is to be added. Concentration techniques must be precise and the analyst should be sure not to let the solvent level get too low or else risk the loss of the lighter PCB compounds.

11.2.4 Soxhlet Extraction of Tissue Samples - Tissue samples are to be extracted using a soxhlet extraction apparatus.

11.2.4.1 Spike 1.0mL of the Labeled standard spiking solution at 10-20ng/mL (Table 3) into each sample and each QC aliquot. Record this addition on the bench sheet. If lipid determination is to be performed on a sample, spike 2.0mL of the Labeled standard spiking solution, instead.

11.2.4.2 Spike 1.0mL of the Matrix standard spiking solution at 5ng/mL (Table 3) into the LCS/DLCS aliquots. Record this addition on the bench sheet. These will serve as both the precision and accuracy for the batch.

11.2.4.3 Add 20-30g anhydrous sodium sulfate to each thimble containing the sample and mix thoroughly to evenly distribute the sodium sulfate. If the mixture is not free-flowing, continue to mix in sodium sulfate until a free-flowing consistency can be obtained.

11.2.4.4 Transfer thimble to soxhlet extractor fitted with a 500mL boiling flask containing boiling chips and 300mL of a 1:1 mixture of hexane/dichloromethane. Alternatively, the micro-soxhlet apparatus utilizes a smaller set up, thus requiring only 150mL of the 1:1 mixture to be added to a 250mL boiling flask.

11.2.4.5 Place the soxhlet apparatus onto a boiling mantle/condenser combination and reflux overnight (16-24 hours). Solvent must cycle 5 times per hour minimum (controller set at "4" or "40%").

11.2.4.6 Cool the extraction apparatus. If lipid determination is to be performed, follow these steps, else, move on to Section 11.2.4.7. Weigh one 250mL jar and record on the bench sheet. Split the extract into two 250mL jars (with the extract for lipids determination added to the pre-weighed jar). Be sure to split the extracts evenly so as to ensure one half of the final extract is in each container. Dry the extract in the pre-weighed jar to complete dryness and record the weight on the bench sheet. Calculate the percent lipids using the following equation:

$$\%lipid = \frac{w_r}{w_t} \times 100$$

Where: w_r = weight of residue, in g, measured as the difference in the jar prior to adding the sample and following concentration.
w_t = weight of tissue, in g.

The un-weighed jar and extract is to be used for target compound determination and is to be concentrated following the procedure in Section 11.2.4.7 below.

11.2.4.7 Concentrate the extract using a rotary evaporator (with heating bath at 45-50°C) or nitrogen evaporator to a volume of approximately 15mL. A toluene keeper may be added at the option of the analyst. Concentration techniques must be precise and the analyst should be sure not to let the solvent level get too low or else risk the loss of the lighter PCB compounds.

11.3 Extract Clean Up – For some aqueous samples and all tissue and solid samples, back extraction with sulfuric acid is necessary to remove the presence of interferences. The presence of color in an aqueous sample determines if back extraction is necessary. If it is not, proceed directly to the silica gel clean up procedure in Section 11.3.4. Optional clean up procedures described in Sections 11.3.2-11.3.3 may be employed. Spike all samples, including QC samples, with 100µL of the labeled clean up standard spiking solution at 10ng/mL (Table 3) prior to performing any extract clean up procedures.

11.3.1 Sulfuric Acid Clean Up

11.3.1.1 After concentration, solvent exchange the extract by adding 60mL hexane to the sample jar. Partition the extract against 10mL sulfuric acid. Shake for 30 seconds minimum with periodic venting into a hood. Remove and discard the acid (bottom) layer.

11.3.1.2 Repeat the acid washings until no color is visible in the hexane layer, up to a maximum of four washings.

11.3.1.3 Partition the extract against 10mL sodium chloride solution and shake in the same way as with the acid. Remove and discard the aqueous (bottom) layer.

11.3.1.4 Proceed with the silica gel clean up procedure.

11.3.2 Optional Sodium Hydroxide Clean Up

11.3.2.1 Prior to performing the silica gel clean up step, an optional base clean up may be performed to remove additional interferences based on the effectiveness of the sulfuric acid clean up or known sample matrix interferences.

11.3.2.2 Perform another solvent exchange step by adding 60mL hexane to the 10mL of solvent. Partition the extract against 10mL of a sodium hydroxide solution. Shake for 30 seconds minimum with periodic venting into a hood. Remove and discard the base (bottom) layer.

11.3.2.2 Repeat the base washings until no color is visible in the hexane layer, up to a maximum of four washings.

11.3.2.3 Partition the extract against 10mL sodium chloride solution and shake in the same way as with the base. Remove and discard the aqueous (bottom) layer.

11.3.2.4 Proceed with the silica gel clean up procedure.

11.3.3 Optional Florisil Clean Up

11.3.3.1 Prior to performing the silica gel clean up step, an optional Florisil clean up may be performed to eliminate polar and non-polar interferences based on the effectiveness of the sulfuric acid clean up or known sample matrix interferences.

11.3.3.2 Perform another solvent exchange step by adding 60mL hexane to the 10mL of solvent. Add approximately 0.5g activated Florisil to the extract.

11.3.3.3 Vortex the sample for 3 minutes. Let sit overnight.

11.3.3.4 Proceed with the silica gel clean up procedure.

11.3.4 Silica Gel Clean Up

11.3.4.1 Pack the tapered glass column as follows: Insert a glass wool plug into the bottom of the column. Place 2g activated neutral silica gel into the column. Add 6g activated sodium hydroxide infused silica gel, 9g activated sulfuric acid infused silica gel, and 3g anhydrous sodium sulfate. Tap the column gently after each addition to settle the silica gel. Elute with 30mL hexane. Check the column for channeling. If channeling is observed, discard the column. Do not tap a wetted column.

11.3.4.2 Place the silica gel column in the clamp on the clean up apparatus. Place a clean, appropriately labeled 250mL jar underneath the column to catch the eluate. The column is now ready to load the sample extract.

11.3.4.3 Transfer the 10mL extract to the silica gel column and allow the solvent to elute until the extract level is at the top of the sodium sulfate.

11.3.4.4 Rinse the 250mL jar containing the extract with 30mL hexane and load the rinsate onto the silica gel column. Allow the hexane to elute until the solvent level is at the top of the sodium sulfate.

11.3.4.5 Slowly add 30mL hexane to the silica gel column, allowing the hexane to pass through the column using a gravity feed. Add additional 30mL portions to each column, as space permits. The total volume of hexane eluant should be maximized to obtain the PCBs most efficiently, yet not overflow the 250mL jar.

11.3.5 Final Extract Concentration

11.3.5.1 Concentrate the extract using a rotary evaporator (with heating bath at 45-50°C) or nitrogen evaporator to a volume of approximately 15mL. Transfer the extract to a clean, appropriately labeled scintillation vial. Concentrate the extract further to a volume of approximately 1.5mL.

11.3.5.2 Transfer the extract quantitatively from the scintillation vial to the auto sampler vial while slowly evaporating the extract utilizing the nitrogen evaporator. Once the entire extract has been transferred to the auto sampler vial, concentrate the extract to approximately 100µL (or 20µL if requested).

11.3.5.3 The sample extract is ready to be spiked with 2µL of the internal injection standard at 5000ng/mL (Table 3).

12. PROCEDURE FOR HRGC/HRMS ANALYSIS AND CALIBRATION

12.1 Chromatographic/Mass Spectrometric Conditions and Data Acquisition Parameters

12.1.1 Gas Chromatograph

Column coating: SPB-Octyl
Film thickness: 0.25µm
Column dimension: 30m x 0.25mm (I.D.)
Injector temperature: 270°C
Splitless valve time: 1 min
Interface temperature: 295°C
Temperature program:

STAGE	INITIAL TEMP °C	INITIAL HOLD TIME, MIN	TEMPERATURE RAMP, °C/MIN	FINAL TEMP °C	FINAL HOLD TIME, MIN
1	75	3.1	15	150	0
2			2.5	290	0

The GC conditions may be optimized for compound separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, blanks, IPR, and OPR standards, and samples.

12.1.2 Mass Spectrometer

12.1.2.1 The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time (including the voltage reset time) of one second or less. It is important to maintain the same set of ions for

both calibration and sample extract analyses. All ions in Table 7 must be monitored.

12.1.2.2 The recommended mass spectrometer tuning conditions are based on the groups of monitored ions. By using a PFK molecular leak, tune the instrument to meet the minimum required resolving power of 10,000 (10% valley) at m/z 330.9792 (PFK) or any other reference signal between 300 and 350. By using peak matching conditions and the PFK reference peak, verify that the deviation between each monitored exact m/z and the theoretical m/z must be less than 5ppm. Each lock mass must be monitored and must not vary by more than ±20% throughout its respective retention time window. Variations of more than 20% indicate the presence of co-eluting interferences that raise the source pressure and may significantly reduce the sensitivity of the mass spectrometer.

12.1.2.3 Obtain a selected ion current profile (SICP) at the two exact m/z's specified in Table 7 and at ≥10,000 resolving power at each LOC for the native congeners and congener groups and for the labeled congeners. Because of the extensive mass range covered in each function, it may not be possible to maintain 10,000 resolution throughout the mass range during the function. Therefore, resolution must be ≥8,000 throughout the mass range and must be ≥10,000 in the center of the mass range for each function.

12.1.3 <u>Ion Abundance Ratios, Minimum Levels, and Signal-to-Noise Ratios</u> – Inject a 1µL aliquot of the CS-1 calibration solution (Table 5) using the GC conditions in Section 12.1.1.

12.1.3.1 Measure the SICP areas for each congener or congener group, and compute the ion abundance ratios at the exact m/z's specified in Table 7. Compare the computed ratio to the theoretical ratio given in Table 8.

12.1.3.2 All PCBs and labeled compounds in the CS-1 standard must be within the QC limits in Table 8 for their respective ion abundance ratios; otherwise, the mass spectrometer must be adjusted and this test repeated until the m/z ratios fall within the limits specified. If the adjustment alters the resolutions of the mass spectrometer, resolution must be verified (Section 12.1.2.2) prior to repeat of the test.

12.1.3.3 Verify that the HRGC/HRMS instrument meets the estimated minimum levels (EMLs) in Table 2). The peaks representing the PCBs and labeled compounds in the CS-1 calibration standard must have signal-to-noise (S/N) ratios ≥10; otherwise, the mass spectrometer must be adjusted and this test repeated until the minimum levels in Table 2 are met.

NOTE: The secondary ion of DiCB is interfered by PFK and requires a resolution of 22,000 to resolve sufficiently. S/N for these compounds in the CS-1 may fall below 10:1.

NOTE: CAS/Houston uses **Method Reporting Limit** (MRL) instead of EML when referencing the minimum levels in Table 2.

12.2 Initial Calibration by Isotope Dilution - Initial calibration is required before any samples can be analyzed for PCB congeners. Initial calibration is also required if any routine calibration does not meet the required acceptance criteria. Isotope dilution is used for calibration of the Toxics/LOC PCBs. The reference compound for each native compound is its labeled analog, as listed in Table 2. A five point calibration encompassing the concentration range is prepared for each native congener.

12.2.1 For the Toxics/LOC PCBs determined by isotope dilution, the relative response factors versus concentration in the calibration solutions is computed over the calibration range according to the procedures described below.

12.2.2 The response of each Toxics/LOC PCBs relative to its labeled analog is determined using the area responses of both the primary and secondary exact m/z's specified in Table 7, for each calibration standard, as follows:

$$RF_n = \frac{(A_n^1 + A_n^2) \times Q_{is}}{(A_{is}^1 + A_{is}^2) \times Q_n}$$

Where:

A_n^1 and A_n^2 = sum of the integrated ion abundances of the quantitation ions for unlabeled PCBs.

A_{is}^1 and A_{is}^2 = sum of the integrated ion abundances of the quantitation ions for the labeled standard PCBs.

Q_{is} = quantity of the labeled standard injected (pg).

Q_n = quantity of the unlabeled PCB analyte injected (pg).

The RF values are dimensionless quantities; the units used to express Q_{is} and Q_n must be the same.

12.2.3 To calibrate the analytical system by isotope dilution, inject calibration standards CS-1 through CS-5. Use a volume identical to the volume chosen in Section 12.1.3 and the conditions in Section 12.1.1. Compute and store the relative response factors for each Native Toxics/LOC PCB at each concentration. Compute the average (mean) RRF, as follows:

$$\overline{RF_n} = \frac{\sum_{j=1}^{5} RF_{n(j)}}{5}$$

Where:
n = The unlabeled PCB congener.
j = The injection number (or calibration solution number; j = 1 to 5).

12.2.4 Linearity – Calculate the RSD of the 5 RRFs. If the RRF for any Native Toxics/LOC PCB is constant (less than 20% RSD), the average RRF may be used for that congener; otherwise, the complete calibration curve for that congener must be used over the calibration range.

12.3 **Initial Calibration by Internal Standard** – Internal standard calibration is applied to determination of the native PCBs for which a labeled compound is not available, to determination of the Labeled Toxics/LOC/window-defining congeners, and labeled clean up congeners for performance tests and intra-laboratory statistics, and to determination of the Labeled injection internal standards. The reference compound for each compound is listed in Table 2. For the native congeners (other than the native Toxics/LOC PCBs), calibration is performed at a single point using the Diluted combined 209 congener solution. For the labeled compounds, calibration is performed using data from the five points in the calibration for the Native Toxics/LOC PCBs (Section 12.2).

12.3.1 Response Factors – Internal standard calibration requires the determination of response factors (RF) defined by the following equation:

$$RF_n = \frac{(A_n^1 + A_n^2) \times Q_{is}}{(A_{is}^1 + A_{is}^2) \times Q_n} \qquad RF_{is} = \frac{(A_{is}^1 + A_{is}^2) \times Q_{is}}{(A_{is}^1 + A_{is}^2) \times Q_{is}}$$

Where:
A_n^1 and A_n^2 = sum of the integrated ion abundances of the quantitation ions for unlabeled PCBs.
A_{is}^1 and A_{is}^2 = sum of the integrated ion abundances of the quantitation ions for the labeled standard PCBs.
A_{is}^1 and A_{is}^2 = sum of the integrated ion abundances of the quantitation ions the internal injection standards.
Q_{is} = quantity of the labeled standard injected (pg).
Q_{is} = quantity of the internal injection standard injected (pg).
Q_n = quantity of the unlabeled PCB analyte injected (pg)

The RF_n and RF_{is} values are dimensionless quantities; the units used to express Q_{is}, Q_{is} and Q_n must be the same.

12.3.2 To single-concentration calibrate the system for native PCBs other than the Native Toxics/LOC PCBs by internal standard, inject the Diluted combined 209 congener solution. Use a volume identical to the volume chosen in 12.1.3 and the conditions in Section 12.1.1.

12.3.3 Compute and store the relative response factor (*RF*) for all native PCBs except the Native Toxics/LOC PCBs. Use the average (mean) response of the labeled compounds at each level of chlorination (LOC) as the quantitation reference, to a maximum of 4 labeled congeners, as shown in Table 2. For the combinations of isomeric congeners that co-elute, compute a combined RF for the co-eluted group. For example, for congener 122, the areas at the exact m/z's for 104L, 114L, 118L, and 123L are summed and the total area is divided by 4 (because there are 4 congeners in the quantitation reference).

NOTE: All labeled congeners at each LOC are used as reference to reduce the effect of an interference if a single congener is used as reference. Other quantitation references and procedures may be used provided that the results produced are as accurate as results produced by the quantitation references and procedures described above.

12.3.4 Compute and store the relative response factor (*RF*) for the labeled compounds, except 138L. For the Labeled Toxics/LOC/window-defining compounds and the labeled clean up standards, use the nearest eluted labeled injection internal standard as the quantitation reference, as given in Table 2. The Labeled injection internal standards are referenced to PCB 138L, as shown in Table 2.

12.3.5 If the requested analysis comprises only those PCBs contained in the Native Toxics/LOC mix, a five point calibration must be followed. Response factors shall be calculated based on the five points for the Native Toxics/LOC, Labeled Toxics/LOC, Labeled clean up, and Labeled injection internal standards. If the requested analysis comprises PCBs other than those contained in the Native Toxics/LOC mix, a five point calibration must be followed for the Native Toxics/LOC compounds while a one point calibration must be followed for the additional compounds.

12.3.6 <u>Secondary Source Verification</u> - Immediately following the analysis of the calibration solutions, analyze another standard at the midpoint of the calibration. This standard must be obtained from a secondary vendor, or be from a lot independent of the lot used for the calibration solutions. Calculate the response factors of all compounds and verify against the initial calibration results.

12.4 **Calibration Verification** - At the beginning of each 12-hour shift during which analyses are performed, GC/MS system performance and calibration are verified for all native PCBs and labeled compounds. For these tests, analysis of the CS-3 calibration verification (VER) standard (Table 5) and the diluted combined 209 congener solution (Table 3) must be used to verify all performance criteria. Adjustment and/or recalibration

must be performed until all performance criteria are met. Only after all performance criteria are met may samples, blanks, IPRs, and OPRs (LCSs) be analyzed.

12.4.1 MS Resolution – Static resolving power checks must be performed at the beginning and end of each shift. If analyses are performed on successive shifts, only the beginning of shift check is required. If the operating criteria cannot be met, the problem must be correct before analyses can proceed. If any of the samples in the previous run may be affected by poor resolution, those samples must be reanalyzed.

12.4.2 Inject the VER (CS-3) standard using the established operating conditions.

12.4.3 The m/z abundance ratios for all PCBs must be within the limits in Table 8; otherwise, the mass spectrometer must be adjusted until the m/z abundance ratios fall within the limits specified when the VER is repeated.

12.4.4 The GC peak representing each native PCB and labeled compound in the VER standard must be present with a S/N of ≥10; otherwise, the mass spectrometer must be adjusted and the VER repeated.

12.4.5 Compute the concentration of the Toxics/LOC PCBs by isotope dilution. These concentrations are computed based on the initial calibration data.

12.4.6 For each compound, compare the concentration with the calibration verification limit in Table 6. If all compounds meet the acceptance criteria, calibration has been verified and analysis of standards and sample extracts may proceed. If, however, any compound fails its respective limit, the measurement system is not performing properly. In this event, prepare a fresh calibration standard or correct the problem and repeat the resolution and VER tests, or recalibrate. If recalibration is necessary, recalibration for the 209 congeners must also be performed.

12.4.7 Retention Times

12.4.7.1 Absolute. The absolute retention times of the Labeled Toxics/LOC/window defining standard congeners in the VER must be within ±15 seconds of the respective retention times in the initial calibration.

12.4.7.2 Relative. The relative retention times of the native PCBs and labeled compounds in the VER must be within their respective RRT limits in Table 2.

12.4.7.3 If the absolute or relative retention time of any compound is not within the limits specified, the GC is not performing properly. In this event, adjust the GC and repeat the VER or recalibrate, or replace the GC column and either verify calibration or recalibrate.

12.4.8 GC Resolution and Minimum Analysis Time

12.4.8.1 As a final step in the calibration verification, inject the diluted combined 209 congener solution.

12.4.8.2 The resolution and minimum analysis time specifications in Section 7.2.2 must be met for the SPB-Octyl column. If these specifications are not met, the GC analysis conditions must be adjusted until the specifications are met, or the column must be replaced and the calibration verification test repeated, or the system must be recalibrated.

12.4.8.3 After the resolution and minimum analysis time specifications are met, update the retention time, relative retention times, and response factors for all congeners except for the response factors for the compounds that are multi-point calibrated. For these compounds, the multi-point calibration data must be used.

12.5 Analysis

12.5.1 Establish the operating conditions that resulted in acceptable calibration.

12.5.2 Add 2µL of the labeled injection internal at 5000ng/mL (Table 3) to the 100µL (or 20µL) sample extract immediately prior to injection to minimize the possibility of loss by evaporation, adsorption, or reaction.

12.5.3 Inject 1µL of the concentrated extract containing the Labeled injection internal standards using splitless injection. The volume injected must be identical to the volume used for calibration.

12.5.3.1 Start the GC column initial isothermal hold upon injection. Start MS data collection after the solvent peak elutes.

12.5.3.2 Monitor the exact m/z's at each LOC throughout the LOC retention time window. Where warranted, monitor m/z's associated with congeners at higher levels of chlorination to assure that fragments are not interfering with the m/z's for congeners at lower levels of chlorination. Also where warranted, monitor m/z's associated with interferents expected to be present.

12.5.3.3 Stop data collection after $^{13}C_{12}$-DeCB has eluted. Return the column to the initial temperature for analysis of the next extract or standard.

12.6 Identification Criteria – A PCB or labeled compound is identified in a standard, blank, or sample when all of the criteria in this Section are met.

12.6.1 The signals for the two exact m/z's in Table 7 must be present and must maximize within the same two scans.

12.6.2 The signal-to-noise ratio (S/N) for the GC peak at each exact m/z must be ≥2.5 for each PCB detected in a sample extract, and ≥10 for all PCBs in the calibration and verification standards.

12.6.3 The ratio of the integrated areas of the two exact m/z's specified in Table 7 must be within the limit in Table 8.

12.6.4 The relative retention time of the peak for a PCB must be within the RRT QC limits specified in Table 2 or within similar limits developed from calibration data.

12.6.5 Because of congener overlap and the potential for interfering substances, it is possible that all of the identification criteria may not be met. It is also possible that loss of one or more chlorines from a highly chlorinated congener may inflate or produce a false concentration for a less-chlorinated congener that elutes at the same retention time. If identification is ambiguous, an experienced spectrometrist must determine the presence or absence of the congener.

12.6.6 If the criteria stated above are not met, the PCB has not been identified and the result for that congener may not be reported or used for permitting or regulatory compliance purposes. If interferences preclude identification, a new aliquot of sample must be extracted, further cleaned up, and analyzed.

13. QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

13.1 The minimum requirements for quality assurance consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. Performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the procedure.

If the procedure is to be applied to sample matrix other than water the most appropriate alternate reference matrix is substituted for the reagent water matrix in all performance tests.

13.1.1 Each analyst must make an initial demonstration of the ability to generate acceptable precision and recovery with this procedure. This demonstration is given in Section 13.2.

13.1.2 In recognition of advances that are occurring in analytical technology, and to overcome matrix interferences, the laboratory is permitted certain options to improve separations or lower the costs of measurements. These options include alternate extraction, concentration, and clean up procedures, and changes in columns and detectors. If an analytical technique other than the techniques

specified in EPA Method 1668A is used, that technique must have a specificity equal to or greater than the specificity of the techniques in EPA Method 1668A for the analytes of interest.

13.1.2.1 Prior to a modification being made to this procedure, analysts must repeat the procedure in Section 13.2. If the detection limits of the procedure will be affected by the change, the analyst is required to demonstrate that the DLs are lower than one-third the regulatory compliance level or one-third the EMDLs in EPA Method 1668A, whichever are greater. If calibration will be affected by the change, the instrument must be recalibrated per Section 12. Once it is demonstrated that the modification produces results equivalent or superior to results produced by this procedure as written, that modification may be used routinely thereafter, so long as the other requirements in this procedure are met, and this SOP is updated to reflect that change.

13.1.2.2 The analyst is required to maintain records of modifications made to this procedure. This includes, at a minimum, results from all quality control tests comparing the two procedures, data that will allow an independent reviewer to validate each determination by tracing the instrument output to the final result, and documentation in this procedure in the form of a modification detailed in Section 21.

13.1.3 Analyses of method blanks are required to demonstrate freedom from contamination. The procedures and criteria for analysis of a method blank are described in Sections 13.1 and 13.6.

13.1.4 The analyst must spike all samples with labeled compounds to monitor procedural performance. This test is described in Section 13.3. When results of these spikes indicate atypical procedural performance for samples, the samples are diluted to bring procedural performance within acceptable limits. Procedures for dilution are given in Section 14.1.4.1.

13.1.5 The analyst must, on an ongoing basis, demonstrate through calibration verification and the analysis of the ongoing precision and recovery standard (OPR; or Laboratory Control Sample, LCS) and blanks that the analytical system is in control. These procedures are given in Section 13.7.

13.2 Initial Precision and Recovery (IPR) – To establish the ability to generate acceptable precision and recovery, the analysts must perform the following operations.

13.2.1 For low solids (aqueous) samples, extract, concentrate, and analyze four 1L aliquots of reagent water spiked with 1.0mL each of the Matrix standard spiking solution, the Labeled standard spiking solution, and the Labeled clean up standard spiking solution, according to the procedures set out for field samples. For an alternative sample matrix, four aliquots of the alternative reference matrix are

used. All sample processing steps that are to be used for processing samples, including preparation, extraction, and clean up, must be included in this test.

13.2.2 Using results of the set of four analyses, compute the average percent recovery (X) of the extracts and the relative standard deviation (RSD) of the concentration for each compound, by isotope dilution for PCBs with a labeled analog, and by internal standard for PCBs without a labeled analog and for the labeled compounds.

13.2.3 For each PCB and labeled compound, compare RSD and X with the corresponding limits for initial precision and recovery in Table 6. If RSD and X for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If, however, any individual RSD exceeds the precision limit or any individual X falls outside the range for recovery, system performance is unacceptable for that compound. Correct the problem and repeat the test.

13.3 To assess the procedural performance on the sample matrix, the laboratory must spike all samples with the Labeled standard spiking solution and all sample extracts with the Labeled clean up standard spiking solution.

13.3.1 Analyze each sample according to the procedures set forth by this SOP.

13.3.2 Compute the percent recovery of the labeled congeners and the labeled clean up congeners using the internal standard method.

13.3.3 The recovery of each labeled compound must be within the limits in Table 6. If the recovery of any compound falls outside of these limits, procedural performance is unacceptable for that compound in that sample. Additional clean up procedures must then be employed to attempt to bring the recovery within the normal range. If the recovery cannot be brought within the normal range after all clean up procedures have been employed, water samples are diluted and smaller amounts of soils, sludges, sediments, and other matrices are analyzed.

13.4 It is suggested but not required that recovery of labeled compounds from samples be assessed and records maintained.

13.4.1 After the analysis of 30 samples of a given matrix type (water, soil, sludge, pulp, etc.) for which the labeled compounds pass the tests in Section 13.3, compute the average percent recovery (R) and the standard deviation of the percent recovery (S_R) for the labeled compounds only. Express the assessment as a percent recovery interval from $R - 2S_R$ to $R + 2S_R$ for each matrix.

13.4.2 Update the accuracy assessment for each labeled compound in each matrix on a regular basis (e.g. after each five to ten new measurements).

13.5 Sample Preparation Batching Requirements

13.5.1 Number – The number of field samples in a preparation batch will not exceed twenty (20).

13.5.2 Matrix - The matrix to be used for the quality control samples must be consistent with the matrix of the field samples (i.e. reagent water for aqueous matrices, quartz sand or sodium sulfate for solid matrices, or blank tissue for tissue matrices).

13.5.3 Reagent Lots – A single lot of each reagent used in the analysis will be used to process the batch of samples.

13.5.4 Batch Time Frame – The maximum time between the start of processing of the first and last samples in the batch must be twenty-four (24) hours.

13.5.5 Batch QC Samples – Each preparation batch must contain, at a minimum, a Method Blank (Section 13.6) to monitor laboratory introduced contamination, an LCS (Section 13.7) to assess analysis performance (i.e. bias, or accuracy), and an additional sample to assess batch precision. This additional sample can be in the form of a DLCS (Section 13.7), an MS/DMS pair (Section 13.8), or a DUP (Section 13.9). Batch QC samples do not count towards the maximum number of sample allowed in a batch (Section 13.5.1).

13.6 Method Blanks – A reference matrix Method Blank is analyzed with each sample batch to demonstrate freedom from contamination. The matrix for the MB must be similar to the sample matrix for the batch.

13.6.1 Spike each of the Labeled standard spiking solution and the Labeled clean up standard spiking solutions into the MB, according to the procedures set forth in this SOP. Prepare, extract, clean up, and concentrate the MB. Analyze the blank immediately after analyses of the OPR (LCS) to demonstrate freedom from contamination and freedom from carryover.

13.6.2 If any PCB is found in the blank at greater than the minimum level (MRL) or one-third the regulatory compliance limit, whichever is greater, or if any potentially interfering compound is found in the blank at the MRL for each PCB given in Table 2 (assuming a response factor of 1 relative to the quantitation reference in Table 2 at that level of chlorination for a potentially interfering compound, i.e. a compound not listed in this procedure), the blank must be evaluated to determine the extent of the contamination. All samples must be associated with an uncontaminated MB or the data must be flagged on the analytical report.

13.7 Laboratory Control Samples – A reference matrix LCS is analyzed (along with a DLCS) with each sample batch to demonstrate the batch's accuracy (and precision). The matrix for the LCS/DLCS must be similar to the sample matrix for the batch. The LCS/DLCS combination is run in lieu of an MS/DMS, unless specifically requested by the client.

13.7.1 Spike each of the Labeled standard spiking solution, Matrix standard spiking solution, and Labeled clean up spiking solutions into the LCS/DLCS, according to the procedures set forth in this SOP. Prepare, extract, clean up, and concentrate the LCS/DLCS. Analyze the LCS/DLCS immediately after analysis of the calibration verification and Diluted combined 209 congener solution.

13.7.2 For the Toxics/LOC CBs and labeled compounds, compare the recovery to the LCS limits given in Table 6. If all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may proceed. If, however, any individual concentration falls outside of the range given, the extraction/concentration processes are not being performed properly for that compound. In this event, correct the problem, re-prepare, extract, and clean up the sample batch and repeat the LCS.

13.7.3 To verify performance for all 209 congeners, the LCS/DLCS must contain all 209 congeners over a period of 2 years. Compare the results of the 27 Native Toxics/LOCs against the requirements in Table 6.

13.8 Matrix Spike Samples – A client may request an MS/DMS be performed on a field sample. If requested, the MS/DMS will demonstrate the batch's accuracy and precision. A DLCS is not required if an MS/DMS is performed.

13.8.1 Spike each of the Labeled standard spiking solution, Matrix standard spiking solution, and Labeled clean up spiking solutions into the MS/DMS, according to the procedures set forth in this SOP. Prepare, extract (being sure to properly homogenize the field sample such that three identical aliquots can be taken), clean up, and concentrate the MS/DMS. If insufficient sample size is received, contact the client for directions on how to proceed.

13.8.2 To verify performance for all 209 congeners, the MS/DMS must contain all 209 congeners over a period of 2 years. Compare the results of the 27 Native Toxics/LOCs against the requirements in Table 6.

13.9 Field Sample Duplicates (DUP) – A client may request a DUP be performed on a field sample. If requested, the DUP will demonstrate the batch's precision. A DLCS is not required if a DUP is performed.

13.9.1 Homogenize the sample and separate two identical aliquots for analysis. Often, for aqueous samples requiring DUP analyses, the client will send a sufficient sample amount to use 1L for each aliquot. Add 1.0mL of the Labeled standard solution at 2-4ng/mL (Table 3). No matrix standard is added to either aliquot.

13.9.2 Extract and analyze according to Section 11. This measurement provides the batch's precision (how closely the result compares with the field sample.). The LCS acts as an accuracy test.

13.9.3 Evaluate the test results according to the acceptance criteria (≤50% RPD). Sample concentrations below five times the Method Reporting limit (5xMRL) may not be significant enough to present reliable RPD data.

13.10 Performance Evaluation Samples – Included among the samples in all batches may be samples (blind or double blind) containing known amounts of unlabeled 2,3,7,8-substituted PCDDs/PCDFs or other PCDD/PCDF congeners.

CAS/Houston participates in performance evaluation studies through R.T. Corp (RTC). Solid, aqueous, and tissue studies are performed semiannually. All studies are performed following the guidelines of the SOP for *Proficiency Testing Sample Analysis*, ***ADM-PTS***.

14. DATA REDUCTION AND REPORTING

14.1 Calculations – A combination of OpusQuan and CAS LIMS is used to calculate concentrations based on the interpretation of the raw data and the preparation information.

14.1.1 Isotope Dilution Quantitation

14.1.1.1 By adding a known amount of the Labeled Toxics/LOC/window-defining compounds to every sample prior to extraction, correction for recovery of the PCB can be made because the native compound and its labeled analog exhibit similar effects upon extraction, concentration, and gas chromatography. Relative response factors (RRFs) are used in conjunction with the calibration data in Section 12.2 to determine concentrations in the final extract, so long as labeled compound spiking levels are constant.

14.1.1.2 Compute the concentrations in the extract of the Native Toxics/LOC PCBs using the RRFs from the calibration data (Section 12.2) and following equation:

$$C_{ex} = \frac{(A^1_n + A^2_n)C_l}{(A^1_l + A^2_l)RF_n}$$

Where:

C_{ex} = The concentration of the PCB in the extract.
A^1_n and A^2_n = The areas of the primary and secondary m/z's for the PCB.
A^1_l and A^2_l = The areas of the primary and secondary m/z's for the labeled compound.
C_l = The concentration of the labeled compound (Table 3).

$\overline{RF_n}$ = Calculated mean relative response factor for the native compound relative to the appropriate labeled compound.

14.1.2 Internal Standard Quantitation and Labeled Compound Recovery

14.1.2.1 Compute the concentrations in the extract of the native compounds other than those in the Native Toxics/LOC standard using the updated response factors determined from the calibration verification data (Section 12.4) and of the labeled internal standards, of the labeled clean up standards, and of the labeled injection internal standards determined from the initial calibration data (Section 12.3) using the following equation:

$$C_{ex} = \frac{(A^1_x + A^2_x)C_{is}}{(A^1_{is} + A^2_{is})\overline{RF_{in}}}$$

Where:

C_{ex} = The concentration of the labeled compound in the extract.
A^1_x and A^2_x = The areas of the primary and secondary m/z's for the PCB.
A^1_{is} and A^2_{is} = The areas of the primary and secondary m/z's for the internal standard.
C_{is} = The concentration of the internal standard (Table 3)
$\overline{RF_{in}}$ = Calculated mean relative response factor for the native compound relative to the appropriate internal standard.

14.1.2.2 Using the concentration in the extract determined above, compute the percent recovery of the Labeled Toxics/LOC/window-defining PCBs and the Labeled clean up standard PCBs using the following equation.

$$\text{Recovery}(\%) = \frac{C_{found}}{C_{spiked}} \times 100$$

14.1.3 The concentration of a native PCB in the sample is computed using the concentration of the compound in the extract and the weight/volume of the sample, as follows:

$$Concentration = \frac{(C_{ex} \times V_{ex})}{wv_s}$$

Where:

C_{ex} = The concentration of the compound in the extract.
V_{ex} = The extract final volume, in mL.
wv_s = The sample dry weight (in kg) or volume (in L)

NOTE: The concentration of the compound in the sample shall be expressed as ng/kg for solid samples and pg/L for aqueous samples.

14.1.4 If the SICP area at either quantitation m/z for any congener exceeds the calibration range of the system, dilute the sample extract by the factor necessary to bring the concentration within the calibration range, adjust the concentration of the Labeled injection standard to 100pg/µL in the extract, and analyze an aliquot of this diluted extract. If the PCBs cannot be measured reliably by isotope dilution, analyze a smaller portion of the aqueous or solid sample. All compounds exceeding the upper calibration limit are flagged with an E flag. Alternative options are to reduce the sample size or split a portion of the extract prior to clean up.

14.1.4.1 To prepare a dilution, use the original analysis data to quantify the labeled standard recoveries. These original labeled standard recoveries will be applied to the dilution, so that the only variable in the dilution is the actual concentrations of PCBs. Dilute the original extract (note that no additional clean up is necessary) with the dilution factor times the usual quantity of internal standards. Reanalyze the diluted sample using the labeled standard recoveries calculated from the initial analysis to correct the diluted results for losses during the original extraction and clean up. Calculate the results as in Section 14.1, multiplying the final PCB concentrations by the dilution factor.

14.1.5 The total concentration for each homologous series of PCBs is calculated by summing up the concentrations of all positively identified compounds of each homologous series. Also, if requested, total PCBs may be reported by summing all congeners identified at all levels of chlorination.

14.1.6 Sample Specific Estimated Detection Limit - The sample specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background noise level. An EDL is calculated for each PCB congener that is not identified. Two methods of calculation can be used, as follows, depending on the type of response produced during the analysis of a particular sample.

14.1.6.1 For samples giving a response for both quantitation ions that are less than 2.5 times the background noise level, use the expression for EDL below. The background level is determined by measuring the range of the noise (peak to peak) for the two quantitation ions of a particular PCB compound within an homologous series, in the window of the SICP trace corresponding to the elution of the labeled standard (if the congener possesses a labeled standard) or in the window of the SICP where the congener is expected to elute by comparison with the routine calibration data (for those congeners that do not have a C13-labeled standard), multiplying that noise height by 2.5, and relating the product

to an estimated concentration that would produce that peak height. Use the formula:

$$EDL = \frac{2.5 \times H_x \times Q_{ls}}{H_{ls} \times \overline{RF_n}}$$

where:

EDL = Estimated detection limit for homologous PCBs.

H_x = Sum of the height of the noise level for each quantitation ion for the unlabeled PCBs.

Q_{ls} = Quantity, in pg, of the labeled standard.

H_{ls} = Sum of the height of the signal level for each quantitation ion for the labeled standard.

$\overline{RF_n}$ = Calculated mean relative response factor for the compound.

14.1.6.2 For compounds characterized by the response of a signal having the same retention time as a PCB, having a S/N in excess of 2.5, and does not meet any of the other qualitative identification criteria, calculate the "Estimated Maximum Possible Concentration" (*EMPC*), with the exception that the variable A represents the sum of the area under the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio.

$$\frac{A_1}{A_2} = i_c \qquad A_1 = A_2 \times i_t \qquad (i_c > i_t)$$

$$A_2 = \frac{A_1}{i_t} \qquad (i_c < i_t)$$

Where:

A_1 = Area of the first ion used for quantitation.

A_2 = Area of the second ion used for quantitation.

i_c = Calculated ion abundance ratio for the compound.

i_t = Theoretical ion abundance ratio for the compound.

If $i_c > i_t$ then use A_2 and adjust the value for A_1. If $i_c < i_t$ then use A_1 and adjust the value for A_2. Use the second and third equations for this adjustment.

14.1.7 The relative percent difference (*RPD*) of any duplicate sample results are calculated as follows:

$$RPD = \frac{|S_1 - S_2|}{(S_1 + S_2)/2} \times 100$$

Where:

S_1, S_2 = Sample and duplicate sample results.

14.1.8 The 2,3,7,8-TCDD toxicity equivalents (TEQ/TEF) of PCBs present in the sample are calculated, if requested by the data user, according to the method recommended by the Chlorinated Dioxins Workgroup (CDWG) of the EPA and the Center for Disease Control (CDC). This method assigns a 2,3,7,8-TCDD toxicity equivalency factor (TEF) to each of the coplanar PCBs. The 2,3,7,8-TCDD equivalent of the PCBs present in the sample is calculated by summing the TEF times their concentration for each of the compounds or groups of compounds. The above procedure for calculating the 2,3,7,8-TCDD toxicity equivalents is not claimed by the CDWG to be based on a thoroughly established scientific foundation. The procedure, rather, represents a "consensus recommendation on science policy." Since the procedure may be changed in the future, reporting requirements for PCB data would still include the reporting of the analyte concentrations of the PCB congeners.

14.2 Data review

14.2.1 It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met.

14.2.2 Refer to the SOP for *HRMS Data Review and Reporting*, ***HMS-DATAREV***, for general instructions for data review.

14.3 Results are reported based on the SOP for *Significant Figures*, ***ADM-SIGFIG***.

14.4 Results for a PCB in a sample that has been diluted are reported at the least dilute level at which the area at the quantitation m/z is within the calibration range. Results for a PCB in a dilution above the reported level do not need to be processed and reviewed.

14.5 For a PCB having a labeled analog, report results at the least dilute level at which the area at the quantitation m/z is within the calibration range and the labeled compound recovery is within the normal range for the method.

14.6 Perform manual integrations of peak areas when interferences preclude computerized calculations. The analyst must use their professional judgment to determine where the signal baseline is located and determine the shape of the peak to be integrated. Using the tools in OpusQuan, the peak must be manually integrated to represent a straight baseline equivalent to the noise. Follow the SOP for *Manual Integration of Chromatographic Peaks*, ***ADM-INT***.

15. METHOD PERFORMANCE

15.1 Method performance information can be found in Method 1668A, Section 21.0.

15.2 Method performance is monitored using a sample specific EDL calculation, as shown above. Method reporting limits (MRLs) are as defined in the reference method (Estimated Minimum Levels, or EMLs, Table 2). MRLs have no correlation to the EDL.

15.3 Limits of Detection (LOD) and Limits of Quantitation (LOQ) are generated and verified according to the requirements in the Department of Defense Quality Systems Manual (Reference 19.2).

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat from solvent and reagents used in this method can be minimized when recycled or disposed of properly. All disposable glassware should be recycled as per the laboratory procedures. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual. Also, see the SOP for *Waste Disposal*, ***SMO-WASTDISP***.

17. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this SOP. Also, refer to the SOP for *Corrective Action*, ***ADM-CA***, for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for *HRMS Data Review and Reporting*. See Table 10 for example corrective actions and required documentation.

17.1 Problems associated with low standard recoveries are usually attributed to one of the following:

17.1.1 Extremely low recoveries of labeled standards (for samples where the clean up standard recoveries are normal) indicate that extraction is inefficient. The cause may also be attributed to matrix effect.

17.1.2 Low recoveries of both labeled standards and clean up standards indicate that losses are taking place during the clean up process. Such losses may be due to inefficient final transfer or may be taking place during clean up. Contaminants in the sample extract may interact with the silica gel and alter the retention times of congeners on the column.

17.1.3 If any of the standards are completely absent from the selected ion profile, the possibility should be considered that addition of that standard to the sample or extract has not taken place. If this is the case, the sample must be re-extracted.

17.1.4 If the chromatographic peak area for a standard is approximately twice the anticipated area, the possibility should be considered that two aliquots of that standard have been added to the sample or extract.

17.2 Low sensitivity (poor signal-to-noise ratio) may be attributed to a number of causes.

17.2.1 A leaking septum might result in losses of sample during injection. Another consequence of a leaking septum (or any leak in the inlet system of the gas chromatograph) is reduced flow of carrier gas, resulting in increased retention times.

17.2.2 Accumulation of particles of septum or ferrule material in the injection port can lead to adsorptive losses. Similar losses are encountered when the injection port or the injection port liner are contaminated by the residues of 'dirty' samples.

17.2.3 A degraded column can cause peak distortion, and may also lead to adsorptive losses. Excessive bleed of stationary phase from the column may result in high background levels and, consequently, elevated noise levels (thus decreasing the signal-to-noise ratio). The problem is commonly restricted to the first few centimeters of the column, and can be corrected by cutting off 10 to 20cm of the column.

17.2.4 High background levels can also be attributed to bleed from an inappropriate septum.

17.2.5 Re-tuning of the mass spectrometer might improve the sensitivity, or the problem may be due to a contaminated ion source. If, over a period of time, continual increases in the electron multiplier voltage are needed to obtain adequate sensitivity, then the photo multiplier may have to be replaced.

17.3 Contamination problems, if they arise, should be investigated systematically.

17.3.1 Contaminated chemicals should be discarded. If one or more additional containers of the same lot number are present in the laboratory, the additional quantity of chemical should be evaluated for contamination prior to use.

17.3.2 Contaminated glassware should be discarded.

17.3.3 Each step of the preparation and analytical procedure must be isolated to determine the particular step, reagent, chemical, or piece of equipment that may be contributing the contamination. Once it has been isolated, corrective action measures must be put in place to remove the contamination and prevent its reoccurrence.

17.4 Problems that require corrective action are to be documented by the analyst.

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

See Section 17.

19. REFERENCES

19.1 "Method 1668 Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids and Tissue by HRGC/HRMS." USEPA. 821-R-00-02. December 1999.
19.2 DoD *Quality Systems Manual*. Final Version 4.1
19.3 2003 NELAC Quality Systems Standard

20. TRAINING PLAN

20.1 Refer to the SOP for *Documentation of Training*, ***ADM-TRANDOC***. The SOP describes the training outline and necessary documentation (Figure 6).

20.2 Review the SOP and published method. Also review safety procedures, any instrument related manuals, and all related SOPs.

20.3 Following these reviews observe the procedure as performed by an experienced analyst at a minimum of three times.

20.4 Once the analyst and trainer are comfortable with the procedure, the analyst must perform the SOP with supervision for an extended period of time. During this period, the analyst is expected to transition from a role of assisting to performing the procedure with minimal oversight from an experienced analyst.

20.5 Independently perform the SOP and complete an Initial Demonstration of Capabilities. This study can be based on either a blind Performance Evaluation sample or four consecutive LCS samples. Document these studies.

21. METHOD MODIFICATIONS

The following modifications have been made to the published method (referenced parenthetically). These modifications offer equivalent performance at a reduced cost

21.1 Modification of separatory funnel extraction by replacing with ½ gal. extraction jars. Solvent volume and total number of rinses has been reduced from 180mL and three rinses to 150mL and 2 rinses (Reference 19.1 Section 12.2.2).
21.2 No TSS for aqueous samples (Ref 19.1 Sec 11.2).
21.3 Addition of ASE extraction technique for solid samples (Ref 19.1 Sec 12.3).
21.4 Soxhlet extraction also performed for solid samples (not SDS). Sample is mixed adequately with sodium sulfate to improve extraction efficiency (Ref 19.1 Sec 11.3).
21.5 No particle size estimation is performed (Ref 19.1 Sec 12.3).

21.6 A kiln is not employed by CAS/Houston for all reusable glassware cleaning (Ref 19.1 Sec 4.2).

21.7 Silica gel activated at >120°C. No GPC, carbon, HPLC, or anthropogenic clean ups are performed. Column packing procedure is slightly different (no neutral gel between layers). Silica gel elution is greater than 25mL (Ref 19.1 Sec 13).

21.8 Sulfuric acid and sodium hydroxide back extraction volumes are reduced (Ref 19.1 Sec 12.5). Sodium hydroxide back extraction and florisil clean ups are optional (Ref 19.1 Secs 12.5 and 13).

21.9 Standard procedure is to bring final volume of extracts to 100uL rather than 20uL (Ref 19.1 Sec 12.7.7).

21.10 All extracts are stored cold at 0-6°C instead of frozen (Ref 19.1 Sec 8.5.2).

21.11 OpusQuan is limited to four quantitation reference compounds. Therefore, all PentaCBs are quantitated against 104L, 114L, 118L, and 123L. The purpose of quantitating against more than one labeled standard is to reduce the effect of interferences. Quantitating against four, rather than five, compounds still has an equivalent effect (Ref 19.1 Table 2).

22. INSTRUMENT SPECIFIC ADDENDUM

None.

23. CHANGES FROM PREVIOUS REVISION

23.1	Section 1.1	Added EPA Method reference
23.2	Sections 3.7-8	Updated.
23.3	Section 5	Updated.
23.4	Section 6.2.1	Updated.
23.5	Section 7.3	New and all following sections renumbered.
23.6	Section 8.8	New and all following sections renumbered.
23.7	Section 8.9	Updated for muffle furnace procedure.
23.8	Section 11.1.2	Updated.
23.9	Section 11.1.4.5	Updated to include corn oil blank matrix.
23.10	Section 11.2	Updated.
23.11	Section 11.3	Updated.
23.12	Section 14.1.4	Updated
23.13	Section 15	Updated.
23.14	Section 19	Updated.
23.15	Section 21	Updated.
23.16	Table 2	Updated (footnote).
23.17	Table 3	Updated.

24. ATTACHMENTS

24.1 **Tables** – See Appendix 1.

24.2 **Figures** – See Appendix 2.

APPENDIX 1

TABLE 1
NAMES, CONGENER NUMBERS, AND CAS REGISTRY NUMBERS FOR NATIVE AND LABELED CHLORINATED BIPHENYL CONGENERS

CB congener[1]	IUPAC number	CAS registry number	Labeled analog	IUPAC analog	CAS registry number
2-MoCB	1	2051-60-7	$^{13}C_{12}$-2-MoCB[2]	1L	234432-85-0
3-MoCB	2	2051-61-8			
4-MoCB	3	2051-62-9	$^{13}C_{12}$-4-MoCB[2]	3L	208263-77-8
2,2'-DiCB	4	13029-08-8	$^{13}C_{12}$-2,2'-DiCB[2]	4L	234432-86-1
2,3-DiCB	5	16605-91-7			
2,3'-DiCB	6	25569-80-6			
2,4-DiCB	7	33284-50-3			
2,4'-DiCB[3]	8	34883-43-7			
2,5-DiCB	9	34883-39-1	$^{13}C_{12}$-2,5-DiCB[2]	9L	256694-89-4
2,6-DiCB	10	33146-45-1			
3,3'-DiCB	11	2050-67-1			
3,4-DiCB	12	2974-92-7			
3,4'-DiCB	13	2974-90-5			
3,5-DiCB	14	34883-41-5			
4,4'-DiCB	15	2050-68-2	$^{13}C_{12}$-4,4'-DiCB[2]	15L	208263-67-6
2,2',3-TriCB	16	38444-78-9			
2,2',4-TriCB	17	37680-66-3			
2,2',5-TriCB[3]	18	37680-65-2			
2,2',6-TriCB	19	38444-73-4	$^{13}C_{12}$-2,2',6-TriCB[2]	19L	234432-87-2
2,3,3'-TriCB	20	38444-84-7			
2,3,4-TriCB	21	55702-46-0			
2,3,4'-TriCB	22	38444-85-8			
2,3,5-TriCB	23	55720-44-0			
2,3,6-TriCB	24	55702-45-9			
2,3',4-TriCB	25	55712-37-3			
2,3',5-TriCB	26	38444-81-4			
2,3',6-TriCB	27	38444-76-7			
2,4,4'-TriCB[3]	28	7012-37-5	$^{13}C_{12}$-2,4,4'-TriCB[2]	28L	208263-76-7
2,4,5-TriCB	29	15862-07-4			
2,4,6-TriCB	30	35693-92-6			
2,4',5-TriCB	31	16606-02-3			
2,4',6-TriCB	32	38444-77-8			
2',3,4-TriCB	33	38444-86-9			
2',3,5-TriCB	34	37680-68-5			
3,3',4-TriCB	35	37680-69-6			
3,3',5-TriCB	36	38444-87-0			
3,4,4'-TriCB	37	38444-90-5	$^{13}C_{12}$-3,4,4'-TriCB[2]	37L	208263-79-0
3,4,5-TriCB	38	53555-66-1			
3,4',5-TriCB	39	38444-88-1			
2,2',3,3'-TeCB	40	38444-93-8			

TABLE 1 (cont.)

CB congener[1]	IUPAC number	CAS registry number	Labeled analog	IUPAC analog	CAS registry number
2,2',3,4-TeCB	41	52663-59-9			
2,2',3,4'-TeCB	42	36559-22-5			
2,2',3,5-TeCB	43	70362-46-8			
2,2',3,5'-TeCB[7]	44	41464-39-5			
2,2',3,6-TeCB	45	70362-45-7			
2,2',3,6'-TeCB	46	41464-47-5			
2,2',4,4'-TeCB	47	2437-79-8			
2,2',4,5-TeCB	48	70362-47-9			
2,2',4,5'-TeCB	49	41464-40-8			
2,2',4,6-TeCB	50	62796-65-0			
2,2',4,6'-TeCB	51	68194-04-7			
2,2',5,5'-TeCB[7]	52	35693-99-3	$^{13}C_{12}$-2,2',5,5'-TeCB[7]	52L	208263-80-3
2,2',5,6'-TeCB	53	41464-41-9			
2,2',6,6'-TeCB	54	15968-05-5	$^{13}C_{12}$-2,2',6,6'-TeCB[7]	54L	234432-88-3
2,3,3',4'-TeCB	55	74338-24-2			
2,3,3',4'-TeCB	56	41464-43-1			
2,3,3',5-TeCB	57	70424-67-8			
2,3,3',5'-TeCB	58	41464-49-7			
2,3,3',6-TeCB	59	74472-33-6			
2,3,4,4'-TeCB	60	33025-41-1			
2,3,4,5-TeCB	61	33284-53-6			
2,3,4,6-TeCB	62	54230-22-7			
2,3,4',5-TeCB	63	74472-34-7			
2,3,4',6-TeCB	64	52663-58-8			
2,3,5,6-TeCB	65	33284-54-7			
2,3',4,4'-TeCB[7]	66	32598-10-0			
2,3',4,5-TeCB	67	73575-53-8			
2,3',4,5'-TeCB	68	73575-52-7			
2,3',4,6-TeCB	69	60233-24-1			
2,3',4',5-TeCB	70	32598-11-1			
2,3',4',6-TeCB	71	41464-46-4			
2,3',5,5'-TeCB	72	41464-42-0			
2,3',5',6-TeCB	73	74338-23-1			
2,4,4',5-TeCB	74	32690-93-0			
2,4,4',6-TeCB	75	32598-12-2			
2',3,4,5-TeCB	76	70362-48-0			
3,3',4,4'-TeCB[7]	77	32598-13-3	$^{13}C_{12}$-3,3',4,4'-TeCB[7]	77L	105600-23-5
3,3',4,5-TeCB	78	70362-49-1			
3,3',4,5'-TeCB	79	41464-48-6			
3,3',5,5'-TeCB	80	33284-52-5			
3,4,4',5-TeCB[7]	81	70362-50-4	$^{13}C_{12}$-3,4,4',5-TeCB[7]	81L	208461-24-9
2,2',3,3',4-PeCB	82	52663-62-4			
2,2',3,3',5-PeCB	83	60145-20-2			
2,2',3,3',6-PeCB	84	52663-60-2			
2,2',3,4,4'-PeCB	85	65510-45-4			
2,2',3,4,5-PeCB	86	55312-69-1			

TABLE 1 (cont.)

CB congener[1]	IUPAC number	CAS registry number	Labeled analog	IUPAC analog	CAS registry number
2,2',3,4,5'-PeCB	87	38380-02-8			
2,2',3,4,6-PeCB	88	55215-17-3			
2,2',3,4,6'-PeCB	89	73575-57-2			
2,2',3,4',5-PeCB	90	68194-07-0			
2,2',3,4',6-PeCB	91	68194-05-8			
2,2',3,5,5'-PeCB	92	52663-61-3			
2,2',3,5,6-PeCB	93	73575-56-1			
2,2',3,5,6'-PeCB	94	73575-55-0			
2,2',3,5',6-PeCB	95	38379-99-6			
2,2',3,6,6'-PeCB	96	73575-54-9			
2,2',3',4,5-PeCB	97	41464-51-1			
2,2',3',4,6-PeCB	98	60233-25-2			
2,2',4,4',5-PeCB	99	38380-01-7			
2,2',4,4',6-PeCB	100	39485-83-1			
2,2',4,5,5'-PeCB[1]	101	37680-73-2	$^{13}C_{12}$-2,2',4,5,5'-PeCB[1]	101L	104130-39-4
2,2',4,5,6'-PeCB	102	68194-06-9			
2,2',4,5',6-PeCB	103	60145-21-3			
2,2',4,6,6'-PeCB	104	56558-16-8	$^{13}C_{12}$-2,2',4,6,6'-PeCB[1]	104L	234432-89-4
2,3,3',4,4'-PeCB[1,9]	105	32598-14-4	$^{13}C_{12}$-2,3,3',4,4'-PeCB[1]	105L	208263-62-1
2,3,3',4,5-PeCB	106	70424-69-0			
2,3,3',4',5-PeCB	107	70424-68-9			
2,3,3',4,5'-PeCB	108	70362-41-3			
2,3,3',4,6-PeCB	109	74472-35-8			
2,3,3',4',6-PeCB	110	38380-03-9			
2,3,3',5,5'-PeCB	111	39635-32-0	$^{13}C_{12}$-2,3,3',5,5'-PeCB[1]	111L	235410-29-2
2,3,3',5,6-PeCB	112	74472-36-9			
2,3,3',5',6-PeCB	113	68194-10-5			
2,3,4,4',5-PeCB[1]	114	74472-37-0	$^{13}C_{12}$-2,3,4,4',5-PeCB[1]	114L	208263-63-2
2,3,4,4',6-PeCB	115	74472-38-1			
2,3,4,5,6-PeCB	116	18259-05-7			
2,3,4',5,6-PeCB	117	68194-11-6			
2,3',4,4',5-PeCB[1,9]	118	31508-00-6	$^{13}C_{12}$-2,3',4,4',5-PeCB[1]	118L	104130-40-7
2,3',4,4',6-PeCB	119	56558-17-9			
2,3',4,5,5'-PeCB	120	68194-12-7			
2,3',4,5',6-PeCB	121	56558-18-0			
2',3,3',4,5-PeCB	122	76842-07-4			
2',3,4,4',5-PeCB[1]	123	65510-44-3	$^{13}C_{12}$-2',3,4,4',5-PeCB[1]	123L	208263-64-3
2',3,4,5,5'-PeCB	124	70424-70-3			
2',3,4,5,6'-PeCB	125	74472-39-2			
3,3',4,4',5-PeCB[1,9]	126	57465-28-8	$^{13}C_{12}$-3,3',4,4',5-PeCB[1]	126L	208263-65-4
3,3',4,5,5'-PeCB	127	39635-33-1			
2,2',3,3',4,4'-HxCB[1]	128	38380-07-3			
2,2',3,3',4,5-HxCB	129	55215-18-4			
2,2',3,3',4,5'-HxCB	130	52663-66-8			
2,2',3,3',4,6-HxCB	131	61798-70-7			
2,2',3,3',4,6'-HxCB	132	38380-05-1			

TABLE 1 (cont.)

CB congener[1]	IUPAC number	CAS registry number	Labeled analog	IUPAC analog	CAS registry number
2,2',3,3',5,5'-HxCB	133	35694-04-3			
2,2',3,3',5,6-HxCB	134	52704-70-8			
2,2',3,3',5,6'-HxCB	135	52744-13-5			
2,2',3,3',6,6'-HxCB	136	38411-22-2			
2,2',3,4,4',5-HxCB	137	35694-06-5			
2,2',3,4,4',5'-HxCB[5]	138	35065-28-2	$^{13}C_{12}$-2,2',3,4,4',5'-HxCB[5]	138L	208263-66-5
2,2',3,4,4',6-HxCB	139	56030-56-9			
2,2',3,4,4',6'-HxCB	140	59291-64-4			
2,2',3,4,5,5'-HxCB	141	52712-04-6			
2,2',3,4,5,6-HxCB	142	41411-61-4			
2,2',3,4,5,6'-HxCB	143	68194-15-0			
2,2',3,4,5',6-HxCB	144	68194-14-9			
2,2',3,4,6,6'-HxCB	145	74472-40-5			
2,2',3,4',5,5'-HxCB	146	51908-16-8			
2,2',3,4',5,6-HxCB	147	68194-13-8			
2,2',3,4',5,6'-HxCB	148	74472-41-6			
2,2',3,4',5',6-HxCB	149	38380-04-0			
2,2',3,4',6,6'-HxCB	150	68194-08-1			
2,2',3,5,5',6-HxCB	151	52663-63-5			
2,2',3,5,6,6'-HxCB	152	68194-09-2			
2,2',4,4',5,5'-HxCB[5]	153	35065-27-1			
2,2',4,4',5',6-HxCB	154	60145-22-4			
2,2',4,4',6,6'-HxCB	155	33979-03-2	$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	234432-96-7
2,3,3',4,4',5-HxCB[5]	156	38380-08-4	$^{13}C_{12}$-2,3,3',4,4',5-HxCB[5]	156L	208263-68-7
2,3,3',4,4',5'-HxCB[5]	157	69782-90-7	$^{13}C_{12}$-2,3,3',4,4',5'-HxCB[5]	157L	235416-30-5
2,3,3',4,4',6-HxCB	158	74472-42-7			
2,3,3',4,5,5'-HxCB	159	39635-35-3			
2,3,3',4,5,6-HxCB	160	41411-62-5			
2,3,3',4,5',6-HxCB	161	74472-43-8			
2,3,3',4',5,5'-HxCB	162	39635-34-2			
2,3,3',4',5,6-HxCB	163	74472-44-9			
2,3,3',4',5',6-HxCB	164	74472-45-0			
2,3,3',5,5',6-HxCB	165	74472-46-1			
2,3,4,4',5,6-HxCB	166	41411-63-6			
2,3',4,4',5,5'-HxCB[5]	167	52663-72-6	$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	208263-69-8
2,3',4,4',5',6-HxCB	168	59291-65-5			
3,3',4,4',5,5'-HxCB[5,6]	169	32774-16-6	$^{13}C_{12}$-3,3',4,4',5,5'-HxCB[5]	169L	208263-70-1
2,2',3,3',4,4',5-HpCB[5]	170	35065-30-6			
2,2',3,3',4,4',6-HpCB	171	52663-71-5			
2,2',3,3',4,5,5'-HpCB	172	52663-74-8			
2,2',3,3',4,5,6-HpCB	173	68194-16-1			
2,2',3,3',4,5,6'-HpCB	174	38411-25-5			
2,2',3,3',4,5',6-HpCB	175	40186-70-7			
2,2',3,3',4,6,6'-HpCB	176	52663-65-7			
2,2',3,3',4',5,6-HpCB	177	52663-70-4			
2,2',3,3',5,5',6-HpCB	178	52663-67-9	$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L	212018-67-4

TABLE 1 (cont.)

CB congener[1]	IUPAC number	CAS registry number	Labeled analog	IUPAC analog	CAS registry number
2,2',3,3',5,6,6'-HpCB	179	52663-64-6			
2,2',3,4,4',5,5'-HpCB[3]	180	35065-29-3			
2,2',3,4,4',5,6-HpCB	181	74472-47-2			
2,2',3,4,4',5,6'-HpCB	182	60145-23-5			
2,2',3,4,4',5',6-HpCB	183	52663-69-1			
2,2',3,4,4',6,6'-HpCB	184	74472-48-3			
2,2',3,4,5,5',6-HpCB	185	52712-05-7			
2,2',3,4,5,6,6'-HpCB	186	74472-49-4			
2,2',3,4',5,5',6-HpCB[3]	187	52663-68-0			
2,2',3,4',5,6,6'-HpCB	188	74487-85-7	$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB[4]	188L	234432-91-8
2,3,3',4,4',5,5'-HpCB[3]	189	39635-31-9	$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB[4]	189L	208263-73-4
2,3,3',4,4',5,6-HpCB	190	41411-64-7			
2,3,3',4,4',5',6-HpCB	191	74472-50-7			
2,3,3',4,5,5',6-HpCB	192	74472-51-8			
2,3,3',4',5,5',6-HpCB	193	69782-91-8			
2,2',3,3',4,4',5,5'-OcCB	194	35694-08-7	$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB[4]	194L	208263-74-5
2,2',3,3',4,4',5,6-OcCB[3]	195	52663-78-2			
2,2',3,3',4,4',5,6'-OcCB	196	42740-50-1			
2,2',3,3',4,4',6,6'-OcCB	197	33091-17-7			
2,2',3,3',4,5,5',6-OcCB	198	68194-17-2			
2,2',3,3',4,5,5',6'-OcCB	199	52663-75-9			
2,2',3,3',4,5,6,6'-OcCB	200	52663-73-7			
2,2',3,3',4,5',6,6'-OcCB	201	40186-71-8			
2,2',3,3',5,5',6,6'-OcCB	202	2136-99-4	$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB[4]	202L	105600-26-8
2,2',3,4,4',5,5',6-OcCB	203	52663-76-0			
2,2',3,4,4',5,6,6'-OcCB	204	74472-52-9			
2,3,3',4,4',5,5',6-OcCB	205	74472-53-0	$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB[4]	205L	234446-64-1
2,2',3,3',4,4',5,5',6-NoCB[3]	206	40186-72-9	$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB[4]	206L	208263-75-6
2,2',3,3',4,4',5,6,6'-NoCB	207	52663-79-3			
2,2',3,3',4,5,5',6,6'-NoCB	208	52663-77-1	$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB[4]	208L	234432-92-9
DeCB[3]	209	2051-24-3	$^{13}C_{12}$-DeCB[4]	209L	105600-27-9

1. Abbreviations for Chlorination Levels

MoCB	Monochlorobiphenyl	HxCB	Hexachlorobiphenyl
DiCB	Dichlorobiphenyl	HpCB	Heptachlorobiphenyl
TrCB	Trichlorobiphenyl	OcCB	Octachlorobiphenyl
TeCB	Tetrachlorobiphenyl	NoCB	Nonachlorobiphenyl
PeCB	Pentachlorobiphenyl	DeCB	Decachlorobiphenyl

2. Labeled level of chlorination (LOC) window-defining congener

3. National Oceanic and Atmospheric Administration (NOAA) congener of interest

4. Labeled injection internal standard

5. Labeled clean up standard

6. World Health Organization (WHO) toxic congener

7. Labeled analog of WHO toxic congener

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TABLE 2
RETENTION TIMES (RT), RT REFERENCES, RELATIVE RETENTION TIMES (RRTS), ESTIMATED METHOD DETECTION LIMITS (EMDLS), AND ESTIMATED MINIMUM LEVELS (EMLS)

CI No.[1]	Congener No.[2,3]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10] Water (pg/L) EMDL	Water (pg/L) EML	Other (pg/kg) EMDL	Other (pg/kg) EML	Extract (pg/uL) EML
Compounds using 9L ($^{13}C_{12}$ 2,5-DiCB) as labeled injection internal standard												
CB congeners												
Monochlorobiphenyls												
1	1	1L	13.44	1.0012	0.9988-1.0036	-1+3	1L	82	300	8	30	10
1	2	3L	16.08	0.9878	0.9847-0.9908	6	1L/3L	4	10	0.4	1	0.5
1	3	3L	16.21	1.0010	0.9990-1.0031	-4+3	3L	88	200	9	20	10
Dichlorobiphenyls												
2	4	4L	16.40	1.0010	0.9990-1.0030	-1+3	4L	172	500	17	50	25
2	10	4L	16.53	1.0140	1.0110-1.0170	6	4L/15L	22	50	2	5	2
2	9	4L	18.55	1.1361	1.1331-1.1391	6	4L/15L	20	50	2	5	2
2	7	4L	19.07	1.1481	1.1451-1.1512	6	4L/15L	15	50	2	5	2
2	6	4L	19.26	1.1672	1.1642-1.1702	6	4L/15L	13	50	1	5	3
2	5	4L	19.48	1.1892	1.1862-1.1922	6	4L/15L	11	50	1	5	2
2	8	4L	19.56	1.1972	1.1942-1.2002	6	4L/15L	121	500	12	50	20
2	14	15L	21.32	0.9267	0.9236-0.9298	6	4L/15L	31	100	3	10	5
2	11	15L	22.42	0.9694	0.9673-0.9715	6	4L/15L	165	200	10	20	10
2	13	15L	23.03	0.9813	0.9822-0.9865	6	4L/15L	28	100	3	10	5
2	12	15L	23.06	0.9865	0.9843-0.9886	6	4L/15L					
2	13+12	15L	23.05	0.9851	0.9829-0.9872	6	4L/15L					
2	15	15L	23.26	1.0007	0.9993-1.0021	-1+3	15L	183	500	18	50	20
Trichlorobiphenyls												
3	19	19L	20.19	1.0008	0.9992-1.0025	-1+3	19L	42	100	4	10	5
3	30	19L	22.13	1.0964	1.0936-1.0985	6	19L/37L	175	500	17	50	20

TABLE 2 (cont.)

CI No.[1]	Congener No.[2,3]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
3	18	19L	22.23	1.1026	1.1002-1.1051	6	19L/37L					
3	30/18	19L	22.19	1.0995	1.0969-1.1018	6	19L/37L					
3	17	19L	22.49	1.1240	1.1215-1.1264	6	19L/37L	86	200	9	20	10
3	27	19L	23.06	1.1379	1.1355-1.1404	6	19L/37L	59	200	6	20	10
3	24	19L	23.15	1.1445	1.1420-1.1470	6	19L/37L	53	200	5	20	10
3	16	19L	23.25	1.1535	1.1511-1.1560	6	19L/37L	35	100	4	10	5
3	32	19L	24.57	1.2291	1.2266-1.2315	6	19L/37L	84	200	8	20	10
3	34	19L	25.17	1.2455	1.2430-1.2479	6	19L/37L	74	200	7	20	10
3	23	19L	25.26	1.2529	1.2504-1.2553	6	19L/37L	50	200	5	20	10
3	29	19L	25.47	1.2701	1.2668-1.2742	10	19L/37L	83	200	8	20	10
3	26	19L	25.48	1.2709	1.2668-1.2750	10	19L/37L					
3	26/29	19L	25.48	1.2709	1.2668-1.2750	10	19L/37L					
3	25	37L	26.04	0.8364	0.8348-0.8380	6	19L/37L	55	200	5	20	10
3	31	37L	26.25	0.8476	0.8460-0.8492	6	19L/37L	152	500	15	50	20
3	28	37L	26.44	0.8578	0.8551-0.8604	10	19L/37L	192	500	19	50	20
3	20	37L	26.49	0.8604	0.8578-0.8631	10	19L/37L					
3	28/20	37L	26.47	0.8594	0.8567-0.8620	10	19L/37L					
3	21	37L	26.88	0.8652	0.8626-0.8679	10	19L/37L	51	200	5	20	10
3	33	37L	27.01	0.8668	0.8642-0.8695	10	19L/37L					
3	21/33	37L	26.96	0.8658	0.8631-0.8684	10	19L/37L					
3	22	37L	27.29	0.8818	0.8802-0.8834	6	19L/37L	90	200	9	20	10
3	36	37L	29.03	0.9332	0.9316-0.9348	6	19L/37L	79	200	8	20	10
3	39	37L	29.36	0.9465	0.9449-0.9481	6	19L/37L	83	200	8	20	10
3	38	37L	30.10	0.9679	0.9663-0.9695	6	19L/37L	83	200	8	20	10
3	35	37L	30.42	0.9830	0.9833-0.9866	6	19L/37L	77	200	8	20	10
3	37	37L	31.11	1.0005	0.9995-1.0014	193	37L	132	500	13	50	20

TABLE 2 (cont.)

CI No.[1]	Congener No.[2,3]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10] Water (pg/L) EMDL	Water (pg/L) EML	Other (ng/kg) EMDL	Other (ng/kg) EML	Extract (pg/uL) EML
	Labeled Compounds											
1	1L	9L	13:43	0.7257	0.7125-0.7390	30	9L					
1	3L	9L	16:20	0.8612	0.8510-0.8774	30	9L					
2	4L	9L	16:39	0.8810	0.8677-0.8942	30	9L					
2	15L	9L	23:25	1.2390	1.2302-1.2478	20	9L					
3	19L	9L	20:18	1.0741	1.0608-1.0873	30	9L					
3	37L	52L	31:10	1.0841	1.0754-1.0928	30	52L					
Compounds using 52L ($^{13}C_{12}$-2,2',5,5'-TeCB) as Labeled Injection Internal standard												
	CB congener											
	Tetrachlorobiphenyls											
1	54	54L	23:51	1.0007	0.9993-1.0021	41-3	54L	118	500	12	50	20
4	50	54L	26:07	1.0958	1.0923-1.0993	10	54L/81L/77L	58	200	6	20	10
4	53	54L	26:09	1.0972	1.0937-1.1007	10	54L/81L/77L					
4	50/53	54L	26:08	1.0965	1.0930-1.1000	10	54L/81L/77L					
4	45	54L	26:55	1.1294	1.1259-1.1329	10	54L/81L/77L	51	200	5	20	10
4	51	54L	26:58	1.1315	1.1280-1.1350	10	54L/81L/77L					
4	45/51	54L	26:57	1.1308	1.1273-1.1343	10	54L/81L/77L					
4	46	54L	27:18	1.1455	1.1434-1.1476	6	54L/81L/77L	101	200	10	20	10
4	52	54L	28:45	1.2063	1.2042-1.2084	6	54L/81L/77L	191	500	19	50	20
4	73	54L	28:52	1.2112	1.2091-1.2133	6	54L/81L/77L	160	500	16	50	20
4	43	54L	28:58	1.2154	1.2133-1.2175	6	54L/81L/77L	94	200	9	20	10
4	69	54L	29:08	1.2224	1.2189-1.2259	10	54L/81L/77L	115	500	11	50	20
4	49	54L	29:16	1.2280	1.2245-1.2315	10	54L/81L/77L					
4	69/49	54L	29:12	1.2252	1.2217-1.2287	10	54L/81L/77L					
4	48	54L	29:33	1.2399	1.2378-1.2420	6	54L/81L/77L	76	200	8	20	10

TABLE 2 (cont.)

CI No.[1]	Congener No.[23]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/uL)
								EMDL	EML	EMDL	EML	EML
4	65	54L	29:49	1.2510	1.2476-1.2545	10	54L/81L/77L	195	500	19	50	20
4	47	54L	29:50	1.2517	1.2483-1.2552	10	54L/81L/77L					
4	44	54L	29:53	1.2538	1.2503-1.2573	10	54L/81L/77L					
4	114/105	54L	29:50	1.2517	1.2483-1.2552	10	54L/81L/77L					
4	62	54L	30:06	1.2629	1.2594-1.2664	10	54L/81L/77L	57	200	6	20	10
4	78	54L	30:08	1.2643	1.2608-1.2678	10	54L/81L/77L					
4	59	54L	30:12	1.2671	1.2636-1.2706	10	54L/81L/77L					
4	59/62/75	54L	30:09	1.2650	1.2615-1.2685	10	54L/81L/77L					
4	42	54L	30:26	1.2769	1.2748-1.2790	6	54L/81L/77L	61	200	6	20	10
4	41	54L	30:52	1.2951	1.2916-1.2986	10	54L/81L/77L	219	500	17	50	20
4	71	54L	30:58	1.2993	1.2958-1.3028	10	54L/81L/77L					
4	40	54L	31:01	1.3014	1.2979-1.3049	10	54L/81L/77L					
4	41/40/71	54L	30:58	1.2993	1.2958-1.3028	10	54L/81L/77L					
4	64	54L	31:12	1.3091	1.3070-1.3113	6	54L/81L/77L	70	200	7	20	10
4	72	81L	31:39	0.8336	0.8323-0.8349	6	54L/81L/77L	158	500	16	50	20
4	68	81L	32:18	0.8419	0.8406-0.8432	6	54L/81L/77L	149	500	15	50	20
4	57	81L	32:36	0.8540	0.8527-0.8553	6	54L/81L/77L	125	500	12	50	20
4	58	81L	33:05	0.8623	0.8610-0.8636	6	54L/81L/77L	127	500	13	50	20
4	67	81L	33:13	0.8658	0.8645-0.8671	6	54L/81L/77L	137	500	13	50	20
4	63	81L	33:30	0.8732	0.8719-0.8745	6	54L/81L/77L	138	500	14	50	20
4	65	81L	33:46	0.8801	0.8775-0.8827	12	54L/81L/77L	171	500	17	50	20
4	70	81L	33:53	0.8834	0.8805-0.8858	12	54L/81L/77L					
4	76	81L	33:55	0.8840	0.8814-0.8866	12	54L/81L/77L					
4	74	81L	33:57	0.8849	0.8827-0.8871	10	54L/81L/77L					
4	61/70/76	81L	33:55	0.8840	0.8814-0.8866	12	54L/81L/77L					
4	66	81L	34:15	0.8927	0.8914-0.8940	6	54L/81L/77L	162	500	16	50	20

TABLE 2 (cont.)

CI No.[1]	Congener No.[2,3]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation references[9]	Detection limits and minimum levels - Matrix and concentration[10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/uL)
								EMDL	EML	EMDL	EML	EML
4	55	81L	34.28	0.8983	0.8970-0.8997	6	52L,81L,77L	129	500	12	50	20
4	56	81L	35.03	0.9136	0.9123-0.9149	6	52L,81L,77L	98	500	10	20	10
4	60	81L	35.16	0.9192	0.9179-0.9205	6	52L,81L,77L	131	500	13	50	20
4	80	81L	35.32	0.9262	0.9248-0.9275	6	52L,81L,77L	173	500	18	50	20
4	79	81L	37.16	0.9713	0.9700-0.9726	6	52L,81L,77L	172	500	17	50	20
4	78	81L	37.52	0.9870	0.9857-0.9883	6	52L,81L,77L	171	500	17	50	20
4	81	81L	38.23	1.0000	0.9986-1.0013	4-5	81L	177	500	18	50	20
4	77	77L	39.02	1.0000	0.9986-1.0013	4-5	77L	169	500	17	50	20
	Labeled compounds											
4	52L	52L	23.45	0.8200	0.8232-0.8308	20	52L					
4	81L	52L	38.22	1.3345	1.3287-1.3403	20	52L					
4	77L	52L	39.01	1.3571	1.3513-1.3629	20	52L					
Compounds using 101L ($^{13}C_{12}$-2,3',4,5,5'-PeCB) as Labeled injection internal standard												
	CB congener											
	Pentachlorobiphenyls											
5	104	101L	29.46	1.0000	0.9984-1.0017	4-5	101L	223	500	23	50	20
5	96	101L	30.17	1.0234	1.0216-1.0252	10	101L,123L,114L,118L	210	500	21	50	20
5	103	101L	32.14	1.0812	1.0795-1.0829	6	101L,123L,114L,118L	225	500	23	50	20
5	94	101L	32.29	1.0913	1.0896-1.0929	6	101L,123L,114L,118L	121	500	12	50	20
5	95	101L	32.00	1.0806	1.0788-1.0824	10	101L,123L,114L,118L	221	500	22	50	20
5	100	101L	33.06	1.1220	1.1202-1.1238	10	101L,123L,114L,118L					
5	93	101L	33.11	1.1265	1.1247-1.1283	10	101L,123L,114L,118L					
5	102	101L	33.21	1.1204	1.1176-1.1232	10	101L,123L,114L,118L					
5	98	101L	33.26	1.1272	1.1244-1.1300	10	101L,123L,114L,118L					
5	95/100/98/102/98	101L	33.13	1.1250	1.1222-1.1287	15	101L,123L,114L,118L					

TABLE 2 (cont.)

Cl No.[1]	Congener No.[2,3]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/uL)
								EMDL	EML	EMDL	EML	EML
5	88	104L	33:48	1.1355	1.1321-1.1389	12	104L/123L/114L/118L	118	500	12	50	20
5	91	104L	33:53	1.1394	1.1366-1.1422	10	104L/123L/114L/118L					
5	88/91	104L	33:52	1.1377	1.1344-1.1411	12	104L/123L/114L/118L					
5	84	104L	34:14	1.1501	1.1483-1.1517	6	104L/123L/114L/118L	124	500	12	50	20
5	89	104L	34:41	1.1669	1.1652-1.1685	6	104L/123L/114L/118L	195	500	19	50	20
5	121	104L	34:57	1.1745	1.1725-1.1758	6	104L/123L/114L/118L	209	500	27	50	20
5	92	123L	35:26	0.8639	0.8627-0.8651	6	104L/123L/114L/118L	115	500	12	50	20
5	113	123L	36:01	0.8781	0.8761-0.8801	10	104L/123L/114L/118L	241	1000	24	100	50
5	90	123L	36:03	0.8789	0.8769-0.8809	10	104L/123L/114L/118L					
5	101	123L	36:04	0.8793	0.8773-0.8813	10	104L/123L/114L/118L					
5	113/90/101	123L	36:03	0.8789	0.8769-0.8809	10	104L/123L/114L/118L					
5	83	123L	36:39	0.8933	0.8911-0.8960	12	104L/123L/114L/118L	217	500	27	50	20
5	99	123L	36:41	0.8943	0.8923-0.8963	10	104L/123L/114L/118L					
5	83/99	123L	36:40	0.8939	0.8915-0.8964	12	104L/123L/114L/118L					
5	112	123L	36:51	0.8983	0.8972-0.8996	6	104L/123L/114L/118L	245	1000	25	100	50
5	119	123L	37:12	0.9069	0.9037-0.9102	16	104L/123L/114L/118L	140	500	15	50	20
5	109	123L	37:12	0.9069	0.9037-0.9102	16	104L/123L/114L/118L					
5	86	123L	37:17	0.9089	0.9057-0.9122	16	104L/123L/114L/118L					
5	97	123L	37:17	0.9089	0.9057-0.9122	16	104L/123L/114L/118L					
5	125	123L	37:21	0.9106	0.9073-0.9139	16	104L/123L/114L/118L					
5	87	123L	37:25	0.9122	0.9105-0.9113	10	104L/123L/114L/118L					
5	109/119/86/97/125/87	123L	37:19	0.9098	0.9065-0.9130	16	104L/123L/114L/118L					
5	117	123L	37:57	0.9252	0.9228-0.9277	12	104L/123L/114L/118L	104	500	10	50	10
5	116	123L	38:02	0.9273	0.9248-0.9297	12	104L/123L/114L/118L					
5	85	123L	38:05	0.9283	0.9265-0.9303	10	104L/123L/114L/118L					
5	116/85	123L	38:08	0.9295	0.9280-0.9309	12	104L/123L/114L/118L					

TABLE 2 (cont.)

CI No.[1]	Congener No.[2,3]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
5	110	123L	38.16	0.9330	0.9309-0.9350	10	104L/123L/114L/118L	242	1000	24	100	50
5	115	123L	38.18	0.9338	0.9317-0.9358	10	104L/123L/114L/118L					
5	110/115	123L	38.17	0.9334	0.9313-0.9355	10	104L/123L/114L/118L					
5	82	123L	38.40	0.9427	0.9415-0.9439	6	104L/123L/114L/118L	133	500	13	50	20
5	111	123L	38.52	0.9476	0.9464-0.9488	6	104L/123L/114L/118L	242	1000	24	100	50
5	120	123L	38.81	0.9594	0.9581-0.9606	6	104L/123L/114L/118L	147	500	15	50	20
5	108	123L	40.39	0.9911	0.9890-0.9931	10	104L/123L/114L/118L	200	1000	27	100	50
5	124	123L	40.40	0.9915	0.9894-0.9935	10	104L/123L/114L/118L					
5	108/124	123L	40.39	0.9911	0.9890-0.9931	10	104L/123L/114L/118L					
5	107	123L	40.54	0.9972	0.9959-0.9984	6	104L/123L/114L/118L	103	200	10	20	10
5	123	123L	41.02	1.0004	0.9996-1.0012	-1+3	123L	150	500	15	50	20
5	106	123L	41.10	1.0037	1.0024-1.0049	6	104L/123L/114L/118L	143	500	14	50	20
5	118	118L	41.22	1.0004	0.9996-1.0012	-1+3	118L	193	500	19	50	20
5	122	118L	41.49	1.0113	1.0101-1.0125	6	104L/123L/114L/118L	117	500	12	50	20
5	114	114L	41.58	1.0004	0.9999-1.0012	-1+3	114L	120	500	12	50	20
5	105	105L	42.43	0.9996	0.9992-1.0012	-2+3	105L	109	200	11	20	10
5	127	105L	44.09	1.0332	1.0320-1.0343	6	104L/123L/114L/118L	278	1000	28	100	50
5	126	126L	45.58	1.0004	0.9996-1.0011	-1+3	126L	136	500	14	50	20
	Labeled compounds											
5	104L	101L	29.46	0.8257	0.8211-0.8303	20	101L					
5	123L	101L	41.09	1.1378	1.1331-1.1424	20	101L					
5	118L	101L	41.21	1.1470	1.1424-1.1516	20	101L					
5	114L	101L	41.57	1.1637	1.1590-1.1683	20	101L					
5	105L	101L	42.44	1.1854	1.1808-1.1900	20	101L					
5	126L	101L	45.57	1.2746	1.2700-1.2792	20	101L					

Compounds using 13B1, ($^{13}C_{12}$-2,2',3,4,4',5'-Hx(CB) as Labeled Injection Internal standard

TABLE 2 (cont.)

CI No.[1]	Congener No.[2,3]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10] Water (pg/L) EMDL	Water (pg/L) EML	Other (ng/kg) EMDL	Other (ng/kg) EML	Extract (pg/uL) EML
	CB congener											
	Hexachlorobiphenyls											
6	155	155L	35.33	1.0000	0.9995-1.0014	3+3	155L	336	1000	34	100	50
6	152	155L	36.27	1.0107	1.0093-1.0121	6	155L/156L/157L/167L/169L	238	1000	24	100	50
6	150	155L	36.13	1.0145	1.0131-1.0159	6	155L/156L/157L/167L/169L	328	1000	33	100	50
6	136	155L	36.44	1.0289	1.0266-1.0294	6	155L/156L/157L/167L/169L	94	200	9	20	10
6	145	155L	37.00	1.0354	1.0340-1.0368	6	155L/156L/157L/167L/169L	317	1000	32	100	50
6	148	155L	34.26	1.0756	1.0742-1.0770	6	155L/156L/157L/167L/169L	324	1000	32	100	50
6	151	155L	39.10	1.0961	1.0938-1.0984	10	155L/156L/157L/167L/169L	112	500	11	50	20
6	135	155L	39.17	1.0993	1.0970-1.1017	10	155L/156L/157L/167L/169L					
6	134	155L	39.21	1.1012	1.0989-1.1035	10	155L/156L/157L/167L/169L					
6	131/135/134	155L	39.15	1.0984	1.0961-1.1007	10	155L/156L/157L/167L/169L					
6	144	155L	39.47	1.1133	1.1110-1.1157	6	155L/156L/157L/167L/169L	167	500	17	50	20
6	147	155L	40.09	1.1236	1.1213-1.1259	10	155L/156L/157L/167L/169L	179	500	18	50	20
6	149	155L	40.12	1.1250	1.1227-1.1273	10	155L/156L/157L/167L/169L					
6	147/149	155L	40.10	1.1241	1.1217-1.1264	10	155L/156L/157L/167L/169L					
6	133	155L	40.27	1.1320	1.1297-1.1343	10	155L/156L/157L/167L/169L	134	500	13	50	20
6	143	155L	40.30	1.1334	1.1311-1.1357	10	155L/156L/157L/167L/169L					
6	133/143	155L	40.29	1.1329	1.1306-1.1353	10	155L/156L/157L/167L/169L					
6	139	155L	40.37	1.1413	1.1390-1.1437	10	155L/156L/157L/167L/169L	196	500	20	50	20
6	140	155L	40.48	1.1418	1.1395-1.1441	10	155L/156L/157L/167L/169L					
6	139/140	155L	40.47	1.1413	1.1390-1.1437	10	155L/156L/157L/167L/169L					
6	130	155L	41.05	1.1688	1.1674-1.1702	6	155L/156L/157L/167L/169L	121	500	12	50	20
6	142	155L	41.13	1.1535	1.1521-1.1549	6	155L/156L/157L/167L/169L	311	1000	31	100	50
6	132	155L	41.36	1.1612	1.1618-1.1665	10	155L/156L/157L/167L/169L	127	500	12	50	20
6	133	155L	41.47	1.1740	1.1726-1.1754	6	155L/156L/157L/167L/169L	166	500	17	50	20

TABLE 2 (cont.)

CI No.[1]	Congener No.[2]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
o	165	167L	42.23	0.8861	0.8853-0.8871	6	155L/156L/157L/167L/169L	361	1000	36	100	50
o	146	167L	42.38	0.8916	0.8906-0.8926	6	155L/156L/157L/167L/169L	182	500	18	50	20
o	161	167L	42.47	0.8917	0.8937-0.8958	6	155L/156L/157L/167L/169L	252	1000	35	100	50
o	153	167L	43.17	0.9052	0.9035-0.9069	10	155L/156L/157L/167L/169L	130	500	13	50	20
o	168	167L	43.21	0.9066	0.9048-0.9083	10	155L/156L/157L/167L/169L					
o	153/168	167L	43.19	0.9059	0.9041-0.9076	10	155L/156L/157L/167L/169L					
o	141	167L	43.34	0.9111	0.9101-0.9122	6	155L/156L/157L/167L/169L	93	200	9	20	10
o	130	167L	44.01	0.9203	0.9195-0.9216	6	155L/156L/157L/167L/169L	136	500	14	50	20
o	137	167L	44.14	0.9251	0.9240-0.9261	6	155L/156L/157L/167L/169L	300	1000	30	100	50
o	164	167L	44.22	0.9278	0.9268-0.9289	6	155L/156L/157L/167L/169L	136	500	14	50	20
o	138	167L	44.42	0.9348	0.9324-0.9373	14	155L/156L/157L/167L/169L	211	500	21	50	20
o	163	167L	44.42	0.9348	0.9324-0.9373	14	155L/156L/157L/167L/169L					
o	129	167L	44.47	0.9366	0.9341-0.9390	14	155L/156L/157L/167L/169L					
o	160	167L	44.53	0.9387	0.9369-0.9404	10	155L/156L/157L/167L/169L					
o	138/163/129/160	167L	44.47	0.9366	0.9341-0.9390	14	155L/156L/157L/167L/169L					
o	158	167L	45.05	0.9428	0.9418-0.9439	6	155L/156L/157L/167L/169L	96	200	10	20	10
o	166	167L	45.50	0.9617	0.9599-0.9634	10	155L/156L/157L/167L/169L	124	500	12	50	20
o	128	167L	46.00	0.9651	0.9634-0.9669	10	155L/156L/157L/167L/169L					
o	128/166	167L	46.04	0.9634	0.9617-0.9651	10	155L/156L/157L/167L/169L					
o	159	167L	46.59	0.9826	0.9815-0.9836	6	155L/156L/157L/167L/169L	348	1000	35	100	50
o	162	167L	47.18	0.9892	0.9881-0.9902	6	155L/156L/157L/167L/169L	355	1000	35	100	50
o	167	167L	47.49	1.0000	0.9997-1.0010	-1+3	167L	115	500	11	50	20
o	156	156L/157L	49.05	0.9993	0.9983-1.0003	6	156L/157L	132	500	13	50	20
o	157	156L/157L	49.09	1.0007	0.9996-1.0021	10	156L/157L					

TABLE 2 (cont.)

CI No.[3]	Congener No.[23]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10] Water (pg/L) PMDL	Water (pg/L) EML	Other (pg/kg) PMDL	Other (pg/kg) EML	Extract (pg/uL) EML
o	156/157	1561/157 1	55.07	1.0000	0.9990-1.0010	6	1562/157L					
o	169	169L	52.31	1.0003	0.9997-1.0010	4-13	169L	161	500	16	55	20
	Labeled compounds											
o	155	155L	55.41	[illegible]	[illegible]	20	155L					
o	167L	156L	47.59	1.0701	1.0651-1.0750	20	156L					
o	156L	156L	49.05	1.0985	1.0934-1.0996	20	156L					
o	157L	156L	49.08	1.0996	1.0959-1.1033	20	156L					
o	156L/157L	156L	49.07	1.0992	1.0981-1.1033	20	156L					
o	169L	169L	52.35	1.1749	1.1738-1.1761	20	169L					
Compounds using 1MLC($^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB) as Labeled Injection Internal standard												
	CB congener											
	Heptachlorobiphenyls											
7	188	188L	41.51	1.0000	0.9986-1.0012	4-13	188L	235	500	23	50	20
7	179	188L	42.19	1.0112	1.0106-1.0123	o	188L/189L	229	500	23	50	20
7	181	188L	42.45	1.0215	1.0203-1.0227	o	188L/189L	463	1000	46	100	50
7	176	188L	43.15	1.0335	1.0323-1.0346	o	188L/189L	384	1000	38	100	50
7	186	188L	43.45	1.0454	1.0442-1.0466	o	188L/189L	407	1000	41	100	50
7	178	188L	43.06	1.0277	1.0266-1.0289	o	188L/189L	221	500	22	50	20
7	175	188L	43.46	1.0436	1.0425-1.0448	o	188L/189L	853	1000	85	100	50
7	187	188L	46.02	1.1000	1.0988-1.1012	o	188L/189L	191	500	19	50	20
7	182	188L	46.14	1.1017	1.1005-1.1030	o	188L/189L	405	1000	40	100	50
7	183	188L	46.42	1.1139	1.1127-1.1151	o	188L/189L	501	1000	50	100	50
7	185	188L	46.53	1.1265	1.1253-1.1278	o	188L/189L					
7	183/185	188L	46.47	1.1170	1.1158-1.1181	o	188L/189L					

TABLE 2 (cont.)

CI No.[2]	Congener No.[23]	RT Ref[3]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10] Water (pg/L) EMDL	Water (pg/L) EML	Other (ng/kg) EMDL	Other (ng/kg) EML	Extract (pg/uL) EML
7	174	188L	47.02	1.1239	1.1227-1.1251	6	188L/189L	186	500	19	50	20
7	177	188L	47.36	1.1380	1.1368-1.1392	6	188L/189L	141	500	14	50	20
7	181	188L	47.52	1.1438	1.1426-1.1450	6	188L/189L	396	1000	40	100	50
7	171	188L	48.10	1.1509	1.1489-1.1529	10	188L/189L	354	1000	37	100	50
7	173	188L	48.11	1.1513	1.1501-1.1525	6	188L/189L					
7	171/173	188L	48.10	1.1509	1.1489-1.1529	10	188L/189L					
7	172	189L	49.47	0.9035	0.9026-0.9044	6	188L/189L	377	1000	38	100	50
7	192	189L	50.06	0.9093	0.9083-0.9102	6	188L/189L	420	1000	42	100	50
7	193	189L	50.26	0.9153	0.9144-0.9162	6	188L/189L	186	500	14	50	20
7	189	189L[11]	50.27	0.9156	0.9147-0.9165	6	188L/189L[11]					
7	189/193	189L	50.26	0.9153	0.9144-0.9162	6	188L/189L					
7	191	189L	50.51	0.9229	0.9220-0.9238	6	188L/189L	118	1000	12	100	50
7	170	189L[11]	51.54	0.9419	0.9410-0.9428	6	188L/189L[11]	162	500	16	50	20
7	190	189L	52.26	0.9516	0.9507-0.9525	6	188L/189L	234	500	23	50	20
7	189	189L	55.07	1.0003	0.9997-1.0009	+1+3	189L	177	500	18	50	20
	Octachlorobiphenyls											
8	202	202L	47.32	1.0004	0.9996-1.0011	-1+3	202L	412	1000	41	100	50
8	201	202L	48.31	1.0210	1.0193-1.0228	10	202L/205L	440	1000	44	100	50
8	204	202L	49.11	1.0351	1.0340-1.0361	6	202L/205L	447	1000	45	100	50
8	197	202L	49.27	1.0407	1.0396-1.0417	6	202L/205L	245	1000	25	100	50
8	200	202L	49.40	1.0452	1.0442-1.0463	6	202L/205L					
8	197/200	202L	49.33	1.0428	1.0417-1.0438	6	202L/205L					
8	198	202L	52.30	1.1019	1.1031-1.1066	10	202L/205L	503	500	20	50	20
8	199	202L	52.32	1.1056	1.1045-1.1066	6	202L/205L					
8	198/199	202L	52.31	1.1052	1.1035-1.1070	10	202L/205L					
8	196	205L	53.13	0.9207	0.9198-0.9216	6	202L/205L	429	1000	43	100	50

TABLE 2 (cont.)

CI No.[1]	Congener No.[2,3]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
2	203	205L	53.26	0.9215	0.9256-0.9253	6	202L, 205L	444	1000	14	100	50
4	195	205L	54.55	0.9501	0.9493-0.9510	6	202L, 205L	427	1000	43	100	50
6	193	205L	57.19	0.9910	0.9968-0.9925	6	202L, 205L	170	500	17	50	20
8	205	205L	57.39	1.0063	0.9987-1.0009	-2+3	205L	449	1000	45	100	50
	Nonachlorobiphenyls											
6	208	208L	54.33	1.0000	0.9937-1.0060	-1+3	206L	355	1000	36	100	50
7	207	208L	55.32	1.0183	1.0137-1.0193	6	208L, 206L	453	1000	45	100	50
9	206	206L	59.37	1.0003	0.9937-1.0006	-1+3	206L	451	1000	45	100	50
	Decachlorobiphenyl											
10	209	209L	61.15	1.0003	0.9997-1.0008	-1+3	209L	153	500	15	50	25
	Labeled compounds											
3	188L	194L	51.54	0.7364	0.7275-0.7333	20	195L					
5	180L	194L	50.27	0.8605	0.8775-0.8633	20	195L					
7	170L	194L	51.53	0.9059	0.9026-0.9084	20	195L					
9	189L	194L	55.16	0.9688	0.9587-0.9645	20	195L					
11	202L	194L	47.31	0.8293	0.8264-0.8372	20	195L					
13	205L	194L	57.28	1.0087	1.0044-1.0131	20	195L					
14	208L	194L	54.32	0.9517	0.9488-0.9546	20	195L					
15	206L	194L	59.36	1.0401	1.0358-1.0445	20	195L					
16	209L	194L	61.11	1.0646	1.0633-1.0730	20	195L					
Labeled clean-up standards												
3	28L	52L	29.11	0.9266	0.9209-0.9321	20	52L					
5	111L	101L	38.40	1.0777	1.0738-1.0823	20	101L					
7	138L	138L	43.95	1.0000	1.0055-1.0127	20	138L					
Labeled injection internal standards												
2	9L	139L	18.54	0.1270	0.1186-0.1276	25	139L					

TABLE 2 (cont.)

CI No.[1]	Congener No.[2,3]	RT Ref[4]	RT[5]	RRT[6]	RRT limits[7]	Window (sec)[8]	Quantitation reference[9]	Detection limits and minimum levels - Matrix and concentration[10]				
								Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
								EMDL	EML	EMDL	EML	EML
1	52L	138L	28:35	0.6431	0.5886-0.6401	25	138L					
7	101L	138L	36:03	0.8000	0.8021-0.8115	25	138L					
9	138L	138L	44:41	1.0000	0.9966-1.0011	100	138L					
8	153L	138L	57:06	1.2824	1.2775-1.2870	25	138L					

1. Number of chlorines on congener.
2. Suffix "L" indicates labeled compound.
3. Multiple congeners in a box indicates a group of congeners that co-elute or may not be adequately resolved on a 30-m SPB-Octyl column. Congeners included in the group are listed as the last entry in the box.
4. Retention time (RT) reference used to locate target congener.
5. Retention time of target congener.
6. Relative retention time (RRT) between the RT for the congener and RT for the reference.
7. RRT limits based on RT window.
8. RT window width necessary to attempt to unambiguously identify the congener in the presence of other congeners.
9. Labeled congeners that form the quantitation reference. Areas from the exact m/z's of the congeners listed in the quantitation reference are summed, and divided by the number of congeners in the quantitation reference. For example, for congener 10, the areas at the exact m/z's for 4L and 15L are summed and the sum is divided by 2 (because there are 2 congeners in the quantitation reference).
10. LMDLs and EMLs (MRLs) with common laboratory interferences present, according to reference method 1668A. Without interferences, LMDLs and EMLs (MRLs) will be, respectively, 5 and 10pg/L for aqueous samples, 0.5 and 1.0ng/kg for soil, tissue, and mixed-phase samples, and EMLs for extracts will be 0.5pg/µL.
11. If congeners 170L and 180L are included in the calibration and spiking solution, these congeners should be used as RT and quantitation references.
12. For 100µL extracts, the LMDL and EML concentrations must be increased by a factor of five (5).

TABLE 3
CONCENTRATIONS OF NATIVE AND LABELED CHLORINATED BIPHENYLS IN STOCK SOLUTIONS, SPIKING SOLUTIONS, AND FINAL EXTRACTS

	Solution Concentrations		
CB Congener	Stock (µg/mL)	Spiking (ng/mL)	Extract (ng/mL)
Native Toxics/LOC			
1	20	5.0	50
3	20	5.0	50
4	20	5.0	50
15	20	5.0	50
19	20	5.0	50
37	20	5.0	50
54	20	5.0	50
77	20	5.0	50
81	20	5.0	50
104	20	5.0	50
105	20	5.0	50
114	20	5.0	50
118	20	5.0	50
123	20	5.0	50
126	20	5.0	50
155	20	5.0	50
156	20	5.0	50
157	20	5.0	50
167	20	5.0	50
169	20	5.0	50
188	20	5.0	50
189	20	5.0	50
202	20	5.0	50
205	20	5.0	50
206	20	5.0	50
208	20	5.0	50
209	20	5.0	50
Native congener mix stock solutions			
MoCB thru TrCB	2.5		
TeCB thru HpCB	5.0		
OcCB thru DeCB	7.5		
Labeled Toxics/LOC/window-defining			
1L	1.0	10.0	100
3L	1.0	10.0	100
4L	1.0	10.0	100
15L	1.0	10.0	100
19L	1.0	10.0	100
37L	1.0	10.0	100
54L	1.0	10.0	100
77L	1.0	10.0	100
81L	1.0	10.0	100

TABLE 3 (cont.)

104L	1.0	10.0	100
105L	1.0	10.0	100
114L	1.0	10.0	100
118L	1.0	10.0	100
123L	1.0	10.0	100
126L	1.0	10.0	100
155L	1.0	10.0	100
156L	1.0	10.0	100
157L	1.0	10.0	100
167L	1.0	10.0	100
169L	1.0	10.0	100
188L	1.0	10.0	100
189L	1.0	10.0	100
202L	1.0	10.0	100
205L	1.0	10.0	100
206L	1.0	10.0	100
208L	1.0	10.0	100
209L	1.0	10.0	100
Labeled Clean Up			
28L	1.0	10.0	100
111L	1.0	10.0	100
178L	1.0	10.0	100
Labeled Injection Internal			
9L	5.0	5000	100
52L	5.0	5000	100
101L	5.0	5000	100
138L	5.0	5000	100
194L	5.0	5000	100
Diluted combined 209 congener solution	**Solution Concentration (µg/mL)**		
Native congeners			
MoCB thru TrCB	50		
TeCB thru HpCB	100		
OcCB thru DeCB	150		
Labeled Toxics/LOC/window-defining	100		
Labeled Clean Up	100		
Labeled Injection Internal	100		

1. Section 8.20
2. Section 8.16
3. Section 8.17
4. Section 8.19
5. Section 8.18
6. Section 8.16

TABLE 4

COMPOSITION OF INDIVIDUAL NATIVE CB CONGENER SOLUTIONS[1]

AccuStandard Catalog Number								
M-1668A-1			M-1668A-2		M-1668A-3		M-1668A-4	M-1668A-5
2	78	161	7	117	13	86	25	1
10	81	153	5	111	17	116	21	3
9	96	130	12	107/108	29	109/107	69	4
6	103	129	18	118	20	154	47	15
8	95	166	24	114	46	147	42	19
14	88	159	23	150	65	140	64	16
11	89	167	28	145	59	146	70	37
30	92	156	22	135	40	141	102	54
27	113	179	39	149	67	164	97	43
32	83	176	53	139	76	158	115	44
34	119	178	51	132	80	182	123	74
26	87	175	73	165	93	174	134	56
31	85	183	48	168	84	173	131	77
33	82	177	62	137	101	193	163	104
36	120	171	71	160	112		180	98
38	124	172	68	128				125
35	106	191	58	162				110
50	122	170	61	157				126
45	105	190	55	184				155
52	127	201/200	60	186				138
49	152	204	94	187				169
75	136	200/199	100	185				188
41	148	198	91	181				189
72	151	196	121	192				202
57	144	195	90	197				205
63	143	194	99	199/201				208
66	142	207	108/109	203				206
79	133							209
Total Number of Congeners								
83			54		29		15	28

1. Congeners present in each standard solution are listed in elution order for each level of chlorination. See Table 3 for concentrations of congeners in stock solutions and Table 5 for concentrations in calibration standards.

TABLE 5
CONCENTRATION OF CB CONGENERS IN CALIBRATION AND CALIBRATION VERIFICATION STANDARDS

		Solution concentration (ng/mL)					
CB congener	IUPAC[1]	CS-0.2 (Hi sens)[2]	CS-1	CS-2	CS-3 (VER)	CS-4	CS-5
Native Toxics/LOC							
2-MoCB	1	0.2	1.0	5.0	50	400	2000
4-MoCB	3	0.2	1.0	5.0	50	400	2000
2,2'-DiCB	4	0.2	1.0	5.0	50	400	2000
4,4'-DiCB	15	0.2	1.0	5.0	50	400	2000
2,2',6'-TriCB	19	0.2	1.0	5.0	50	400	2000
3,4,4'-TriCB	37	0.2	1.0	5.0	50	400	2000
2,2',6,6'-TeCB	54	0.2	1.0	5.0	50	400	2000
3,3',4,4'-TeCB	77	0.2	1.0	5.0	50	400	2000
3,4,4',5-TeCB	81	0.2	1.0	5.0	50	400	2000
2,2',4,6,6'-PeCB	104	0.2	1.0	5.0	50	400	2000
2,3,3',4,4'-PeCB	105	0.2	1.0	5.0	50	400	2000
2,3,4,4',5-PeCB	114	0.2	1.0	5.0	50	400	2000
2,3',4,4',5-PeCB	118	0.2	1.0	5.0	50	400	2000
2',3,4,4',5-PeCB	123	0.2	1.0	5.0	50	400	2000
3,3',4,4',5-PeCB	126	0.2	1.0	5.0	50	400	2000
2,2',4,4',6,6'-HxCB	155	0.2	1.0	5.0	50	400	2000
2,3,3',4,4',5-HxCB	156	0.2	1.0	5.0	50	400	2000
2,3,3',4,4',5'-HxCB	157	0.2	1.0	5.0	50	400	2000
2,3',4,4',5,5'-HxCB	167	0.2	1.0	5.0	50	400	2000
3,3',4,4',5,5'-HxCB	169	0.2	1.0	5.0	50	400	2000
2,2',3,4',5,6,6'-HpCB	188	0.2	1.0	5.0	50	400	2000
2,3,3',4,4',5,5'-HpCB	189	0.2	1.0	5.0	50	400	2000
2,2',3,3',5,5',6,6'-OcCB	202	0.2	1.0	5.0	50	400	2000
2,3,3',4,4',5,5',6-OcCB	205	0.2	1.0	5.0	50	400	2000
2,2',3,3',4,4',5,5',6-NoCB	206	0.2	1.0	5.0	50	400	2000
2,2',3,3',4,5,5',6,6'-NoCB	208	0.2	1.0	5.0	50	400	2000
DeCB	209	0.2	1.0	5.0	50	400	2000
Labeled Toxics/LOC/window-defining							
$^{13}C_{12}$-2-MoCB	1L	100	100	100	100	100	100
$^{13}C_{12}$-4-MoCB	3L	100	100	100	100	100	100
$^{13}C_{12}$-2,2'-DiCB	4L	100	100	100	100	100	100
$^{13}C_{12}$-4,4'-DiCB	15L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',6'-TriCB	19L	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4'-TriCB	37L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4'-TeCB	77L	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4',5-TeCB	81L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	100	100	100	100	100
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	100	100	100	100	100

TABLE 5 (cont.)

		Solution concentration (ng/mL)					
CB congener	IUPAC[1]	CS-0.2 (Hi sens)[2]	CS-1	CS-2	CS-3 (VER)	CS-4	CS-5
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5-HxCB	156L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB	157L	100	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB	189L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4',5,5',6,6'-NoCB	208L	100	100	100	100	100	100
$^{13}C_{12}$-DeCB	209L	100	100	100	100	100	100
Labeled clean-up							
$^{13}C_{12}$-2,4,4'-TrCB	28L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L	100	100	100	100	100	100
Labeled injection internal							
$^{13}C_{12}$-2,5-DiCB	9L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',5,5'-TeCB	52L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4',5,5'-PeCB	101L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3',4,4',5'-HxCB	138L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB	194L	100	100	100	100	100	100

1. Suffix "L" indicates labeled compound.

2. Additional concentration used for calibration of high sensitivity HRGC/HRMS systems (not used by CAS/Houston).

TABLE 6
QC ACCEPTANCE CRITERIA FOR CHLORINATED BIPHENYLS IN VER, IPR, OPR, AND SAMPLES[1]

Congener	Congener number[2]	Test conc (ug/mL)[3]	VER[4] (%)	IPR		OPR (%)	Labeled compound recovery in samples (%)
				RSD (%)	X (%)		
2-MoCB	1	50	70-130	40	60-140	50-150	
4-MoCB	3	50	70-130	40	60-140	50-150	
2,2'-DiCB	4	50	70-130	40	60-140	50-150	
4,4'-DiCB	15	50	70-130	40	60-140	50-150	
2,2'6-TrCB	19	50	70-130	40	60-140	50-150	
3,4,4'-TrCB	37	50	70-130	40	60-140	50-150	
2,2'6,6'TeCB	54	50	70-130	40	60-140	50-150	
3,3',4,4'-TeCB	77	50	70-130	40	60-140	50-150	
3,4,4',5-TeCB	81	50	70-130	40	60-140	50-150	
2,2',4,6,6'-PeCB	104	50	70-130	40	60-140	50-150	
2,3,3',4,4'-PeCB	105	50	70-130	40	60-140	50-150	
2,3,4,4',5-PeCB	114	50	70-130	40	60-140	50-150	
2,3',4,4',5-PeCB	118	50	70-130	40	60-140	50-150	
2',3,4,4',5-PeCB	123	50	70-130	40	60-140	50-150	
3,3',4,4',5-PeCB	126	50	70-130	40	60-140	50-150	
2,2',4,4',6,6'-HxCB	155	50	70-130	40	60-140	50-150	
2,3,3',4,4',5-HxCB[5]	156	50	70-130	40	60-140	50-150	
2,3,3',4,4',5'-HxCB[5]	157	50	70-130	40	60-140	50-150	
2,3',4,4',5,5'-HxCB	167	50	70-130	40	60-140	50-150	
3,3',4,4',5,5'-HxCB	169	50	70-130	40	60-140	50-150	
2,2',3,4',5,6,6'-HpCB	188	50	70-130	40	60-140	50-150	
2,3,3',4,4',5,5'-HpCB	189	50	70-130	40	60-140	50-150	
2,2',3,3',5,5',6,6'-OcCB	202	50	70-130	40	60-140	50-150	
2,3,3',4,4',5,5',6-OcCB	205	50	70-130	40	60-140	50-150	
2,2',3,3',4,4',5,5',6-NoCB	206	50	70-130	40	60-140	50-150	
2,2',3,3',4,5,5',6,6'-NoCB	208	50	70-130	40	60-140	50-150	
DeCB	209	50	70-130	40	60-140	50-150	
$^{13}C_{12}$-2-MoCB	1L	100	50-150	50	20-135	15-140	15-150
$^{13}C_{12}$-4-MoCB	3L	100	50-150	50	20-135	15-140	15-150
$^{13}C_{12}$-2,2'-DiCB	4L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-4,4'-DiCB	15L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',6-TrCB	19L	100	50-150	50	35-135	30-140	25-150

TABLE 6 (cont.)

$^{13}C_{12}$-3,4,4'-TrCB	37L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-3,3',4,4'-TeCB	77L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-3,4,4',5-TeCB	81L	100	50-150	50	35-135	30-140	25-15
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,3,3',4,4',5 -HxCB[5]	156L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB[5]	157L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',3,4',5,5',6,6'-HpCB	188L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB	189L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB	208L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DeCB	209L	100	50-150	50	35-135	30-140	25-150
Cleanup standard							
$^{13}C_{12}$-2,4,4'-TrCB	28L	100	60-130	45	45-120	40-125	30-135
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L	100	60-130	45	45-120	40-125	30-135
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L	100	60-130	45	45-120	40-125	30-135

1. QC acceptance criteria for IPR, OPR, and samples based on a 20µl. extract final volume.

2. Suffix "L" indicates labeled compound.

3. See Table 5.

4. See Section 12.4.

5. PCBs 156 and 157 are tested as the sum of two concentrations.

TABLE 7
SCAN DESCRIPTORS, LEVELS OF CHLORINATION, M/Z INFORMATION, AND SUBSTANCES MONITORED BY HRGC/HRMS

Function and chlorine level	m/z	m/z type	m/z formula	Substance
Fn-1; Cl-1	188.0393	M	$^{12}C_{12}H_9{}^{35}Cl$	Cl-1 CB
	190.0363	M+2	$^{12}C_{12}H_9{}^{37}Cl$	Cl-1 CB
	200.0795	M	$^{13}C_{12}H_9{}^{35}Cl$	$^{13}C_{12}$ Cl-1 CB
	202.0766	M+2	$^{13}C_{12}H_9{}^{37}Cl$	$^{13}C_{12}$ Cl-1 CB
	218.9856	lock	C_4F_9	PFK
Fn-2; Cl-2,3	222.0003	M	$^{12}C_{12}H_8{}^{35}Cl_2$	Cl-2 PCB
	223.9974	M+2	$^{12}C_{12}H_8{}^{35}Cl^{37}Cl$	Cl-2 PCB
	225.9944	M+4	$^{12}C_{12}H_8{}^{37}Cl_2$	Cl-2 PCB
	234.0406	M	$^{13}C_{12}H_8{}^{35}Cl_2$	$^{13}C_{12}$ Cl-2 PCB
	236.0376	M+2	$^{13}C_{12}H_8{}^{35}Cl^{37}Cl$	$^{13}C_{12}$ Cl-2 PCB
	242.9856	lock	C_6F_9	PFK
	255.9613	M	$^{12}C_{12}H_7{}^{35}Cl_3$	Cl-3 PCB
	257.9584	M+2	$^{12}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Cl-3 PCB
Fn-3	255.9613	M	$^{12}C_{12}H_7{}^{35}Cl_3$	Cl-3 PCB
Cl-3,4,5	257.9584	M+2	$^{12}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Cl-3 PCB
	259.9554	M+4	$^{12}C_{12}H_7{}^{35}Cl{}^{37}Cl_2$	Cl-3 PCB
	268.0016	M	$^{13}C_{12}H_7{}^{35}Cl_3$	$^{13}C_{12}$ Cl-3 PCB
	269.9986	M+2	$^{13}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	$^{13}C_{12}$ Cl-3 PCB
	280.9825	lock	C_6F_{11}	PFK
	289.9224	M	$^{12}C_{12}H_6{}^{35}Cl_4$	Cl-4 PCB
	291.9194	M+2	$^{12}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Cl-4 PCB
	293.9165	M+4	$^{12}C_{12}H_6{}^{35}Cl_2{}^{37}Cl_2$	Cl-4 PCB

TABLE 7 (cont.)

Function and chlorine level	m/z	m/z type	m/z formula	Substance
	301.9626	M	$^{12}C_{12}H_6{}^{35}Cl_4$	$^{13}C_{12}$ Cl-4 PCB
	303.9597	M+2	$^{12}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	$^{13}C_{12}$ Cl-4 PCB
	323.8834	M	$^{12}C_{12}H5{}^{35}Cl_5$	Cl-5 PCB
	325.8804	M+2	$^{12}C_{12}H5{}^{35}Cl_4{}^{37}Cl$	Cl-5 PCB
	327.8775	M+4	$^{12}C_{12}H5{}^{35}Cl_3{}^{37}Cl_2$	Cl-5 PCB
	337.9207	M+2	$^{13}C_{12}H5{}^{35}Cl_4{}^{37}Cl$	$^{13}C_{12}$ Cl-5 PCB
	339.9178	M+4	$^{13}C_{12}H5{}^{35}Cl_3{}^{37}Cl_2$	$^{13}C_{12}$ Cl-5 PCB
Fn-4	289.9224	M	$^{12}C_{12}H_6{}^{35}Cl_4$	Cl-4 PCB
Cl-4,5,6	291.9194	M+2	$^{12}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Cl-4 PCB
	293.9165	M+4	$^{12}C_{12}H_6{}^{35}Cl_2{}^{37}Cl_2$	Cl-4 PCB
	301.9626	M+2	$^{13}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	$^{13}C_{12}$ Cl-4 PCB
	303.9597	M+4	$^{13}C_{12}H_6{}^{35}Cl_2{}^{37}Cl_2$	$^{13}C_{12}$ Cl-4 PCB
	323.8834	M	$^{12}C_{12}H_5{}^{35}Cl_5$	Cl-5 PCB
	325.8804	M+2	$^{12}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Cl-5 PCB
	327.8775	M+4	$^{12}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Cl-5 PCB
	330.9792	lock	C_7F_{15}	PFK
	337.9207	M+2	$^{13}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	$^{13}C_{12}$ Cl-5 PCB
	339.9178	M+4	$^{13}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	$^{13}C_{12}$ Cl-5 PCB
	359.8415	M+2	$^{12}C_{12}H_4{}^{35}Cl_6{}^{37}Cl$	Cl-6 PCB
	361.8385	M+4	$^{12}C_{12}H_4{}^{35}Cl_5{}^{37}Cl_2$	Cl-6 PCB
	363.8356	M+6	$^{12}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Cl-6 PCB
	371.8817	M+2	$^{13}C_{12}H_4{}^{35}Cl_6{}^{37}Cl$	$^{13}C_{12}$ Cl-6 PCB
	373.8788	M+4	$^{13}C_{12}H_4{}^{35}Cl_5{}^{37}Cl_2$	$^{13}C_{12}$ Cl-6 PCB
Fn-5	323.8834	M	$^{12}C_{12}H_5{}^{35}Cl_5$	Cl-5 PCB

TABLE 7 (cont.)

Function and chlorine level	m/z	m/z type	m/z formula	Substance
Cl-5,6,7	325.8804	M+2	$^{12}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Cl-5 PCB
	327.8775	M+4	$^{12}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Cl-5 PCB
	337.9207	M+2	$^{13}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	$^{13}C_{12}$ Cl-5 PCB
	339.9178	M+4	$^{13}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	$^{13}C_{12}$ Cl-5 PCB
	354.9792	lock	C_9F_{19}	PFK
	359.8415	M+2	$^{12}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Cl-6 PCB
	361.8385	M+4	$^{12}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Cl-6 PCB
	363.8356	M+6	$^{12}C_{12}H_4{}^{35}Cl_3{}^{37}Cl_3$	Cl-6 PCB
	371.8817	M+2	$^{13}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	$^{13}C_{12}$ Cl-6 PCB
	373.8788	M+4	$^{13}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	$^{13}C_{12}$ Cl-6 PCB
	393.8025	M+2	$^{12}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Cl-7 PCB
	395.7995	M+4	$^{12}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Cl-7 PCB
	397.7966	M+6	$^{12}C_{12}H_3{}^{35}Cl_4{}^{37}Cl_3$	Cl-7 PCB
	405.8428	M+2	$^{13}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	$^{13}C_{12}$ Cl-7 PCB
	407.8398	M+4	$^{13}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	$^{13}C_{12}$ Cl-7 PCB
	454.9728	QC	$C_{11}F_{17}$	PFK
Fn-6	393.8025	M−2	$^{12}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Cl-7 PCB
Cl-7,8,9,10	395.7995	M−4	$^{12}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Cl-7 PCB
	397.7966	M−6	$^{12}C_{12}H_3{}^{35}Cl_4{}^{37}Cl_3$	Cl-7 PCB
	405.8428	M+2	$^{13}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	$^{13}C_{12}$ Cl-7 PCB
	407.8398	M+4	$^{13}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	$^{13}C_{12}$ Cl-7 PCB
	427.7635	M+2	$^{12}C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Cl-8 PCB
	429.7606	M+4	$^{12}C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Cl-8 PCB
	431.7576	M+6	$^{12}C_{12}H_2{}^{35}Cl_5{}^{37}Cl_3$	Cl-8 PCB

TABLE 7 (cont.)

Function and chlorine level	m/z	m/z type	m/z formula	Substance
	439.8038	M+2	$^{13}C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	$^{13}C_{12}$ Cl-8 PCB
	441.8008	M+4	$^{13}C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	$^{13}C_{12}$ Cl-8 PCB
	442.9728	QC	$C_{10}F_{13}$	PFK
	454.9728	lock	$C_{11}F_{13}$	PFK
	461.7246	M+2	$^{12}C_{12}H_1{}^{35}Cl_8{}^{37}Cl$	Cl-9 PCB
	463.7216	M+4	$^{12}C_{12}H_1{}^{35}Cl_7{}^{37}Cl_2$	Cl-9 PCB
	465.7187	M+6	$^{12}C_{12}H_1{}^{35}Cl_6{}^{37}Cl_3$	Cl-9 PCB
	473.7648	M+2	$^{13}C_{12}H_1{}^{35}Cl_8{}^{37}Cl$	$^{13}C_{12}$ Cl-9 PCB
	475.7619	M+4	$^{13}C_{12}H_1{}^{35}Cl_7{}^{37}Cl_2$	$^{13}C_{12}$ Cl-9 PCB
	495.6856	M+2	$^{12}C_{12}H_4{}^{35}Cl_9{}^{37}Cl$	Cl-10 PCB
	497.6826	M+4	$^{12}C_{12}{}^{35}Cl_8{}^{37}Cl_2$	Cl-10 PCB
	499.6797	M+6	$^{12}C_{12}{}^{35}Cl_7{}^{37}Cl_3$	Cl-10 PCB
	507.7258	M+2	$^{13}C_{12}H_4{}^{35}Cl_9{}^{37}Cl$	$^{13}C_{12}$ Cl-10 PCB
	509.7229	M+4	$^{13}C_{12}H_4{}^{35}Cl_8{}^{37}Cl_2$	$^{13}C_{12}$ Cl-10 PCB
	511.7199	M+6	$^{13}C_{12}H_4{}^{35}Cl_7{}^{37}Cl_3$	$^{13}C_{12}$ Cl-10 PCB

Note: CAS/Houston utilizes a 7 function acquisition program.

Isotopic masses used for accurate mass calculation:

^{1}H	1.0078
^{12}C	12.0000
^{13}C	13.0034
^{35}Cl	34.9689
^{37}Cl	36.9659
^{19}F	18.9984

TABLE 8
THEORETICAL ION ABUNDANCE RATIOS AND QC LIMITS

Chlorine atoms	m/z's forming ratio	Theoretical ratio	Lower QC limit	Upper QC limit
1	m/m+2	3.13	2.66	3.60
2	m/(m+2)	1.56	1.33	1.79
3	m/(m+2)	1.04	0.88	1.20
4	m/(m+2)	0.77	0.65	0.89
5	(m+2)/(m+4)	1.55	1.32	1.78
6	(m+2)/(m+4)	1.24	1.05	1.43
7	(m+2)/(m+4)	1.05	0.89	1.21
8	(m+2)/(m+4)	0.89	0.76	1.02
9	(m+2)/(m+4)	0.77	0.65	0.89
10	(m+2)/(m+4)	0.69	0.59	0.79

TABLE 9
SUGGESTED SAMPLE QUANTITIES TO BE EXTRACTED FOR VARIOUS MATRICES[1]

Sample matrix[2]	Example	Percent solids	Phase	Quantity extracted
Single-phase				
Aqueous	Drinking water	<1	—[3]	10-100mL
	Groundwater			
	Treated wastewater			
Solid	Dry soil	>20	Solid	5-10g
	Compost			
	Ash			
Organic	Waste solvent	<1	Organic	5-10g
	Waste oil			
	Organic polymer			
Tissue	Fish	—	Organic	5-10g
	Human adipose			
Multi-phase				
Liquid/Solid				
Aqueous/Solid	Wet soil	1-30	Solid	5-10g
	Untreated effluent			
	Digested municipal sludge			
	Filter cake			
	Paper pulp			
Organic/solid	Industrial sludge	1-100	Both	5-10g
	Oily waste			
Liquid/Liquid				
Aqueous/organic	In-process effluent	<1	Organic	5-10g
	Untreated effluent			
	Drum waste			
Aqueous/organic/solid	Untreated effluent	>1	Organic & solid	5-10g
	Drum Waste			

1. The quantity of sample to be extracted is adjusted to provide 10g of solids (dry weight). One liter of aqueous samples containing one percent solids will contain 10g of solids. For aqueous samples containing greater than one percent solids, a lesser volume is used so that 10g of solids (dry weight) will be extracted.

2. The sample matrix may be amorphous for some samples. In general, when the CBs are in contact with a multi-phase system in which one of the phases is water, they will be preferentially dispersed in or adsorbed on the alternate phase because of their low solubility in water.

3. Aqueous samples are filtered after spiking with the labeled compounds. The filtrate and the materials trapped on the filter are extracted separately, and the extracts are combined for clean up and analysis.

TABLE 10
CORRECTIVE ACTION SUMMARY TABLE

QC Activity	Unacceptable Criteria	Recommended Corrective Action	Documentation
Method Blank	>MRL, >1/10 sample conc.	Analyze an instrument blank to demonstrate instrument is free of possible contamination. Evaluate whether entire sample batch must be re-extracted, depending on client specifications.	Corrective actions must be documented as a non-conformity. Compounds associated with a value in the MB are flagged with 'B' on Form 1s.
Instrument Blank	>MRL	Determine cause of contamination. Analyze an acceptable instrument blank prior to analyzing analytical sequence.	Resolve blank issue before proceeding.
Initial Calibration Standards	± 40% native standards ± 50% labeled standards	Reanalyze standards. If still unacceptable, remake standards and reanalyze.	Resolve ICAL standards issue before proceeding.
Continuing Calibration Verification Standard (CCV/VER)	> RRF: ± 40% native and ± 50% labeled for beginning cal Ending CCAL not required for 1668A	Reanalyze standard. If still unacceptable, recalibrate and reanalyze samples from last acceptable CCV.	Comment in case narrative.
Laboratory Control Sample (LCS) & Duplicate Laboratory Control Sample (DLCS)	50% >value>150% >50% RSD	If LCS or DLCS out, evaluate the labeled standard(s) associated with the compound(s). If associated labeled standards out, evaluate the associated labeled standard(s) in the sample(s). If labeled standard(s) in the sample(s) is out, re-extract the samples, else, release the test results.	Comment in case narrative.
Labeled Standard Recoveries	> acceptance criteria for all labeled standards or if any labeled standard recovery is <10%	Complete an NCAR form and re-extract the sample using a smaller sample size.	Add an 'RE' suffix to the end of the Lab Sample ID, indicating 'Re-extraction'
Recovery of labeled standard for compounds	Outside acceptable limits	Report values and flag results.	Flag associated results with 'Y' on Form 1s.
Sample Results	Quantified value > upper end of linear calibration range.	Report values; dilute sample and report both undiluted and diluted test results.	Flag associated results with 'E' on Form 1s.
Sample Results	Quantified value < lower end of linear calibration range and > EDL.	Report values and flag results.	Flag associated results with 'J' on Form 1s.
Sample Results	Compound not detected.	Report values and flag results.	Flag associated results with 'U' on Form 1s.
Native ions	Outside ion abundance ratio QC limit	Report as EMPC	Flag associated results with 'K' on Form 1s.

APPENDIX 2

FIGURE 1
FLOW CHART FOR ANALYSIS OF AQUEOUS AND SOLID SAMPLES

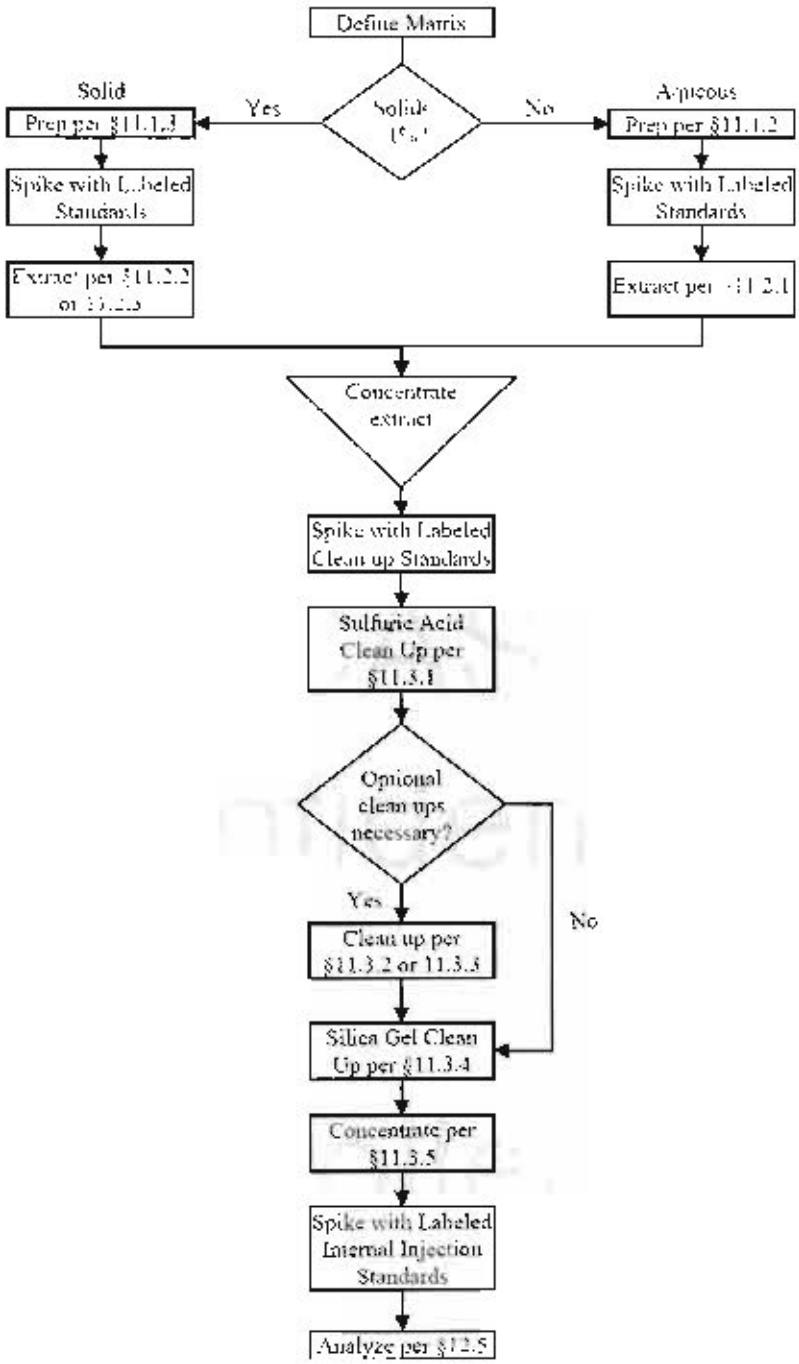

FIGURE 2
FLOW CHART FOR ANALYSIS OF TISSUE SAMPLES

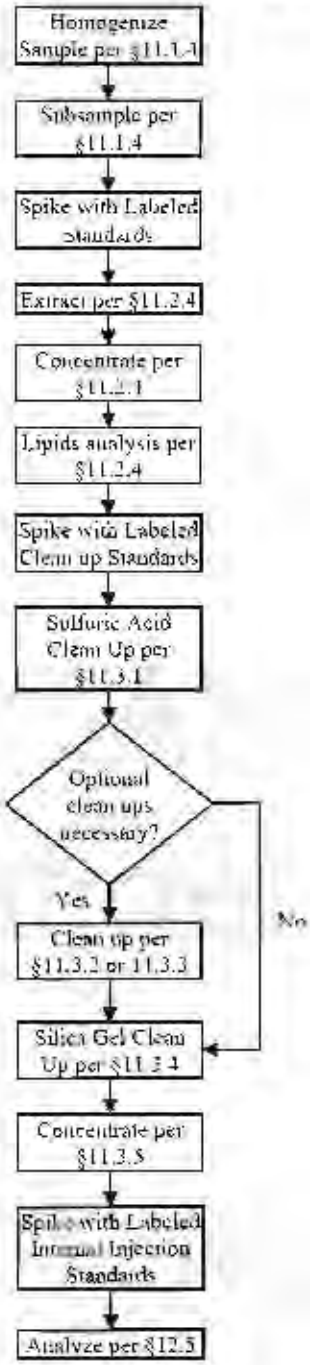

FIGURE 3
OCTYL COLUMN RESOLUTION TEST #1: SEPARATION OF CL-3 CONGENERS 34 AND 23 WITH VALLEY <40% (I.E. 100X/Y < 40%)

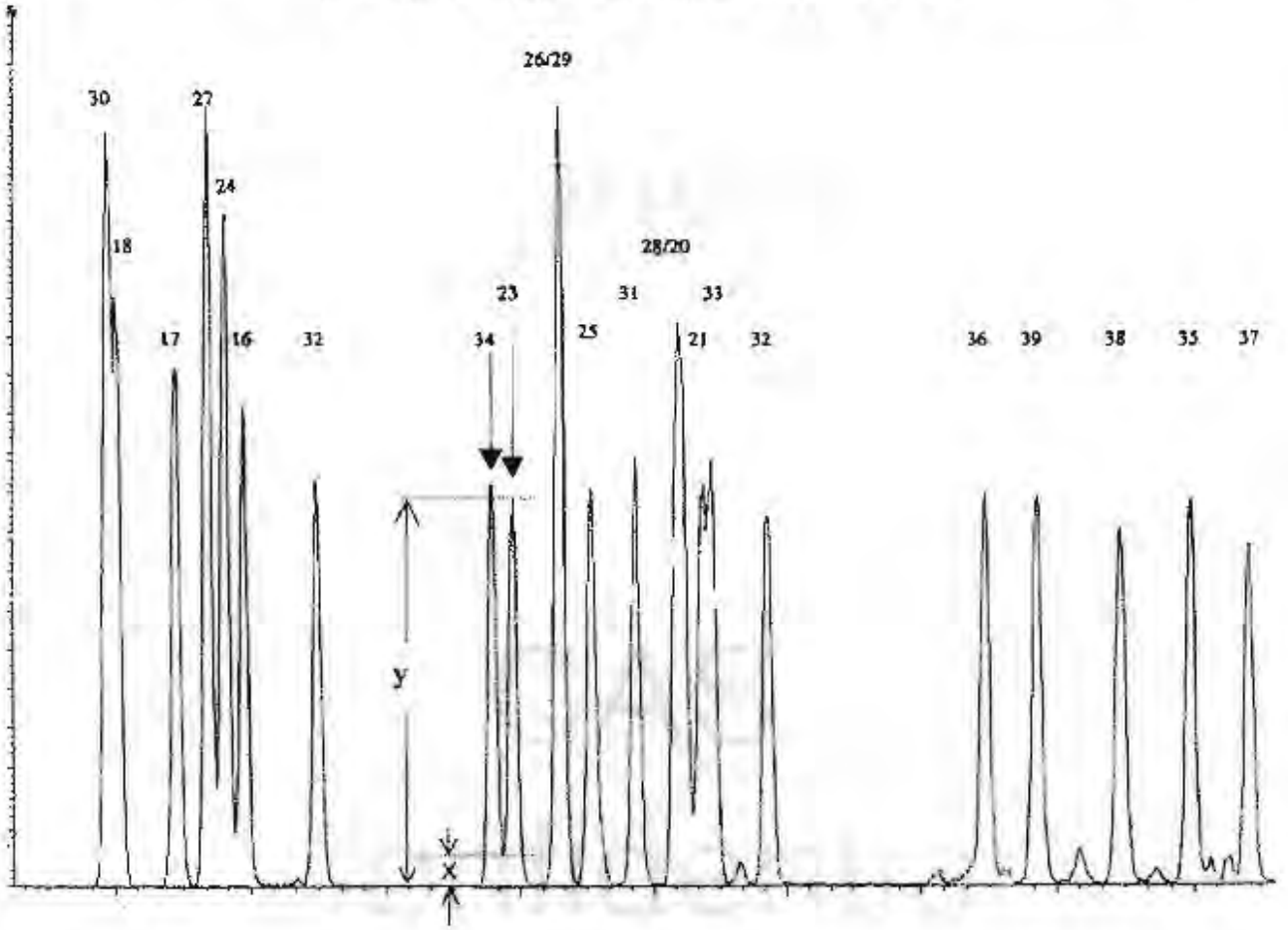

FIGURE 4
OCTYL COLUMN RESOLUTION TEST #2: SEPARATION OF CL-7 CONGENERS 187 AND 182 WITH VALLEY <40% (I.E. 100X/Y < 40%)

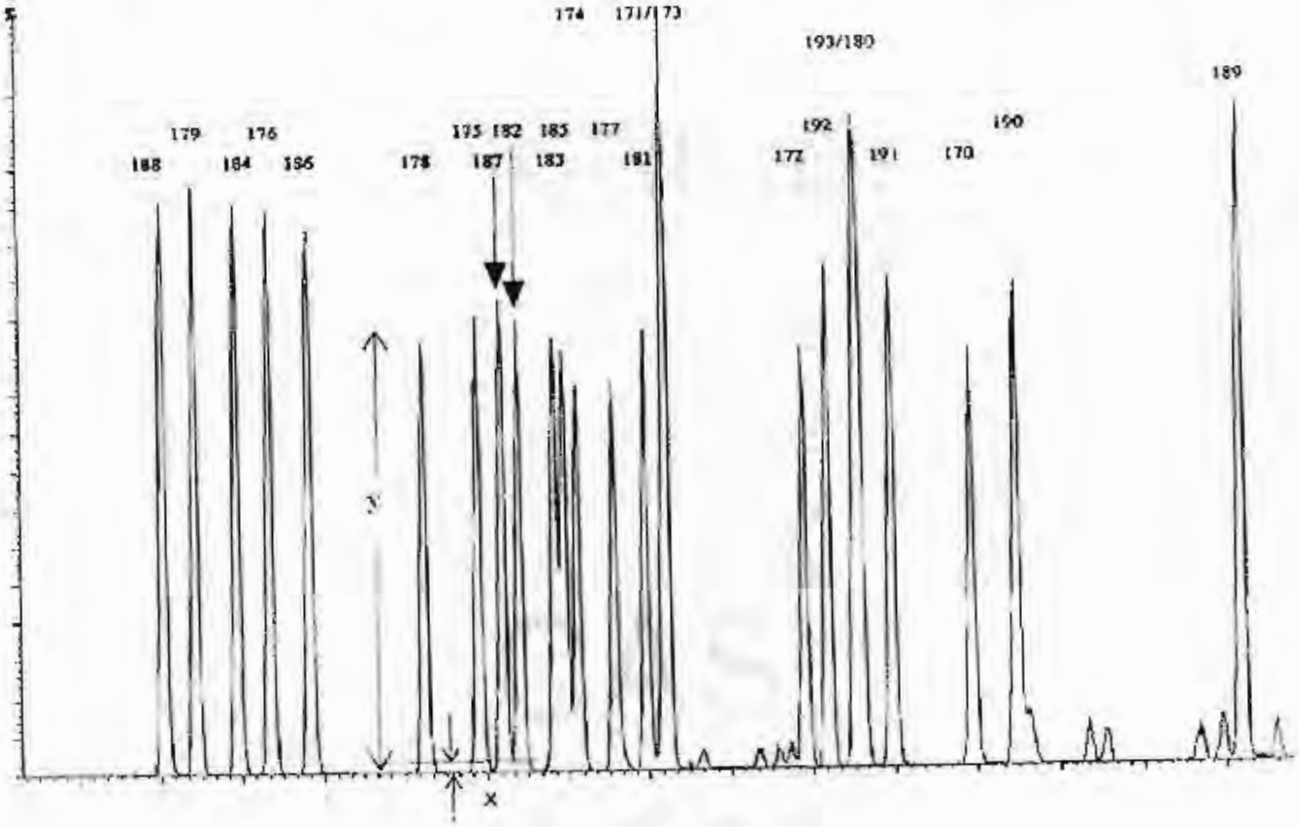

FIGURE 5
CB CONGENERS AT EACH LEVEL OF CHLORINATION ON THE SPB-OCTYL COLUMN

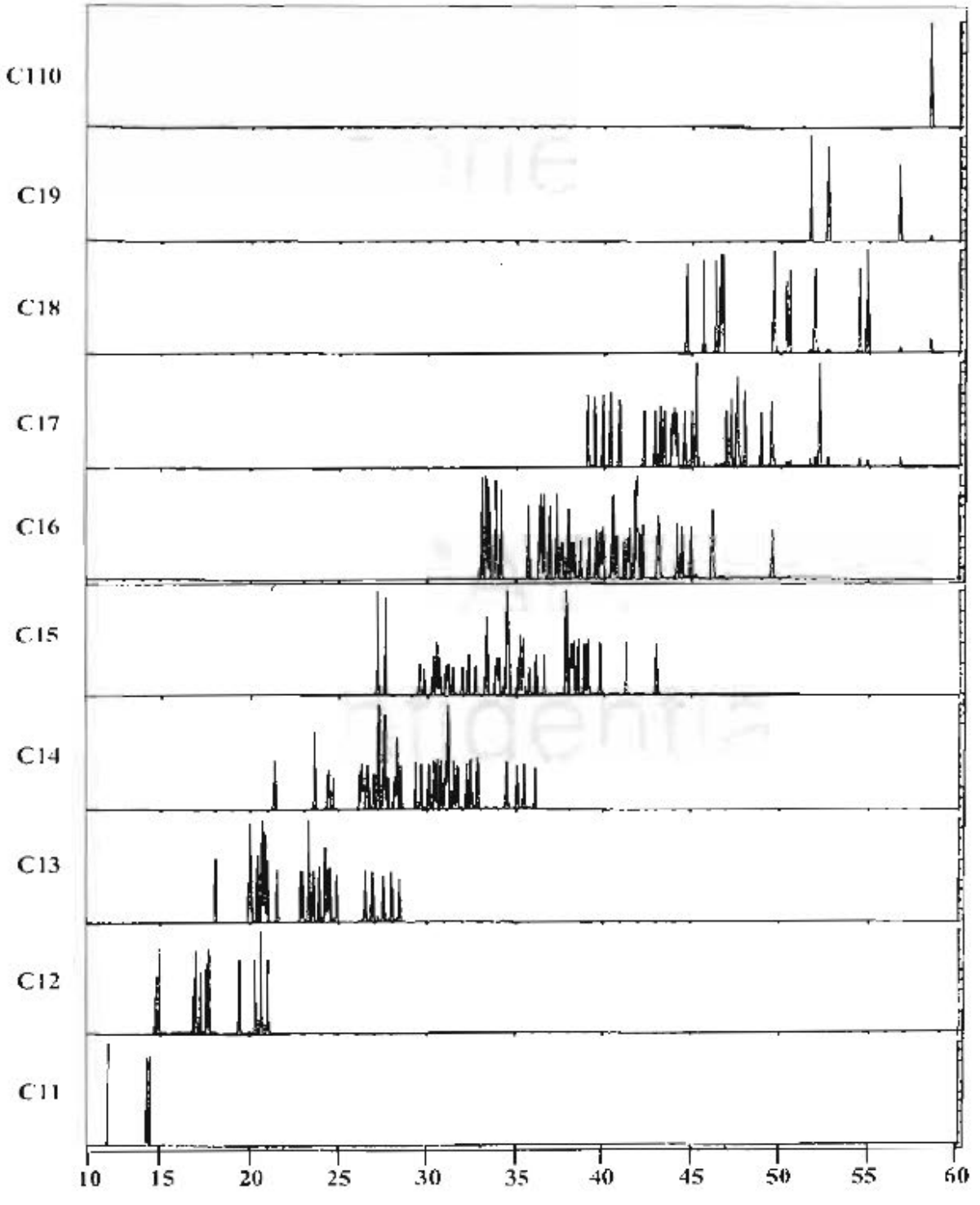

FIGURE 6
FORMAT FOR A TRAINING PLAN

1. Read and study SOP — Trainer: ____ Trainee: ____ Date: _____

2. Read Published Method — Trainer: ____ Trainee: ____ Date: _____

3. Demonstrated scientific understanding of the analysis — Trainer: ____ Trainee: ____ Date: _____
   Sample preparation
   HR-Gas chromatography
   HR-Mass spectrometry

4. Demonstrated familiarity with related SOPs — Trainer: ____ Trainee: ____ Date: _____
   SOP for Analytical Batches and Analytical Sequences
   SOP for Making Entries into Logbooks and onto Bench sheets
   SOP for Manual Integration of Chromatographic Peaks
   SOP for Significant Figures
   SOP for Nonconformity and Corrective Action Documentation
   SOP for Determination of Method Detection Limits

5. Observe performance of SOP — Trainer: ____ Trainee: ____ Date: _____
   - sample preparation and sample loading
   - analytical sequence setup
   - initial calibration and continuing calibration verification
   - sample analysis
   - software introduction
   - data reduction and reporting

6. Perform SOP with supervision — Trainer: ____ Trainee: ____ Date: _____
   - sample preparation and sample loading
   - analytical sequence setup
   - initial calibration and continuing calibration verification
   - sample analysis
   - software use
   - data reduction and reporting

7. Independent performance of the SOP — Trainer: ____ Trainee: ____ Date: _____
   - sample preparation and sample loading
   - analytical sequence setup
   - initial calibration and continuing calibration verification
   - sample analysis
   -software proficiency
   - data reduction and reporting
   - initial demonstration of competency
   - IPR study
     - single blind PE sample
     - four consecutive LCSs

8. Instrument operation and maintenance — Trainer: ____ Trainee: ____ Date: _____
   - autosampler
   - gas chromatograph and capillary column installation
   - mass spectrometer
   - data system

Attachment 7

SOP for Sample Preparation for Black Carbon (Soot) in Sediment by Chemothermal Oxidation Pretreatment and Combustion / Thermoconductivity or Infrared Detection. ALS SOP Code: GEN-BLACK C SOOT PREP

STANDARD OPERATING PROCEDURE

for

Sample Preparation for Black Carbon (Soot) in Sediment By Chemothermal Oxidation Pretreatment and Combustion / Thermoconductivity or Infrared Detection

SOP Code: GEN- BLACK C SOOT PREP
Revision: .01

DATE: 9/20/2010

Approved by:_____ _____
Ralph V. Poulsen, Laboratory Director **Date**

_____ _____
Wendy Hyatt, Laboratory Supervisor/Project Chemist **Date**

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Effect. Date: _____
Initials: _____ Effect. Date: _____
Initials: _____ Effect. Date: _____

DOCUMENT CONTROL

NUMBER: GEN- BLACK C SOOT PREP RV1
Effective
Initials: _____ Date: _____

Standard Operating Procedure
for
Sample Preparation for Black Carbon (Soot) in Sediment
By Chemothermal Oxidation Pretreatment and
Combustion / Thermoconductivity or Infrared Detection

1.0 SCOPE AND APPLICATION

1.1 Black Carbon (BC) or Soot consists primarily of highly condensed carbon particles formed from pyrolysis (incomplete combustion) of fossil fuels, biofuel, and biomass. BC is very stable and resists oxidation via biological and chemical processes. It is considered to be a major contributor to global warming, second only to carbon dioxide gas.

1.2 BC in sediment and soil can be determined by first pre-treating the sample using a Chemothermal Oxidation (CTO) sample preparation procedure to remove interfering inorganic carbon (carbonates) and nonpyrogenic organic carbon (NPOC). The residue is then analyzed for Total Carbon (TC), which represents the BC in the original sample.

Total Carbon analysis is performed using a high temperature combustion Carbon or Carbon/Hydrogen/Nitrogen (CHN) analyzer at 950°C or higher. Several different instruments can be used: Perkin Elmer model 2400 CHN analyzer, LECO Micro Truspec CHN analyzer, LECO Macro TruSpec CHNS analyzer, or Eltra CS500 Carbon - Sulfur analyzer.

1.3 The lower concentration range for BC is approximately 0.05% (dry wt. basis).
.

2.0 SUMMARY OF METHOD

A sediment sample is initially dried at 60°C and then ground to a fine powder. A 10 mg aliquot of the dried and ground material is sub-sampled into a silver capsule for pre-treatment prior to final analysis for BC. The sample is treated with hydrochloric acid to remove inorganic carbon and then subjected to heating at 375°C in an air-purged muffle furnace to remove NPOC. The sediment residue is then analyzed by an elemental CHN or Carbon analyzer. These instruments employ direct combustion of the sample in ultra-pure oxygen at 935°C to 1350°C depending on the instrument used. The carbon is converted to the gaseous phase as carbon dioxide by the combustion process and quantified as a function of it's thermal conductivity or infrared absorption. Concentration is calculated from the mass of carbon detected after the treatment process as a function of the initial 10 mg sample aliquot.

Note that this SOP is intended to address the sample preparation process up to the point where the sediment residue is ready for instrumental analysis. The quality control (QC) samples associated with the preparation batch are defined in this SOP, but QC samples associated with the instrumental analysis are defined in the associated SOP for that part of the process (i.e. CHN by Combustion/Thermo-Conductivity Detection; SOP Code: TUCSON-CHN).

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3.0 DEFINITIONS

3.1 Analytical Protocol: Samples are analyzed in a set referred to as an analytical protocol or sequence. Since this SOP addresses the sample preparation portion of the process only, the protocol includes QC samples associated with the preparation only (i.e. Prep Blank, Duplicate, and Standard Reference Materials – SRM).

3.2 Benchsheet: A form used to record the analytical protocol, sample information, and data.

3.3 DI Water: DI Water is laboratory pure water that has been passed through an initial deionizing system followed by a polishing deionizing system (ultra-pure or nanopure) producing water that meets ASTM Type I criteria.

3.4 Prep Blank (PB): The prep blank for this method consists of carrying a blank silver capsule through the entire sample preparation procedure, including addition of reagents, reaction steps, and heating/cooling steps. The purpose of the PB is to determine the presence and magnitude of analyte contribution from reagents, labware, other equipment, and/or atmosphere.

3.6 Standard Reference Material (SRM): The SRM is a standard that represents the matrix of the associated samples being analyzed. For the purposes of this procedure, an SRM that contains primarily carbon, half of which is BC (NIST SRM-1650 – Truck Diesel Particulate Matter), is used in lieu of a chemical standard. To supplement it, a sediment SRM (NIST SRM-1941 or SRM-1944) that contains a significant amount of BC is used to accurately represent the sample matrix. The SRM has a certified or documented true value associated with it, so the analytical batch can be controlled for accuracy. The SRM is purchased from a qualified vendor (National Institute of Standards and Technology – NIST). For this analysis, the SRM is synonymous with a Laboratory Control Sample (LCS).

4.0 INTERFERENCES

4.1 NPOC is a potential positive interference that is removed by the preliminary heating at 375°C. As long as the temperature and heating time are observed, the interference is removed.

4.2 BC values may exhibit a high bias for samples with high concentration of carbonates that do not fully react during the HCl pretreatment step. A more aggressive acid treatment may be required in these situations. Particular attention should be paid to marine sediments potentially containing shells, coral, and associated fragments.

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5.0 SAFETY

5.1 Follow all CAS safety practices as described in the CAS Safety Manual.

5.2 The toxicity or carcinogenicity of each compound or reagent used in the method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to the compounds should be reduced to the lowest possible level. A reference file of material safety data sheets is available to all personnel involved in these analyses. Columbia Analytical Services also maintains a file of OSHA regulations regarding the safe handling of chemicals specified in these procedures.

6.0 SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1 Samples can be collected in glass or plastic containers.

6.2 Environmental samples can be adversely affected by biological activity and must be collected, preserved, and stored using appropriate precautions. Preservation for BC consists of freezing at -20°C or refrigerating at 4±2°C.

6.3 Holding times have not been established for this method. Samples should be handled as detailed above to avoid degradation of the samples.

7.0 APPARATUS AND EQUIPMENT

7.1 Oven, 103-105°C.

7.2 Muffle Furnace, 4” x 4” x 6”, 375°C (with air purge capability).

7.3 Micro Balance, 1 ug sensitivity.

7.4 Analytical Balance, 0.1 mg sensitivity.

7.5 Stainless Steel Fine Tip Forceps.

7.6 Silver Capsules (5 X 9 mm for micro CHN analyzer).

7.7 Aluminum Capsule Block for Silver Capsules.

7.8 Ceramic Boats (for Eltra CS500).

7.9 Refrigerator, 4°C.

7.10 Freezer, -20°C.

7.11 Mortar and Pestle.

7.12 Mini Ball Mill w/Tungsten Carbide Balls.

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8.0 STANDARDS AND REAGENTS

8.1 Standard Reference Material, NIST-1650 Truck Diesel Particulate Matter.

8.2 Standard Reference Material, NIST-1941b Baltimore Harbor Marine Sediment or NIST-1944 New York/New Jersey Waterway Sediment.

8.3 Hydrochloric Acid (HCl), Concentrated, ACS Reagent grade.

8.3.1 1 N HCl working solution – 17 mL of HCl into 83 mL laboratory pure water.

9.0 PREVENTIVE MAINTENANCE

General preventive maintenance is required of support equipment used in this method. Follow the equipment manual for operation and preventive maintenance.

10.0 RESPONSIBILITIES

10.1 This SOP is intended for use by experienced analysts. It should also be used for training of technicians and chemists in the above referenced method, and as a reference for data reviewers for data generated by use of this SOP.

10.2 **Analyst -** The analyst is responsible for:

- Performing the analysis according to the equipment manual, the method SOP, QA/QC criteria, and company safety procedures.
- Properly operating and/or maintaining the equipment.
- Entering the appropriate information onto the benchsheets and/or logbooks.
- Documenting and notifying the laboratory director of any operational problems or failed QC data.

10.3 **Laboratory Director -** The laboratory director or his/her assignee is responsible for:

- Training, scheduling, and supervising the preparation of samples.
- Reviewing and approving sample data, benchsheets, and logbooks.
- Reviewing the final sample preparation data for completeness.
- Reviewing and maintaining the SOP for this method.

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11.0 PROCEDURE

11.1 Initial Drying and Homogenization/Milling of Samples

11.1.1 Record samples to be tested on a BC Sample Preparation bench sheet form (see Attachment A).

11.1.2 Transfer approximately 5-10 g of wet sediment into a suitable drying vessel.

11.1.3 Dry the sample at 60°C for approximately 18 hours. Remove from oven and cool in a desiccator.

11.1.4 Homogenize the dried sample with mortar and pestle or ball mill, depending on the composition of the sample. Samples containing fine grain sand and silt, but no pebbles, can generally be ground to a fine powder using a mortar and pestle. However, most sediment samples will require use of mechanical milling via the ball mill to achieve satisfactory consistency (i.e. powder with consistency of flour).

11.1.5 Weigh an aliquot (approximately 10 mg) of the dried and ground sample into a silver capsule using the micro balance. Record the mass to three significant figures (the nearest 1 ug when weighing ≥10 mg). For samples with high Carbon contents (> 50 wt%) use approximately 5 mg of sample. Use the aluminum capsule block to hold the silver capsules. Note the position of the capsule with the corresponding sample identification and record it on the original raw data sheet.

11.1.6 Retain the remainder of the dried and ground sample for subsequent 105°C moisture determination so the final result can be converted from a 60°C basis to a 105°C basis.

11.1.7 The sample is now ready for hydrochloric acid treatment to remove inorganic carbon.

11.2 HCl Treatment for Inorganic Carbon Removal

11.2.1 **NOTE – all of the following steps must be conducted in a fume hood to prevent exposure to HCl fumes. Proper personal protection gear including lab coat, safety glasses, and gloves must be worn**.

11.2.2 Add 1 to 2 drops of 1N HCl to each of the samples. Observe whether effervescence occurs and note the corresponding sample identifications.

11.2.3 Dry the samples at 60°C for 1 hr.

11.2.4 Repeat steps 11.2.2 through 11.2.3 for samples that effervesced.

11.2.5 The sample is now ready for 375°C heating to remove NPOC.

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11.3 NPOC Removal via 375°C Muffle Furnace Heating

11.3.1 Transfer the aluminum capsule block to the muffle furnace pre-set at 375°C.

11.3.2 Start the flow of air at 1100 mL/min (2.5 mL/min per cm3 of furnace volume).

11.3.3 Heat the sample for 18 hours, remove, and cool.

11.3.4 Crimp the silver capsule closed and fold the corners to form a cubic or rounded package using the stainless steel forceps.
Note: No further weighing is necessary, as the sample residue in the silver capsule represents the BC plus inert material (silica, clay, salts, etc.) in the original dried and ground sample aliquot.

11.3.5 The sample is now ready for carbon analysis as per SOP "CHN by Combustion/Thermo-Conductivity Detection"; SOP Code: TUCSON-CHN.

12.0 QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

12.1 Preparation Blank: Analyze one PB in ten or less samples.

12.2 Standard Reference Material: Analyze one each of SRM 1650 and SRM 1941b (or 1944) with every ten or fewer samples. The SRMs must have recoveries of 80-120% of the true value listed in the literature.

12.3 Duplicate Analyses: Analyze one duplicate in ten or less samples. A Relative Percent Difference (RPD) for analyses should be less than 20%.

12.4 Any deviations of the QA/QC requirements must be documented on the benchsheet and the Laboratory Supervisor or his/her designate notified prior to submitting data for approval.

13.0 CALCULATIONS, DATA REDUCTION AND REPORTING

13.1 Calculations:

13.1.1 NA.

13.2 Validation:

The data generated for the preparation of samples for BC must be reviewed and validated for completeness. The review of results is performed as part of the instrumental analysis performed under SOP Code: TUCSON-CHN.

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13.3 Documentation

13.3.1 Record the start time and finish time and temperatures of the furnace and oven in their respective logbooks.

13.3.2 Record the calibration verification of the balance in its logbook.

13.3.2 Record the information detailed on the bench sheet (see attached example).

13.4 SOP Annual Review

13.4.1 This SOP must be reviewed at least once a year to determine if any changes are required. This review is performed by the laboratory director, laboratory supervisor, analyst performing the method, and the QA office.

13.4.2 If changes are required then a new revision is prepared and issued according to ADM-SOP.

13.4.3 If no changes are required, then each applicable person must document that this SOP has been reviewed on the front page of this SOP. A memo stating that the SOP has been reviewed and is still in effect issued by the appropriate Supervisor.

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14.0 METHOD PERFORMANCE

14.1 Reporting Limit

The Reporting Limit (RL) for BC is based on a 10 mg initial dry mass and an instrument reporting limit of 0.005 mg of carbon. The RL is calculated as follows:

%BC = (mg carbon ÷ initial sample mass, mg) x 100

= (0.005 mg carbon ÷ 10 mg) x 100

= 0.05%, Dry Wt. Basis

14.2 Method Detection Limit

For data to be reported to the MDL, determine the MDL using the following procedure.

14.2.1 An MDL study for BC is conducted by analyzing 7 or more replicates of a low level SRM or a small mass of an SRM with known BC concentration. For example, a 1 mg mass of SRM 1641b yields approximately 0.006 mg of BC. This is an appropriate mass of this SRM to conduct an MDL study for BC in sediment.

14.2.2 The MDL is calculated by multiplying the standard deviation by the Student's t value for n-1 degrees of freedom at the 99% confidence level (3.143 for 7 replicates).

14.3 Initial Demonstration of Capability

Before analyzing client samples for reporting purposes, the analyst must analyze four sample/sample duplicate pairs.

14.3.1 Analyze four SRMs.

14.3.2 After analysis is completed, calculate Recovery**.** The recoveries of the four LCSs must be 80-120% of the value in the literature.

14.4 Practical Range

The practical range for this method is 0.05% to 100 wt%.

14.5 Precision and Accuracy

See section 12

15.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

15.1 It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Reagents are prepared in volumes consistent with laboratory use in order to minimize the volume of reagents for disposal. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

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

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16.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

16.1 Corrective Action:

The corrective action process is initiated when data quality problems are observed or suspected. These cases include incomplete documentation of sample preparation process, deviations from the prescribed process, or omission of required QC samples.

16.2 Quality Control Failures:

Quality control failures associated with the actual sample results are handled under SOP Code: TUCSON-CHN.

16.4 Nonconformity Documentation:

Out of control events, conditions adverse to quality, are reported, documented and corrected. Out of control events (OOCE) may arise from the failure of a process, human error, non-compliance with requirements, inadequate controls, or sample matrix problems.

16.4.1 Data quality issues must be documented on the analytical raw data and/or the data review checklist. All appropriate data qualifiers must be added to the final reported results.

16.4.2 Problems that arise from actions under laboratory control (incomplete documentation, omission of QC samples, etc.), affect more than one batch, are more serious in nature, or are indicative of an ongoing problem are documented on a Nonconformity and Corrective Action Form (NCAR).

16.4.3 **Appendix B** contains copies of the NCAR. It is filled out by the person identifying the event. Corrective action may require consultation with the Department Manager, the QA Manager, and the Laboratory Director. The corrective action is then approved by the Supervisor and/or section Manager. The QA Manager gives final approval, and if necessary, provides to Project Chemists(s) for client notification. A copy of the form is kept with the raw data, in the project file, and the original is filed in the QA file.

17.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Any event that arises and does not conform to what is expected either by the instrument or the QC generated. An NCAR must be filled out in order to document the corrective action measures taken. In addition, the Laboratory Manager and/or the individual Project Chemists must be notified.

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18.0 REFERENCES

18.1 Gustafsson, et al, Evaluation of a Protocol for the Quantification of Black Carbon in Sediments, *Global Biogeochemical Cycles, 15,* 881-890, 2001.

18.2 Columbia Analytical Services, Tucson, Arizona , *Standard Operating Procedure for CHN by Combustion / Thermo-Conductivity Detection*, most current version.

19.0 TRAINING PLAN

19.1 Review literature (see references Section 18). Review the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

19.2 The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from assisting, to performing the procedure with minimal oversight from the experienced analyst.

19.3 Perform the initial precision and recovery (IPR) study, or initial demonstration of capability (IDC). Summaries of the studies are reviewed and signed by the supervisor and quality assurance program manager. Copies are maintained in the employee's training file.

20.0 METHOD MODIFICATIONS

None

21.0 INSTRUMENT-SPECIFIC ADDENDUM

None

22.0 CHANGES FROM PREVIOUS REVISION

None – new SOP.

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ATTACHMENTS

23.1 Appendix A – Example BC Sample Preparation bench sheet.

23.2 Appendix B - Form for documenting nonconformity.

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Appendix A

Example BC Sample Preparation Bench Sheet.

Columbia Analytical Services - Tucson, AZ
Black Carbon (Soot) in Sediment and Soil - Chemothermal Oxidation Pretreatment
Gustafsson, et al, 2001

Analyst:	
Analyst:	
Analysis Date:	
Analyst Review:	
Supervisor Review:	

Air Dry Moisture Determination				Residual Moisture Determination				Thermo Oxidation Pretreatment					
Heating Method	Temp °C	Start Date Time	End Date Time	Heating Method	Temp °C	Start Date Time	End Date Time	Heating Method		Temp °C	Start Date Time	End Date Time	Air Flow mL/min
oven	60			oven	105			Muffle Furnace		375			
	Air Dry Moisture			Residual Moisture							HCl Treatment		
Sample Lab Number	Cruc. Wt. g	Cruc. & Wet Sx Wt. g	Cruc. & Dried Sx Wt. g	Air Dry Loss Moisture wt%	Cruc. Wt. g	Cruc. & Wet Sx Wt. g	Cruc. & Dried Sx Wt. g	Residual Moisture wt%	Total Moisture wt%	Sample Prep Method	Sample Weight mg	Step 1 Effervesce yes / no	Step 2 Effervesce yes / no	Thermo Pretreat yes / no

Columbia Analytical Services, Inc.
3860 S. Palo Verde Rd #302
Tucson, AZ 85714
520.5733.1061

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Benchsheet - Black Carbon Prep.xls, 9/23/2010

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Appendix B
Example Form for Documenting Non-Conformance.

Columbia Analytical Services

Nonconformity and Corrective Action Report

SAMPLES/SYSTEM/JOB/CLIENT AFFECTED N&CA Report No. 2008-_____

NONCONFORMITY

Analysis/Event: _____

Instrument/System: _____ Date: _____

Detailed Description of Nonconformity:

Originator (name): _____ Date: _____

Supervisor Verification: _____ Date: _____

CORRECTIVE ACTION AND OUTCOME

Detailed Description: (Re-establishment of conformity must be demonstrated and documented. Describe the steps that were taken, or are planned to be taken, to correct the particular Nonconformity and prevent the reoccurrence of the Nonconformity.)

Is the data to be flagged in the Analytical Report with an appropriate qualifier? **No** **Yes**

Person Responsible: _____ Date: _____

Supervisor Verification: _____ Date: _____

NOTIFICATION - CUSTOMER/CLIENT - INTERNAL/EXTERNAL

Project Chemist Notified by: _____ Date: _____

Customer Notified? **No** **Yes** If Yes, Notifier: _____ Date: _____

Project Chemist/Customer Comments (Retain record, e.g. telephone record, e-mail) _____

ACCEPTANCE OF CORRECTIVE ACTION

Comments:

Corrective Action(s) have been implemented. QA Pgm Mgr: _____ Date: _____

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