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**Appendix A**  
**Field Sampling Plan**

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# PORTLAND HARBOR SUPPLEMENTAL SEDIMENT STUDY PORTLAND, OREGON

## *FIELD SAMPLING PLAN*

**OCTOBER 2015**

*Prepared for:*

**ExxonMobil**

*Prepared by:*





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## ACRONYMS AND ABBREVIATIONS

Alpha	Alpha Analytical
ARI	Analytical Resources, Inc.
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COCs	Contaminants of potential concern
CRD	Columbia River Datum
FSP	Field Sampling Plan
GPS	Global positioning system
HASP	Health and Safety Plan
LWG	Lower Willamette Group
MSS	Marine Sampling Systems, Inc.
NAD83	North American Datum of 1983
PAH	Polycyclic aromatic hydrocarbons
PCB	Polychlorinated biphenyl
PPE	Personal protection equipment
PTW	Principal threat waste
QA	Quality assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RAL	Remedial action level
RI/FS	Remedial investigation/feasibility study
RM	River Mile
RTK	Real time kinematic
SFS	Superfund Site
SGS	SGS North America, Inc.
SMA 9D	Sediment Management Area 9D
SOP	Standard operating procedures
TOC	Total organic carbon
TPH	Total Petroleum Hydrocarbons

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## 1.0 INTRODUCTION

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Polycyclic aromatic hydrocarbons (PAH) are chemicals of potential concern in sediments found in the Portland Harbor Superfund Site (SFS). Based on several rounds of remedial investigation data developed by the Lower Willamette Group (LWG) and from available private party studies, it is evident that PAH in the Portland Harbor SFS are ubiquitous, and found both in the surface sediment layer (< 30 cm), as well as deeper horizons of the sediment column. The widespread occurrence of PAH in urban sediments are common, and have been tied to non-point discharges of PAHs from storm water runoff, and from certain kinds of industrial point sources that can release PAH to waterways (Stout et al, 2004). Thus, focused investigations are typically needed to identify the principal and secondary sources of PAH to urban waterways.

The former ExxonMobil Terminal (9420 NW St. Helens Road, Portland OR) is proximal to Sediment Management Area 9D (SMA 9D), an area studied during the Portland Harbor remedial investigation which spans the western shoreline of the Willamette River from approximately the Terminal to the margins of the upstream U.S. Moorings property. The former Terminal handled light petroleum fuel products and so, in theory, fugitive petroleum from the facility reaching the river could potentially contribute some low molecular weight (2- and 3-ring) petrogenic PAH to the nearby sediments.

The concentrations of the sum of the 16 EPA Priority Pollutant PAH among surface sediments within SMA 9D range from 1,245 µg/kg to 229,073 µg/kg. The highest concentrations of PAH in SMA 9D are found in the most upstream reach of the SMA offshore of U.S. Moorings property. The concentrations of PAH generally decline rapidly downstream of U.S. Moorings.

The Portland Harbor remedial investigation/feasibility study (RI/FS) developed remedial action levels (RALs) for contaminants of concern (COCs), including Total PAH (LWG 2015). A range of Total PAH RALs (5,400 to 170,000 µg/kg) was developed to identify areas of surface sediments where capping and/or dredging could be used as remediation methods to meet cleanup objectives. All six of the different PAH remedial alternatives developed consist of substantial capping and/or dredging within the area between River Mile (RM) 5.0 and the St. Johns Bridge, located at approximately RM 5.8 (LWG 2015; Figure 3.3-8).

### 1.1 Project Description

In 2014, ExxonMobil collected surface and subsurface sediment samples for the purpose of determining the specific chemistry and extent of PAH found in SMA 9D. This sampling effort provided PAH data along the immediate shoreline of the former ExxonMobil Terminal and a limited number of mid-channel locations. Currently, there remains limited data of forensic chemistry quality to fully describe the characteristics of PAH in surface and subsurface sediments of the remedial alternative areas proposed for capping and/or dredging located between RM 5.0 and 5.8. The ability to fully understand potential contribution of PAH from proximal properties and upriver sources is limited by this lack of data.

Supplemental sediment sampling is proposed in a study area between RM 5.0 and 5.8 to improve the spatial and vertical coverage of sediment chemistry data in locations proposed for active remediation in the RI/FS. The purpose of this sediment field sampling plan (FSP), in conjunction with the Quality Assurance Project Plan (QAPP) (Appendix A), is to describe the approach and procedures to collect and analyze sediment samples within the study area in order to complete the objectives for this investigation:

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1.0 Perform a forensic study intended to characterize the nature of PAH in sediments. The data developed in this investigation can in turn be used to determine:

- The specific chemistry of PAH found in the sediments within the study area.
- The extent to which, if any, petroleum-source PAH from the former ExxonMobil facility attributed to the PAH found in sediments within the study area.
- The extent to which natural attenuation of the petroleum-source PAH has occurred.

2.0 In addition to PAH, characterize sediment for certain principal threat waste (PTW) chemicals that, based on previous investigations, may be found in the proposed study area sediments.

This document provides the overall study design, team responsibilities, sediment collection methods, chemical analysis, quality assurance methods, and data reporting requirements for this study. A site specific health and safety plan (HASP) has also been prepared for this field effort (Appendix B).

## **1.2 Document Organization**

The remaining sections of this document describe the sampling plan and field procedures that will be used during sediment collection. Section 2 describes the sampling approach. Section 3 describes the project organization and key personnel roles. Section 4 includes the detailed procedures that will be used in the field, including specific sampling methods. Section 5 discusses the data management plan for the field sampling program. Section 6 summarizes the laboratory analysis program. Section 7 summarizes how the data will be reported. Finally, references are provided in Section 8.

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## 2.0 SAMPLING APPROACH

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This section describes the sampling design and sampling locations that will support the objectives of this investigation. Conditions encountered in the field may result in modifications to the sampling design; however, ExxonMobil will be contacted when modifications to the sampling design are necessary.

Both the number of samples and specific sampling locations were developed using Visual Sampling Plan software (Matzke et al. 2014). This tool supports the development of a defensible sampling plan based on statistical sampling theory and the statistical analysis of sample results to support confident decision making. A non-parametric systematic sampling approach with a random start was employed such that the resulting data can be used to construct a confidence interval on the true population median value within the remedial alternative area.

Target sample locations are shown in Figure 1. Coordinates and other information about the target sampling locations are provided in Table 1.

Samples collected for this study will be used for one or both of the following purposes:

- Forensic chemical analysis; and
- PTW characterization.

Table 2 identifies the samples to be collected at each location and the analyses to be performed. The following subsections describe the types of samples to be collected and parameters to be analyzed for each of the above purposes.

### 2.1 Forensic Chemical Analysis

#### 2.1.1 Sample Type

All sediment samples, both surface and subsurface, collected for this study will be analyzed for a suite of compounds used for forensic chemical analysis. Sediments at all locations will be collected using a vertically-driven sediment vibracorer. Depending upon the elevation of the mudline, up to 4 sediment depth intervals (1 surface and 3 subsurface) will be collected from each core (Figure 2):

- Interval A. Surface sediment samples will be collected from 0 to 1 foot below the mudline at all locations.
- Interval B. Subsurface samples will be collected from a depth of -48 to -49 feet Columbia River Datum (CRD). This interval will not be collected from locations where the mudline is below -47 feet CRD (see Figure 2).
- Interval C. Subsurface samples will be collected from a depth of -51 to -52 feet CRD. This interval will not be collected from locations where the mudline is below -50 feet CRD.
- Interval D. Subsurface samples will be collected from a depth of -53 to -54 feet CRD. This interval will not be collected from locations where the mudline is below -52 feet CRD.

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### **2.1.2 Sample Analyses**

The suite of compounds used for forensic chemical analysis consists of:

- Total petroleum hydrocarbons (TPH) and n-alkanes
- PAHs and alkyl PAHs

Additionally, all samples for forensic chemical analysis will be analyzed for the ancillary measurements:

- Grain Size
- Total organic carbon (TOC)

Further descriptions of all analyses and target analytes are presented in Appendix A.

## **2.2 PTW Characterization**

### **2.2.1 Sample Type**

A subset of the sediment samples undergoing forensic chemical analysis will also be characterized for additional PTW chemicals (Table 2). A randomized sampling strategy was used to determine the specific samples for PTW characterization. The data resulting from this randomized approach will allow statistical inferences to be drawn regarding the PTW concentration distributions within the remedial alternative area.

### **2.2.2 Sample Analyses**

In addition to the forensic chemistry analyte list (Section 2.1.2), samples for PTW characterization will be analyzed for:

- Organochlorine pesticides
- Polychlorinated biphenyl (PCB) congeners
- Dioxin/furan congeners

Further descriptions of these analyses and target analytes are presented in Appendix A.

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## 3.0 PROJECT ORGANIZATION

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This section presents the organizational structure for sampling and analysis activities associated with this investigation, including fieldwork, laboratory analyses, and data management.

### 3.1 Team Organization and Responsibilities

Sampling and analysis activities will be performed by NewFields and their subcontractors. The organizational structure of the lead sampling and analysis personnel and associated laboratories are described below.

#### 3.1.1 Project Manager

Dr. Allen Uhler of NewFields will serve as the Project Manager for this investigation. The Project Manager is responsible for all technical aspects of the contract. The Project Manager assures the technical requirements of the contract are met, monitors contract performance, and maintains communication with the ExxonMobil project manager.

##### **Project Manager**

Dr. Allen Uhler

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#### 3.1.2 Project Coordinator

Ms. Kerylynn Krahforst of NewFields will serve as the Sampling and Analysis Coordinator, responsible for all facets of the sampling and analysis program. Her specific responsibilities include the following:

- Coordinate the field and laboratory analyses;
- Ensure that laboratory capacity is sufficient to undertake the required analyses in a timely manner;
- Ensure adherence to the schedule by tracking sampling, laboratory analysis, validation, and data management tasks; and
- Inform the Project Coordinator of any decisions that involve changes to the FSP and QAPP.

##### **Project Coordinator**

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#### 3.1.3 Field Coordinator

Dr. Jonathan Nuwer of NewFields will serve as the Field Coordinator, responsible for overall coordination of all field sampling tasks. Specifically, he will be responsible for the following:

- Oversee the planning and coordination for all sampling efforts;

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- Coordinate vessel and equipment logistics;
  - Oversee all aspects of the sampling to ensure that the appropriate procedures and methods are used; and
  - Maintain the field logs.

He will work closely with the Sampling and Analysis Coordinator and will be immediately notified if problems occur in the field. If changes to the FSP or QAPP are warranted, he will immediately notify the Sampling and Analysis Coordinator.

**Field Manager**

Dr. Jonathan Nuwer

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**3.1.4 Field Crews**

Field staff for all sampling events will consist of NewFields employees. The operators of the sampling vessel and equipment, as appropriate, will supply additional staff. Station positioning will be the responsibility of the vessel operator. For all sampling tasks, the field crews will include the following individuals: site safety officer, field task leader, and field crew.

The site safety officer will have the following responsibilities:

- Correct any work practices or conditions that may result in personnel injury or exposure to hazardous materials;
- Determine appropriate personal protection levels and the necessary clothing and equipment, and oversee its proper use;
- Verify that the field crew is aware of the provisions of the HASP and instructed in safe work practices; and
- Verify that the field crew has received the required safety training.

The field task leader will have the following responsibilities:

- Ensure that all activities adhere to the FSP and QAPP;
- Inform the field coordinator of any decisions that involve changes to the FSP and QAPP;
- Mobilize and prepare for field work; and
- Ensure sample custody, including chain-of-custody.

Various field staff from NewFields will assist in sample collection, handling, and storage. They may maintain field sampling logs and notebooks and will be responsible for properly labeling sample containers. It is the responsibility of the field staff to report any problems or potential changes to the FSP and QAPP to the field task leader.

**3.1.5 Data Management**

Kerylynn Krahforst of NewFields will have primary responsibility for data management.

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### **3.1.6 Laboratory Services**

The laboratory contracted to perform the analysis of forensic chemicals, PCB congeners, organochlorine pesticides, grain size and TOC is Alpha Analytical (Alpha). The laboratory contracted to perform dioxin/furan congener analysis is SGS North America, Inc. (SGS).

The laboratories will assign a project manager responsible for assuring that all analyses performed meet project and measurement quality objectives. It is the Project Manager's responsibility to report any problems or to the Project Coordinator. The Project Coordinator will provide technical coordination and oversight to the project managers and laboratory activities. The laboratory Project Managers are:

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## 4.0 SAMPLE COLLECTION PROCEDURES

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This section describes the detailed procedures for positioning and sample collection; record keeping, sample handling, storage, and shipping; and field quality control procedures.

### 4.1 Sampling Vessel

The 36-foot vessel R/V *Nancy Anne*, owned and operated by Marine Sampling Systems, Inc. (MSS), will be used for all sediment collection activities. The *Nancy Anne* is a welded aluminum catamaran with a large forward working deck, purpose-built by MSS in 1994 to deploy sediment sampling equipment. Sediments will be collected from the platform using MSS's sampling equipment, including a proprietary 'Marine Sampling Systems Vibracorer' and power grab sampler. MSS has more than 20 years of experience sediment sampling, including numerous Portland Harbor sediment investigations.

### 4.2 Station Positioning and Vertical Control

Latitude and longitude coordinates will be obtained using a differential global positioning and longitude coordinates will be obtained using a real time kinematic (RTK) global positioning system (GPS). The standard projection method to be used during field activities is Horizontal Datum: North American Datum of 1983 (NAD83), State Plane Coordinate System, Oregon North Zone. The positioning objective is to accurately determine and record the positions of all sampling locations to within  $\pm 2$  meters.

Station positioning from the sampling vessel will be accomplished using RTK-GPS, which consists of a GPS receiver on the sampling platform and a differential receiver located at a horizontal control point. At the control point, the GPS-derived position is compared with the known horizontal location, offsets or biases are calculated, and the correction factors are telemetered to the GPS receiver located on the sampling platform. The GPS receiver routes latitude and longitude to an integrated navigation system, which displays the platform's position in plan view. Navigation data, such as range and bearing from the target sampling location, are provided at a user-defined scale to guide the sampling platform's pilot to the desired location.

Vertical positioning is required to establish the elevation of the riverbed at the sampling locations. While the sampling device is in place at the sampling station, the tidal height (water surface elevation) is determined by RTK-GPS. Further, the depth to mudline will be measured using a fathometer immediately prior to or during the sampling. These two pieces of elevation information allow determination of the mudline elevation. Vertical measurements will be recorded to the nearest 0.1 foot.

During sediment sampling, the combination of real-time river levels and subsurface obstructions may preclude collecting a sample at the target location. Attempts will be made to relocate the sampling location to a nearby area that has comparable sediment characteristics and rationale objectives as the initial location.

### 4.3 Field Documentation

A complete record of field activities will be maintained. Documentation necessary to meet quality assurance (QA) objectives for this project include field notes and field forms (Appendix C), sample container labels, and chain-of-custody forms. The field documentation will provide descriptions of all sampling activities, sampling personnel, and weather conditions, and will record all modifications, decisions, and/or corrective actions to the study design and procedures.

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Field logbook(s) will be kept on site during field operations. Daily activities will be recorded in a bound field logbook of water-resistant paper. Separate logbooks, consisting of bound, paginated field forms, will be kept for sediment cores and an inventory of sample containers (separate from chain-of-custody documentation). Examples of the field forms to be used are presented in Appendix C. All entries will be made legibly, in indelible ink, and will be signed and dated. Information recorded will include the following:

- Date, time, place, and location of sampling;
- Onsite personnel and visitors;
- Daily safety discussion and any safety issues;
- Quality control samples;
- Field measurements and their units;
- Observations about site, location, and samples (weather, current, odors, appearance, etc.); and
- Equipment decontamination verification.

Field logbooks are intended to provide sufficient data and observations to enable participants to reconstruct events that occur during project field activities. Entries should be factual, detailed, and objective. Unless restricted by weather conditions, all original data recorded in field logbooks and on sample identification labels, chain-of-custody records, and field forms will be written in waterproof ink. If an error is made, the individual responsible may make corrections simply by crossing out the error and entering the correct information. The erroneous information should not be obliterated. All corrections must be initialed and dated. All documentation, including voided entries, must be maintained within project files.

#### **4.4 Sediment Collection and Processing Procedures**

Specific sediment collection, processing, and sample handling methods are described in this section. Standard operating procedures (SOPs) for collection of sediments are presented in Appendix D.

##### **4.4.1 Equipment**

Core samples will be collected from the locations shown in Figure 1 (target coordinates listed in Table 1) using a vibracorer equipped with aluminum core tubes up to 12 feet in length. The vibracorer offers a high rate of production, superior retention of shallow samples, and a greater sample volume compared to conventional drilling equipment. It also provides greater penetration capabilities than piston-type or conventional gravity corers when encountering compact subsurface sediments.

The MSS vibracorer system is well-suited for sediment coring in Portland Harbor:

- The corer has a guide/base assembly which sits on the sediment surface, maintaining the core tube and driving assembly vertical to the surface. If hard materials are encountered at the surface, as was often the case in ExxonMobil's 2014 sampling (NewFields 2014), there is no tendency to tip when the lift cable to the vessel is slacked.
- The guide/base assembly allows a 2:1 block system to double the vessel winch pull-out force, which is especially important in coarse sand or gravel materials that may be encountered in Portland Harbor.
- The vibracorer is hydraulically driven. The driving head assembly is proprietary and generates a 60 Hz, 3000 pound force on the core tube assembly.
- Penetration is monitored ultrasonically.

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At some locations the vibracorer may be unable to penetrate deep enough into the sediment bed to recover an intact core. This may be the case where either gravel or bedrock material is encountered. In the event that the vibracorer is unable to recover an intact sediment core from a location after multiple attempts, collection of a surface sediment sample (0 to 1 foot below the mudline) will be attempted with a power grab sampler.

#### **4.4.2 Collection**

Prior to sampling, target station coordinates will be entered into the navigation system. Once the sampling equipment has been deployed, the actual position will be recorded when the equipment is on the riverbed. All samples will be collected within 10 to 15 meters of the target sampling location, when possible.

Sediment cores will be collected using a customized vibracorer deployed from the R/V *Nancy Anne* operated by MSS. The vibracorer uses a hydraulic system that vibrates and drives a 4-inch outside diameter, aluminum core tube into the sediment. A continuous sediment sample is retained within the tubing with the aid of a stainless-steel core cutter/catcher attached to the bottom of each aluminum tube.

The vibracorer will be deployed off the foredeck of the vessel and slowly lowered to the sediment surface. The vibracorer will be allowed to penetrate beyond the target sampling depth or refusal, with penetration monitored in real-time using an ultrasonic depth sensor. Once penetration is complete, the vibracorer is retrieved and placed on the deck. The core will be examined at each end to verify that sufficient sediment was retained and that over-penetration did not occur.

Cores may be cut horizontally to create smaller core sections that are easier to handle. All core ends will be capped, sealed with duct tape, and labeled with the sampling location, core number, date and time of collection, depth, and direction arrows indicating the top end. Core sections will be stored vertically. The sampling location, core number, station coordinates, date and time of collection, field crew, and weather conditions will be recorded in the field log. The coring log will include penetration depth, length of core recovered, calculated recovery, measured water depth, and the calculated mudline elevation.

#### **4.4.3 Processing**

Sediment cores will be transferred daily from the sampling vessel to a cargo truck for transport to the processing facility. Cores will be transported vertically in ice baths. All sediment processing will take place at Analytical Resources, Inc. (ARI) in Tukwila, Washington.

At the processing facility the aluminum core tubes will be cut along the vertical axis using a circular saw. The saw blade depth will be set just deep enough to cut cleanly through the aluminum tube without disturbing the sediment inside. Two passes with the circular saw are made on opposite sides of the core tube, splitting the core tube directly in half down the centerline. The aluminum core tube is then removed and the sediment core is rolled out onto a clean, aluminum-foil-covered processing table.

The sediment will then be split along this axis using a spatula. Once a core is split, it will be inspected, described, and photographed. The following information will be recorded for each core:

- Physical sediment description
- Odor
- Visual stratification

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- Vegetation
  - Debris
  - Evidence of biological activity
  - Presence of sheen
  - Presence of tar balls
  - Other distinguishing characteristics or features

Because the actual sediment core sampling locations and mudline depths may differ from those presented in Table 1, the water depth, tidal height (as discussed in Section 4.2), and percent recovery data will be used in the field to calculate the maximum depth of sample intervals using the following equation:

$$D = (E_{ML} - E_S) \times (PR/100)$$

where:

D = Depth from surface of sediment core (feet)

E<sub>ML</sub> = Mudline elevation (feet CRD)

E<sub>S</sub> = Sample elevation (feet CRD)

PR = Percent recovery

For example, if a core with 75 percent recovery is collected at a mudline elevation of -46 feet CRD, a sample interval with a bottom elevation of -52 feet CRD is located at 4.5 feet deep in the sediment core. The above calculation will be performed for all sample intervals in all cores collected using a pre-prepared spreadsheet available on the field laptop computer. Once the core has been split, sampling depth intervals will be delineated in the sediment using a stainless steel spoon based on the above equation.

Sediment will be collected from throughout the entire acceptable sample interval, with the exception of sediment coming in direct contact with the sampling equipment, and will be placed in a decontaminated stainless steel pan. The pan will be covered with foil until a sufficient volume of material is collected. Once a sufficient volume of sediment has been collected, the sample will be homogenized with a decontaminated stainless steel spoon until a consistent color and texture is achieved. Aliquots of the homogenized sediment will be placed in the appropriate pre-cleaned containers for chemical testing.

#### **4.4.4 Sample Handling and Storage**

The homogenized sample will be distributed to the appropriate sample containers according to the sample requirements identified in Table 3. All sample containers will be filled, leaving 0.5 to 1 inch of headspace to prevent the jars from breaking during storage. Sediment samples in the containers will be stored on ice, and will remain iced until packaged for shipping to the analytical laboratories. Rinsate blanks will be collected once per day and submitted to the laboratory for analysis.

Sample containers and preservatives, as well as coolers and packing material, will be supplied by the analytical laboratory. Commercially available pre-cleaned jars will be used, and the laboratory will maintain a record of certification from the suppliers. Sample containers will be clearly labeled at the time of sampling. Labels will include the project name, sample location and number, sampler's initials, analysis to be performed, date, and time. The nomenclature used for designating field samples is described in Section 5.4

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## 4.5 Equipment Decontamination Procedures

Sediment handling equipment that comes into direct contact with a sample, such as scoops, spoons, and mixing bowls, will be decontaminated in the following manner prior to use at each station:

- Rinse with site water
- Wash with brush and Liquinox or other phosphate-free detergent
- Double rinse with distilled water
- Rinse with methanol (omit if sampling for volatiles)

Decontamination of stainless-steel bowls and utensils will be performed before sampling and between each sediment core depth interval sample. Sample handling equipment also will be wrapped in aluminum foil following the methanol rinse. Before being used to remove sediment from the samplers, all equipment will be rinsed with distilled water. To minimize sample contamination, gloves will be replaced or thoroughly washed using Liquinox or other phosphate-free detergent and rinsed with distilled water before and after handling each sample, as appropriate.

Aluminum core tubes will not be reused.

## 4.6 Waste Disposal

Any excess sediment remaining on the vibracorer frame after core collection will be returned to the river in the vicinity of the collection site at the time of collection. Any sediment or water spilled on the deck of the sampling vessel will be washed into the surface waters at the collection site before proceeding to the next station.

Any excess sediment remaining after sediment core processing will be collected at the processing facility in 55 gallon waste drums. Full drums will be picked up from the processing facility by Clean Harbors and disposed of as non-hazardous material.

Used personal protective equipment (PPE) such as disposable gloves and supplies (e.g., paper towels and packaging) will be placed in plastic storage bags and disposed of as municipal waste. If PPE contains residual sediments, the PPE will be decontaminated using the procedures outlined in Section 4.5 and will be disposed of as non-hazardous material. Recyclable waste material (e.g., cardboard, aluminum cores) will be recycled as feasible.

## 4.7 Sample Handling and Transport

Sample possession must be traceable from the time of sample collection through laboratory analysis. To ensure samples are traceable, the following procedures will be followed.

### 4.7.1 Chain-of-Custody Procedures

Samples will be retained at all times in the field crew's custody until samples are delivered or shipped to the appropriate laboratory by NewFields personnel. All samples will be held and transported in coolers with ice or frozen gel-packs at approximately 0 to 6°C. Chain-of-custody forms will be initiated at the time of sample collection to ensure that all collected samples are properly documented and traceable through storage, transport, and analysis. Information tracked by the chain-of-custody records will include sample identification, date and time of sample collection and receipt, and analyses required.

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When all line items on the chain-of-custody form are completed or when the samples are relinquished, the sample collection custodian will sign and date the form, list the time, and confirm the completeness of all descriptive information contained on the form. Each individual who subsequently assumes responsibility for the samples will sign and date the chain-of-custody form. The signed and dated chain-of-custody forms will be enclosed in a plastic bag and taped to the inside lid of the cooler prior to shipping. The field chain-of-custody terminates when the laboratory receives the samples. The field sample custodian should retain a copy of the completed, signed chain-of-custody form(s) for project files.

Upon receipt of samples at the laboratories, the shipping container seal will be broken and the receiver will record the condition of the samples and the temperature of the temperature blank. The laboratories will maintain chain-of-custody internally to track handling and final disposition of all samples.

#### **4.7.2 Sample Shipping**

All samples will be stored in insulated coolers and preserved by cooling with ice or frozen gel-packs to a temperature of 0 to 6°C. Maximum sample holding and extraction times will be strictly adhered to by field personnel and the analytical laboratories. Preparation of jars for shipment will be performed in the following manner:

- Place individual sample containers in plastic bubble-pack bags, or zip-lock and wrap in bubble pack and secure with packaging tape.
- Prepare an empty insulated cooler by placing three to four ice packs in a garbage bag at the bottom of the cooler. Place sample containers in a garbage bag and fill with the sample bottles. Add additional bags of ice as needed to surround the bag containing the samples.
- Include a temperature blank in the cooler.
- Seal the cooler with strapping tape and a custody seal.

Samples for chemical analyses will be shipped via overnight courier to the analytical laboratories accompanied by the chain-of-custody record, which identifies the shipment contents. The chain-of-custody form will be signed by the individual relinquishing samples to the laboratory. Samples will be packaged and shipped in accordance with U.S. Department of Transportation regulations as specified in 49 Code of Federal Regulations (CFR) 173.6 and 49 CFR 173.24. NewFields field personnel will be responsible for the following:

- Packaging the samples;
- Ensuring the signed chain-of-custody forms are enclosed in a plastic bag and taped to the inside lid of the cooler;
- Applying a shipping label, a waybill, a custody seal, and strapping tape to the cooler; and
- Shipping the samples in accordance with the maximum holding time allowed for the analyses to be performed.

#### **4.8 Quality Control Procedures**

Quality control (QC) requirements will be instituted during sampling, laboratory analysis, and data management to ensure that the data quality objectives are met. Detailed information on QA/QC procedures, limits, and reporting are described in detail in the QAPP (Appendix A). Field QC requirements are described in the following sections. If QC problems are encountered, they will be brought to the attention of the Project Coordinator. Corrective actions, if appropriate, will be implemented to meet the project's data quality objectives.

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#### **4.8.1 Field QC Samples**

Field QC samples are used to evaluate the effectiveness of sample homogenization and within-sample variability (e.g., field duplicates), evaluate potential sources of sample contamination (e.g., rinsate), or confirm proper storage conditions (e.g., temperature blanks). The types of QC samples that will be collected are described in this section. The estimated numbers of field QC samples are included in Table 2.

##### **Duplicate Samples**

Field duplicate samples are multiple samples taken from a single sample composite after it is fully homogenized. Field duplicates will be designated for the same analyses as the original sample. The resulting data will provide information on the variability associated with sample handling and laboratory analysis operations. Field duplicates will be collected for approximately 5% of the sediment samples collected.

##### **Rinsate Blanks**

Introduction of chemical contaminants during sampling and analytical activities will be assessed by the analysis of blanks. Rinsate blanks, consisting of sample processing equipment rinsates, will be generated for all chemical parameter groups at a frequency of once per day of sediment processing and submitted for analysis to the laboratory.

##### **Temperature Blanks**

Temperature blanks are used to measure and ensure cooler temperature upon receipt to the laboratory. One temperature blank will be prepared and submitted with each cooler shipped to the analytical laboratory. The temperature blank will consist of a sample jar containing deionized water that will be packed into the cooler in the same manner as the rest of the samples and labeled "temp blank."

#### **4.8.2 Performance Audits**

The Field QA Manager will conduct field performance audits at least once during each field program. The audits will involve assessing the sample collection and processing procedures relative to the procedures described in this FSP and to standard collection procedures. Data recording procedures will be reviewed for completeness.

#### **4.8.3 Corrective Actions**

Results of the field performance audit may identify the need for corrective actions. The Field QA Manager will immediately institute the necessary corrective actions, complete a corrective action form (see Appendix C), and conduct an additional audit to ensure that the correct procedures continue to be followed.

If corrective actions require a departure from the FSP, these changes will be documented on a field change request form (see Appendix C). In any other circumstances where sampling conditions are unexpected, the appropriate sampling actions consistent with project objectives will be conducted after the Field QA Manager informs the Sampling and Analysis Coordinator. This change will be noted in the field log, and a change request form will be completed for the project files.

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## 5.0 FIELD DATA MANAGEMENT PLAN

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During field operations, effective data management is the key to providing consistent, accurate, and defensible documentation of data quality. Field data will include descriptive and geographical information associated with sediment and water collection. Daily field records (a combination of field logbooks and field data sheets) and navigational records will make up the main documentation for field activities. The plan components most applicable to field activities are summarized in the following sections.

### 5.1 Field Logbooks

All field activities and observations will be noted in field logbooks during the fieldwork. The procedures and requirements for logbook entries are detailed in Section 4.3.

### 5.2 Field Data Sheets

Field data sheets and sample description forms will be completed for all samples and kept in the project file as a permanent record of the sampling activities. If field data sheet entries are entered in an electronic format, each sheet will indicate who completed the data entry and when. The field task leader is responsible for ensuring that all field data sheets are correct and that they become part of the permanent file.

### 5.3 Field Data Management

As soon after collection as possible, field notes and data sheets will be scanned to create an electronic record for use in creating the cruise report. Field data will be hand-entered into the database. Twenty percent of the transferred data will be verified based on hard copy records. Electronic QA checks to identify anomalous values will also be conducted following entry.

### 5.4 Sample Identification

Station location identification numbers for each target location are listed in Table 2. During sample collection, a unique code will be assigned to each sample as part of the data record. This code will indicate the project, sampling location, sample type, and level of replication/duplication.

All samples will be assigned a unique identification number based on a sample designation scheme designed to meet the needs of the field personnel, laboratory data management, validation chemists, and data users (Table 2). Sample identifiers will consist of three to four components separated by dashes:

- The first component, PH15, identifies the data as belonging to the Portland Harbor 2015 investigation.
- The second component will consist of a two-digit number identifying the coring station. Leading zeros will be used for stations with numbers below 10 for ease of data management and correct sorting (e.g., 05).

- 
- The third component will be used to code sediment sampling depth intervals. A single letter will be placed after the station number to designate the following sample intervals:

A – Surface sediment sample (0 to 1 foot below mudline)

B – Subsurface sediment sample collected between -48 and -49 feet CRD

C – Subsurface sediment sample collected between -51 and -52 feet CRD

D – Subsurface sediment sample collected between -53 and -54 feet CRD

- The fourth component will only be used for filed QC samples and will consist of two letters. These field samples are described further in Section 4.9.1. Designations for field QC samples are as follows:

FD – “field duplicate”

RB – “rinsate blank”

## 5.5 Chain-of-Custody

The chain-of-custody forms and procedures are described in Section 4.7.1.

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## 6.0 LABORATORY ANALYSIS

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As noted in Section 2.0, samples collected for this study will be used for one or both of the following purposes:

- Forensic chemical analysis
- PTW characterization

All sediment samples will be analyzed for forensic chemicals and the ancillary measurements grain size and TOC. A subset of the samples will also be analyzed for PTW chemicals. The required analyses for each sample that will be collected are summarized in Table 2. The analytical methods to be applied are listed in Table 3, with details regarding laboratory methodology, target analytes, and detection limits provided in the QAPP (Appendix A).

Analytical methods and QC measurements and criteria are based on current Contract Laboratory Program (CLP) and SW-856 requirements, and EPA guidance. All samples will be maintained according to the appropriate holding times and temperatures for each analysis, as summarized in Table 3. Field QC sample requirements are described in Section 4.8.1.

Laboratory QA will be implemented as described in the QAPP and according to each of the identified laboratories' respective QA programs, plans, and SOPs. Additional information on analytical methods and laboratory QA program plans for each laboratory is provided in the QAPP (Appendix A).

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## 7.0 REPORTING

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NewFields will prepare a sediment field sampling report and submit to ExxonMobil within 30 days of completing the field sample collection effort described in this FSP. The field sampling report will summarize field sampling activities, including sample location coordinates, sample collection methods, and any deviations from the FSP.

Subsequently, NewFields will prepare a written report documenting all results of analytical testing and forensic analyses. At a minimum, the following will be included in the Final Report:

- Summary of sampling, chemical testing, QA/QC procedures, and any deviations from the approved QAPP;
- Tables with sampling location coordinates, mudline elevations, and penetration depths.
- Figures showing actual sampling locations;
- Tables indicating the analyses performed on each sample;
- Tables with analytical results for chemical testing with laboratory qualifiers provided; and
- A summary of QC data.

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## 8.0 REFERENCES

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- LWG (Lower Willamette Group). (2015). Portland Harbor Remedial Investigation/Feasibility Study. Draft Final Feasibility Study Report. Prepared for the Lower Willamette Group by Anchor QEA, Windward Environmental, Kennedy/Jenks Consultants, and Integral Consulting, Inc. July 2015.
- Matzke BD, Wilson JE, Newburn LL, Dowson ST, Hathaway JE, Sego LH, Bramer LM, and Pulsipher BA. (2014) Visual Sampling Plan Version 7.0 User's Guide. PNNL-23211, Pacific Northwest National Laboratory, Richland, Washington.
- NewFields (2014) Portland Harbor Sediment Forensic Chemistry Study, Portland, Oregon. Cruise Report. Prepared for ExxonMobil by NewFields, Edmonds, Washington. September 2014.
- Stout SA, Uhler AD, and Emsbo-Mattingly SD. (2004). Comparative evaluation of background and anthropogenic hydrocarbons in surficial sediments from nine urban waterways. Environ. Sci. Technol., 38(11): 2987-2044.

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## TABLES

Table 1. Target Sediment Core Sampling Locations

Station ID	Coordinates (NAD 83 HARN)		Mudline Elevation (feet) <sup>1</sup>		Core length to - 54 ft CRD <sup>1</sup>	Number of depth intervals to be sampled
	Latitude	Longitude	NAVD88	CRD		
PH15-01	45.592545	-122.775140	-40.2	-45.4	8.6	4
PH15-02	45.591918	-122.774568	-40.6	-45.8	8.2	4
PH15-03	45.591401	-122.774032	-40.4	-45.6	8.4	4
PH15-04	45.590825	-122.773449	-40.3	-45.5	8.5	4
PH15-05	45.590351	-122.772853	-40.0	-45.2	8.8	4
PH15-06	45.589848	-122.772025	-40.6	-45.8	8.2	4
PH15-07	45.589355	-122.771323	-40.1	-45.3	8.7	4
PH15-08	45.588702	-122.770957	-40.9	-46.1	7.9	4
PH15-09	45.588149	-122.770418	-40.5	-45.7	8.3	4
PH15-10	45.586846	-122.768702	-41.3	-46.5	7.5	4
PH15-11	45.586260	-122.767969	-45.7	-50.9	3.1	2
PH15-12	45.585658	-122.767278	-44.7	-49.9	4.1	3
PH15-13	45.585084	-122.766506	-41.7	-46.9	7.1	4
PH15-14	45.592446	-122.774475	-43.7	-48.9	5.1	3
PH15-15	45.591945	-122.773834	-42.5	-47.7	6.3	3
PH15-16	45.591427	-122.773236	-40.9	-46.1	7.9	4
PH15-17	45.590878	-122.772615	-41.1	-46.3	7.7	4
PH15-18	45.590361	-122.771995	-41.9	-47.1	6.9	3
PH15-19	45.589860	-122.771332	-43.3	-48.5	5.5	3
PH15-20	45.589326	-122.770733	-44.6	-49.8	4.2	3
PH15-21	45.588809	-122.770114	-43.2	-48.4	5.6	3
PH15-22	45.588329	-122.769474	-42.7	-47.9	6.1	3
PH15-23	45.587928	-122.768899	-42.1	-47.3	6.7	3
PH15-24	45.587547	-122.768297	-42.2	-47.4	6.6	3
PH15-25	45.586999	-122.767603	-43.0	-48.2	5.8	3
PH15-26	45.586369	-122.766889	-44.8	-50.0	4	3
PH15-27	45.585797	-122.766178	-46.4	-51.6	2.4	2
PH15-28	45.585196	-122.765466	-44.2	-49.4	4.6	3
PH15-29	45.592574	-122.773526	-44.7	-49.9	4.1	3
PH15-30	45.592072	-122.772907	-42.9	-48.1	5.9	3
PH15-31	45.591554	-122.772309	-44.1	-49.3	4.7	3
PH15-32	45.591006	-122.771644	-43.1	-48.3	5.7	3
PH15-33	45.590473	-122.771023	-46.2	-51.4	2.6	2
PH15-34	45.589972	-122.770404	-47.1	-52.3	1.7	1
PH15-35	45.589439	-122.769784	-49.1	-54.3	0	1
PH15-36	45.588859	-122.769101	-45.5	-50.7	3.3	2
PH15-37	45.588462	-122.768612	-41.2	-46.4	7.6	4
PH15-38	45.588063	-122.768013	-41.7	-46.9	7.1	4
PH15-39	45.587724	-122.767235	-41.2	-46.4	7.6	4
PH15-40	45.587137	-122.766524	-40.6	-45.8	8.2	4
PH15-41	45.586537	-122.765812	-42.9	-48.1	5.9	3
PH15-42	45.585936	-122.765099	-47.6	-52.8	1.2	1
PH15-43	45.592717	-122.772578	-45.5	-50.7	3.3	2
PH15-44	45.592200	-122.771958	-45.7	-50.9	3.1	2
PH15-45	45.591667	-122.771315	-46.3	-51.5	2.5	2
PH15-46	45.591135	-122.770673	-47.7	-52.9	1.1	1
PH15-47	45.590617	-122.770053	-48.0	-53.2	0.8	1
PH15-48	45.590084	-122.769432	-47.7	-52.9	1.1	1
PH15-49	45.589567	-122.768813	-48.5	-53.7	0.3	1
PH15-50	45.588574	-122.767854	-44.0	-49.2	4.8	3

Notes:

- Based on bathymetric survey data collected in April 2004
- CRD – Columbia River Datum

Table 2. Samples and Analyses

Station ID	Sample ID	Depth Interval	Forensic Chemical Analysis		Ancillary Measurements		PTW Characterization		
			PAHs and alkyl PAHs	TPH and n-alkanes	Grain Size	TOC	Organo-chlorine Pesticides	PCB Congeners	Dioxin/Furan Congeners
PH15-01	PH15-01-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-01-B	-48 to -49 ft CRD	X	X	X	X			
	PH15-01-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-01-D	-53 to -54 ft CRD	X	X	X	X			
PH15-02	PH15-02-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-02-B	-48 to -49 ft CRD	X	X	X	X			
	PH15-02-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-02-D	-53 to -54 ft CRD	X	X	X	X			
PH15-03	PH15-03-A	0 to 1 ft below mudline	X	X	X	X	X	X	X
	PH15-03-B	-48 to -49 ft CRD	X	X	X	X	X	X	X
	PH15-03-C	-51 to -52 ft CRD	X	X	X	X	X	X	X
	PH15-03-D	-53 to -54 ft CRD	X	X	X	X	X	X	X
PH15-04	PH15-04-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-04-B	-48 to -49 ft CRD	X	X	X	X			
	PH15-04-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-04-D	-53 to -54 ft CRD	X	X	X	X			
PH15-05	PH15-05-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-05-B	-48 to -49 ft CRD	X	X	X	X			
	PH15-05-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-05-D	-53 to -54 ft CRD	X	X	X	X			
PH15-06	PH15-06-A	0 to 1 ft below mudline	X	X	X	X	X	X	X
	PH15-06-B	-48 to -49 ft CRD	X	X	X	X	X	X	X
	PH15-06-C	-51 to -52 ft CRD	X	X	X	X	X	X	X
	PH15-06-D	-53 to -54 ft CRD	X	X	X	X	X	X	X
PH15-07	PH15-07-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-07-B	-48 to -49 ft CRD	X	X	X	X			
	PH15-07-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-07-D	-53 to -54 ft CRD	X	X	X	X			
PH15-08	PH15-08-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-08-B	-48 to -49 ft CRD	X	X	X	X			
	PH15-08-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-08-D	-53 to -54 ft CRD	X	X	X	X			
PH15-09	PH15-09-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-09-B	-48 to -49 ft CRD	X	X	X	X			
	PH15-09-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-09-D	-53 to -54 ft CRD	X	X	X	X			

Table 2. (Continued)

Station ID	Sample ID	Depth Interval	Forensic Chemical Analysis		Ancillary Measurements		PTW Characterization		
			PAHs and alkyl PAHs	TPH and n-alkanes	Grain Size	TOC	Organo-chlorine Pesticides	PCB Congeners	Dioxin/Furan Congeners
PH15-10	PH15-10-A	0 to 1 ft below mudline	X	X	X	X	X	X	X
	PH15-10-B	-48 to -49 ft CRD	X	X	X	X	X	X	X
	PH15-10-C	-51 to -52 ft CRD	X	X	X	X	X	X	X
	PH15-10-D	-53 to -54 ft CRD	X	X	X	X	X	X	X
PH15-11	PH15-11-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-11-D	-53 to -54 ft CRD	X	X	X	X			
PH15-12	PH15-12-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-12-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-12-D	-53 to -54 ft CRD	X	X	X	X			
PH15-13	PH15-13-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-13-B	-48 to -49 ft CRD	X	X	X	X			
	PH15-13-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-13-D	-53 to -54 ft CRD	X	X	X	X			
PH15-14	PH15-14-A	0 to 1 ft below mudline	X	X	X	X	X	X	X
	PH15-14-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-14-D	-53 to -54 ft CRD	X	X	X	X			
PH15-15	PH15-15-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-15-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-15-D	-53 to -54 ft CRD	X	X	X	X			
PH15-16	PH15-16-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-16-B	-48 to -49 ft CRD	X	X	X	X			
	PH15-16-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-16-D	-53 to -54 ft CRD	X	X	X	X			
PH15-17	PH15-17-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-17-B	-48 to -49 ft CRD	X	X	X	X			
	PH15-17-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-17-D	-53 to -54 ft CRD	X	X	X	X			
PH15-18	PH15-18-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-18-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-18-D	-53 to -54 ft CRD	X	X	X	X			
PH15-19	PH15-19-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-19-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-19-D	-53 to -54 ft CRD	X	X	X	X			
PH15-20	PH15-20-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-020-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-20-D	-53 to -54 ft CRD	X	X	X	X			
PH15-21	PH15-21-A	0 to 1 ft below mudline	X	X	X	X	X	X	X
	PH15-21-C	-51 to -52 ft CRD	X	X	X	X	X	X	X
	PH15-21-D	-53 to -54 ft CRD	X	X	X	X	X	X	X

Table 2. (Continued)

Station ID	Sample ID	Depth Interval	Forensic Chemical Analysis		Ancillary Measurements		PTW Characterization		
			PAHs and alkyl PAHs	TPH and n-alkanes	Grain Size	TOC	Organo-chlorine Pesticides	PCB Congeners	Dioxin/Furan Congeners
PH15-22	PH15-22-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-22-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-22-D	-53 to -54 ft CRD	X	X	X	X			
PH15-23	PH15-23-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-23-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-23-D	-53 to -54 ft CRD	X	X	X	X			
PH15-24	PH15-24-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-24-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-24-D	-53 to -54 ft CRD	X	X	X	X			
PH15-25	PH15-25-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-25-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-25-D	-53 to -54 ft CRD	X	X	X	X			
PH15-26	PH15-26-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-26-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-26-D	-53 to -54 ft CRD	X	X	X	X			
PH15-27	PH15-27-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-27-D	-53 to -54 ft CRD	X	X	X	X			
PH15-28	PH15-28-A	0 to 1 ft below mudline	X	X	X	X	X	X	X
	PH15-28-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-28-D	-53 to -54 ft CRD	X	X	X	X			
PH15-29	PH15-29-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-29-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-29-D	-53 to -54 ft CRD	X	X	X	X			
PH15-30	PH15-30-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-30-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-30-D	-53 to -54 ft CRD	X	X	X	X			
PH15-31	PH15-31-A	0 to 1 ft below mudline	X	X	X	X	X	X	X
	PH15-31-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-31-D	-53 to -54 ft CRD	X	X	X	X			
PH15-32	PH15-32-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-32-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-32-D	-53 to -54 ft CRD	X	X	X	X			
PH15-33	PH15-33-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-33-D	-53 to -54 ft CRD	X	X	X	X			
PH15-34	PH15-34-A	0 to 1 ft below mudline	X	X	X	X	X	X	X
PH15-35	PH15-35-A	0 to 1 ft below mudline	X	X	X	X			
PH15-36	PH15-36-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-36-D	-53 to -54 ft CRD	X	X	X	X			

Table 2. (Continued)

Station ID	Sample ID	Depth Interval	Forensic Chemical Analysis		Ancillary Measurements		PTW Characterization		
			PAHs and alkyl PAHs	TPH and n-alkanes	Grain Size	TOC	Organo-chlorine Pesticides	PCB Congeners	Dioxin/Furan Congeners
PH15-37	PH15-37-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-37-B	-48 to -49 ft CRD	X	X	X	X			
	PH15-37-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-37-D	-53 to -54 ft CRD	X	X	X	X			
PH15-38	PH15-38-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-38-B	-48 to -49 ft CRD	X	X	X	X			
	PH15-38-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-38-D	-53 to -54 ft CRD	X	X	X	X			
PH15-39	PH15-39-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-39-B	-48 to -49 ft CRD	X	X	X	X			
	PH15-39-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-39-D	-53 to -54 ft CRD	X	X	X	X			
PH15-40	PH15-40-A	0 to 1 ft below mudline	X	X	X	X	X	X	X
	PH15-40-B	-48 to -49 ft CRD	X	X	X	X	X	X	X
	PH15-40-C	-51 to -52 ft CRD	X	X	X	X	X	X	X
	PH15-40-D	-53 to -54 ft CRD	X	X	X	X	X	X	X
PH15-41	PH15-41-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-41-C	-51 to -52 ft CRD	X	X	X	X			
	PH15-41-D	-53 to -54 ft CRD	X	X	X	X			
PH15-42	PH15-42-A	0 to 1 ft below mudline	X	X	X	X			
PH15-43	PH15-43-A	0 to 1 ft below mudline	X	X	X	X	X	X	X
	PH15-43-D	-53 to -54 ft CRD	X	X	X	X			
PH15-44	PH15-44-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-44-D	-53 to -54 ft CRD	X	X	X	X			
PH15-45	PH15-45-A	0 to 1 ft below mudline	X	X	X	X			
	PH15-45-D	-53 to -54 ft CRD	X	X	X	X			
PH15-46	PH15-46-A	0 to 1 ft below mudline	X	X	X	X			
PH15-47	PH15-47-A	0 to 1 ft below mudline	X	X	X	X			
PH15-48	PH15-48-A	0 to 1 ft below mudline	X	X	X	X			
PH15-49	PH15-49-A	0 to 1 ft below mudline	X	X	X	X			
PH15-50	PH15-50-A	0 to 1 ft below mudline	X	X	X	X	X	X	X
	PH15-50-C	-51 to -52 ft CRD	X	X	X	X	X	X	X
	PH15-50-D	-53 to -54 ft CRD	X	X	X	X	X	X	X
Total			146	146	146	146	27	27	27
Field Quality Control Samples	Field Duplicates (5%)		8	8	8	8	2	2	2
	Rinsate Blanks (1 per day)		5	5			5	5	5
TOTAL Field Samples			159	159	154	154	34	34	34

Table 3. Sample Preservation Requirements, Holding Times, and Containers

Analysis Group	Analysis	Method	Sample Size	Preservation	Holding Times	Containers	Laboratory
<b>Sediment</b>							
Forensic Chemical Analysis	PAHs and alkyl PAHs	EPA 8270 Modified	30 g	4 ± 2°C; store in the dark	14 days to extraction	8 ounce (oz) wide-mouth glass jar	Alpha
				< -10°C; store in the dark	1 year to extraction		
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
	TPH and n-alkanes	EPA 8015 Modified	30 g	4 ± 2°C; store in the dark	14 days to extraction		
				< -10°C; store in the dark	1 year to extraction		
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
Ancillary Measurements	TOC	EPA 9060 Modified	20 g	4 ± 2°C; store in dark	28 days	8 oz plastic bottle	
	Grain Size	ASTM D422	100 g	Ambient	None established		
PTW Characterization	Organochlorine Pesticides	EPA 8081A	30 g	4 ± 2°C; store in the dark	14 days to extraction	8 oz wide-mouth glass jar	Alpha
				< -10°C; store in the dark	1 year to extraction		
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
	PCB Congeners	EPA 8270 Modified	30 g	4 ± 2°C; store in the dark	14 days to extraction		Alpha
				< -10°C; store in the dark	1 year to extraction		
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
	Dioxin/Furan Congeners	EPA 1613B	10g	0-4°C; store in the dark	45 days to extraction	4 oz amber wide-mouth glass jar	SGS
				< -10°C; store in the dark	1 year to extraction		
Archive				< -20°C; store in the dark	1 year to extraction	8 oz wide-mouth glass jar	Alpha
<b>Water (Rinsate Blanks)</b>							
Forensic Chemical Analysis	PAHs and alkyl PAHs	EPA 8270 Modified	1 L	4 ± 2°C; store in the dark	14 days to extraction	2x 1 liter (L) amber bottle	Alpha
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
	TPH and n-alkanes	EPA 8015 Modified	1 L	4 ± 2°C; store in the dark	14 days to extraction		
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
PTW Characterization	Organochlorine Pesticides	EPA 8081A	1 L	4 ± 2°C; store in the dark	14 days to extraction	1 liter (L) amber bottle	Alpha
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
	PCB Congeners	EPA 8270 Modified	1 L	4 ± 2°C; store in the dark	14 days to extraction	1 liter (L) amber bottle	Alpha
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
	Dioxin/Furan Congeners	EPA 1613B	1 L	0-4°C; store in the dark	45 days to extraction	2 x 1 liter (L) amber bottle	SGS

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## FIGURES

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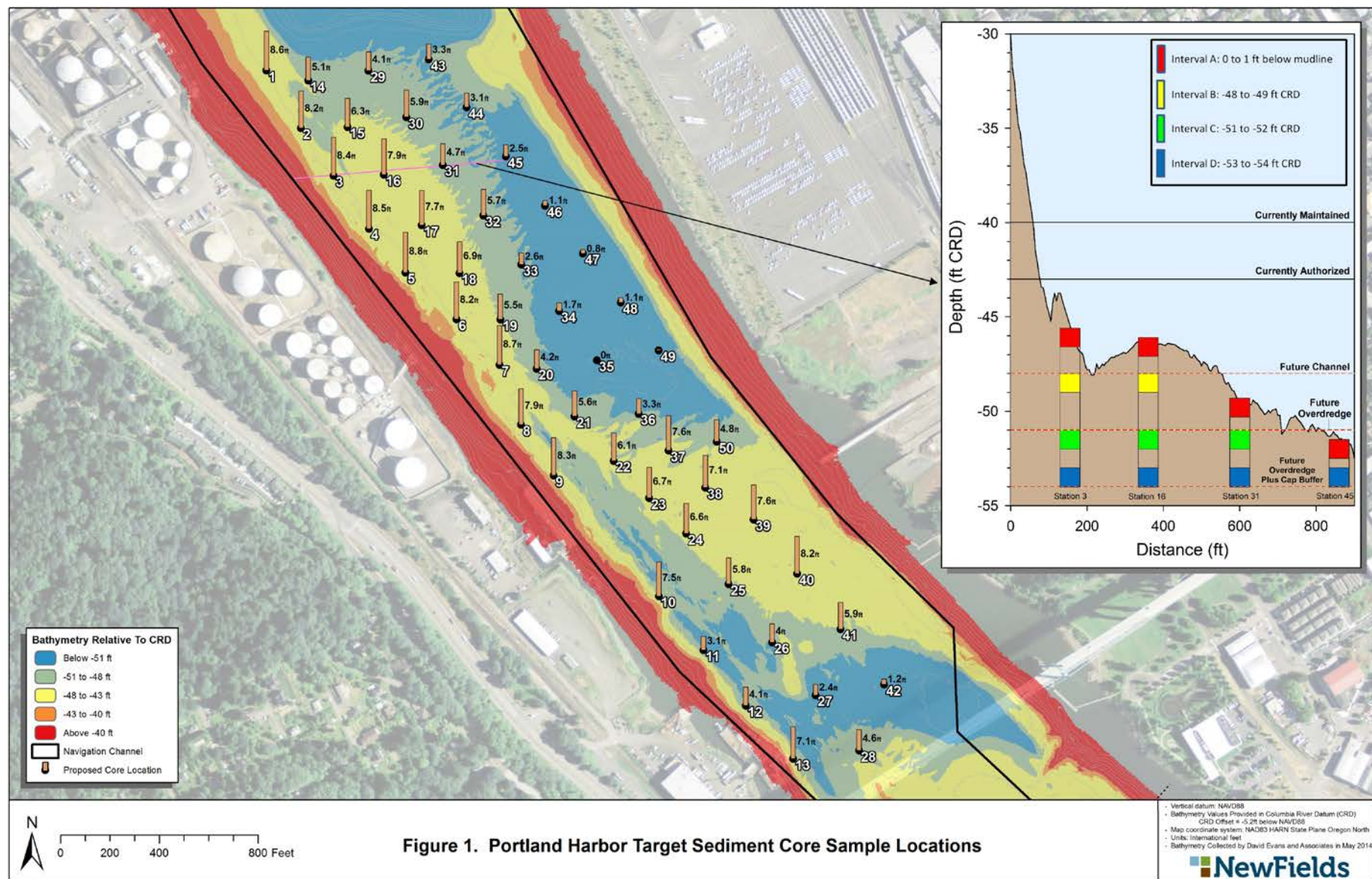
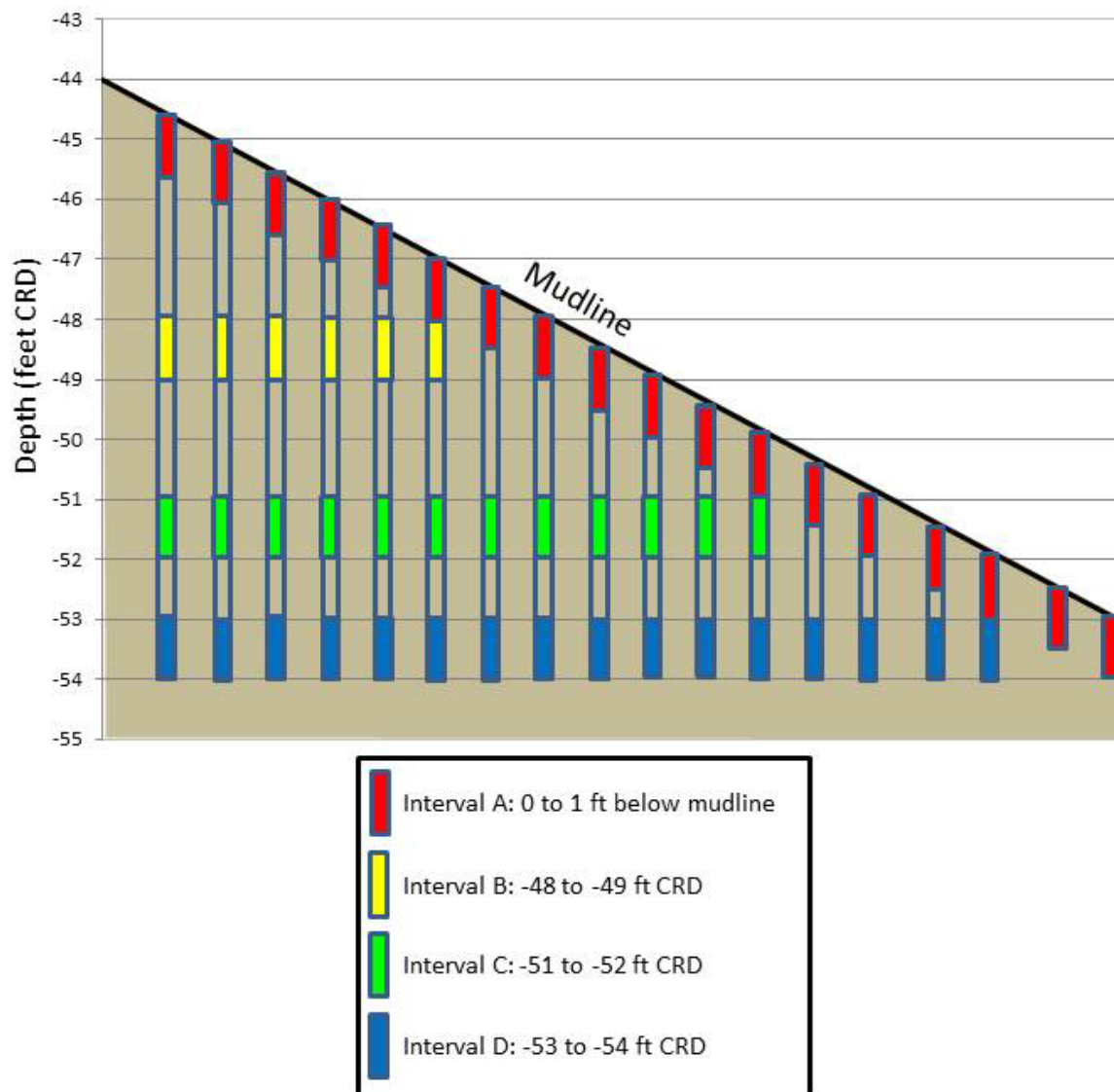


Figure 2. Sediment Core Sampling Schematic



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## **APPENDIX A: QUALITY ASSURANCE PROJECT PLAN**

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**PORTLAND HARBOR SUPPLEMENTAL SEDIMENT STUDY  
PORTLAND, OREGON**

**ANALYTICAL QUALITY ASSURANCE PROJECT PLAN (QAPP)**

**OCTOBER 14, 2015**

Prepared for:

**ExxonMobil**

Prepared by:



**300 Ledgewood Place, Suite 305  
Rockland, MA 02370**

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## Acronyms and Abbreviations

CCV	Continuing calibration verification
CFR	Code of Federal Regulations
COPCs	Contaminants of potential concern
DUP	Duplicate sample
EDD	Electronic data deliverable
FSP	Field Sampling Plan
GC	Gas chromatography
GC/FID	Gas chromatography/flame-ionization detection
GC/MS	Gas chromatography/mass spectrometry
HPLC	High performance liquid chromatography
HRGC/HRMS	High-resolution gas chromatography with high-resolution mass spectrometry
ICV	Initial calibration verification
LCS/LCSDs	Laboratory control sample/laboratory control sample duplicates
MDL	Method detection limit
MQOs	Measurement Quality Objectives
MS	Mass spectrometer
MS/MSDs	Matrix spike/matrix spike duplicates
PARCC	Precision, accuracy, representativeness, completeness, and comparability
OSHA	Occupational Safety and Health Administration
PAH	Polycyclic aromatic hydrocarbons
PCBs	Polychlorinated biphenyls
PTW	Principal Threat Waste
QA	Quality assurance
QC	Quality Control
RL	Reporting limit
RMs	Reference materials
RPD	Relative percent difference
RSD	Relative standard deviation
SDG	Sample delivery group
SFS	Portland Harbor Superfund Site
SIM	Select ion monitoring mode
SMA 9D	Sediment Management Area 9D
SOPs	Standard Operating Procedures
TOC	Total Organic Carbon
Total PAH16	16 EPA Priority Pollutant PAH
TPH	Total Petroleum Hydrocarbons

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## INTRODUCTION

Polycyclic aromatic hydrocarbons (PAH) are chemicals of potential concern in sediments found in the Portland Harbor Superfund Site (SFS). Based on several rounds of remedial investigation data developed by the Lower Willamette Group and from available private party studies, it is evident that PAH in the Portland Harbor SFS are ubiquitous, and found both in the surface sediment layer (< 30 cm), as well as deeper horizons of the sediment column. The widespread occurrence of PAH in urban sediments are common, and have been tied to non-point discharges of PAHs from storm water runoff, and from certain kinds of industrial point sources that can release PAH to waterways (Stout et al, 2004). Thus, focused investigations are typically needed to identify the principal and secondary sources of PAH to urban waterways.

The former ExxonMobil Terminal (9420 NW St. Helens Road, Portland OR) is proximal to Sediment Management Area 9D (SMA 9D), which spans the western shoreline of the Willamette River from approximately the Terminal to the margins of the upstream U.S. Moorings property. The former Terminal handled light petroleum fuel products and so, in theory, fugitive petroleum from the facility reaching the river could potentially contribute some low molecular weight (2- and 3-ring) petrogenic PAH to the nearby sediments.

The concentrations of the sum of the 16 EPA Priority Pollutant PAH (Total PAH16) among surface sediments within SMA 9D range from 1,245 µg/Kg to 229,073 µg/Kg. The highest concentrations of PAH in SMA 9D are found in the most upstream reach of the SMA offshore of U.S. Moorings property. The concentrations of PAH generally decline rapidly downstream of U.S. Moorings.

The Portland Harbor Remedial Investigation/Feasibility Study (RI/FS) developed remedial action levels (RALs) for contaminants of concern (LWG, 2015). A range of Total PAH RALs (5,400 to 170,000 µg/Kg) was developed to identify areas of surface sediments where capping and/or dredging could be used as remediation methods to meet cleanup objectives. All six of the different PAH remedial alternatives developed consist of substantial capping and/or dredging within the remediation envelope between approximately River Mile (RM) 5 and 5.8 (LWG, 2015; Figure 3.3-8).

### 1.0 PROJECT DESCRIPTION

In 2014, ExxonMobil collected surface and subsurface sediment samples for the purpose of determining the nature and extent of PAH found in SMA 9D. This sampling effort provided PAH data along the immediate shoreline of the former ExxonMobil Terminal and a limited number of mid-channel locations. Currently, there remains limited data of forensic chemistry quality to fully describe the characteristics of PAH in surface and subsurface sediments of the remedial alternative areas proposed for capping and/or dredging, specifically between the RM 5 and 5.8. The ability to fully understand potential contribution of PAH from proximal properties and upriver sources is limited by this lack of data.

Supplemental sediment sampling is proposed in a study area between RM 5 and 5.8 to improve the spatial and vertical coverage of sediment chemistry in locations proposed for active remediation in the RI/FS. The project objectives for this investigation is twofold:

1. Perform a forensic study intended to characterize the nature of PAH in the remediation envelope between approximately River Mile 5 and 6. The data developed in this investigations can in turn be used to determine:
  - The specific chemistry of PAH found in the sediments within the study area.

- The extent to which, if any, petroleum-source PAH from the former ExxonMobil facility attributed to the PAH found in sediments with the study area.
  - The extent to which natural attenuation of the petroleum-source PAH has occurred.
2. In addition to PAH, characterize sediment for certain principal threat waste (PTW) chemicals that, based on previous investigations, may be found in the proposed study area sediments.

The following sections describe the analyses performed in order to complete the objectives stated above. The table below is an approximation of the total number of field samples collected and the analyses to be performed for each matrix. Figure 1.1 shows the targeted sampling core locations

Matrix	Forensic Chemical Analyses		Ancillary Measurements		PTW Characterization
	TPH and n-alkanes	Parent and Alkyl PAH	Total Organic Carbon	Grain Size	
Sediment Samples	146	146	146	146	27
Field Duplicates	8	8	8	8	2
Rinsate Blanks	5	5			5
Total Field Samples	159	159	154	154	34

## 1.1. Forensic Chemical Analyses

Forensic analytical chemistry will be conducted by NewFields alliance laboratory, Alpha Analytical Laboratory (Mansfield MA), and overseen by NewFields technical staff. Sediment samples collected for the forensic investigation will be analyzed following chemical methods of analysis developed specifically for the forensic analysis of hydrocarbons in environmental media (Douglas et al, 2007). These methods are based on standard EPA SW-846 methods of analysis, and optimized for hydrocarbon measurement and characterization. These are the same methods conducted by the same laboratory as used by the Federal Government and British Petroleum (BP) in the Deepwater Horizon oil spill investigation (NOAA, 2011).

Sediment cores will be collected from 50 locations. The cores will be section as described in the Field Sampling Plan (FSP), and will be analyzed using complimentary methods that will allow for qualitative identification of the type(s) of hydrocarbons comprising the samples by gas chromatography, and the quantitative measurement of an extended list of alkylated PAH by gas chromatography/ mass spectrometry. Together, the resulting data can be used to reconcile PAH patterns and concentrations with likely sources. The analytes of specific interest and brief descriptions of the analytical methods are presented below. Table 1.1 lists the Standard Operating Procedures (SOPs) for the laboratory methods utilized for this project and are maintained on file at NewFields.

### 1.1.1. Total Petroleum Hydrocarbons (TPH), n-Alkanes, and Hydrocarbon Fingerprinting

The sediment samples will be extracted as described in Alpha Analytical SOP 2261, *Shaker Table Extraction*. Approximately 30 grams of sediment (less for highly contaminated samples) will be spiked with surrogate compounds for TPH, PAH and biomarkers (see note below) and serial extracted three times with dichloromethane (DCM) using orbital shaker table techniques. Solvent extracts will be dried over sodium sulfate and concentrated to an appropriate volume. Activated copper will be added to the sample extracts to remove residual sulfur. The extracts will then be purified using an aluminum column

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cleanup procedure. The extracts will be spiked with internal standards (IS) and submitted for instrument analysis.

Rinsate blanks will be extracted as described in Alpha Analytical SOP 2165, *Extraction of Water Samples by Separatory Funnel*. Water samples will be spiked with surrogate compounds and serially extracted three times with DCM. The extracts will be dried over sodium sulfate and concentrated to an appropriate volume. The extracts will then be spiked with internal standards (IS) and submitted for instrument analysis.

The sample extracts will be analyzed using a modified EPA Method 8015D following Alpha Analytical SOP 2246, *Total Petroleum and Saturated Hydrocarbons by Gas Chromatography/Flame Ionization Detector*. The method will be used to determine the total petroleum hydrocarbon (TPH) concentration (C<sub>9</sub>-C<sub>44</sub>), the concentrations of n-alkanes (nC<sub>9</sub> – nC<sub>40</sub>) and selected (C<sub>15</sub>-C<sub>20</sub>) acyclic isoprenoids (e.g., pristane and phytane), and simultaneously provide a detailed fingerprint of the hydrocarbons present in each sample. This analysis allows for characterization of the general boiling range(s) and type(s) of petroleum or other product(s) present, as well as the degree(s) of weathering. Target analytes and detection limits are listed in Table 1.2.

#### **1.1.2. Polycyclic Aromatic Hydrocarbons (PAH)**

Sample extracts will be prepared as stated above and will be analyzed for parent and alkylated PAH using a modified EPA Method 8270D following Alpha Analytical SOP 2247, *Analysis of Parent and Alkylated Polynuclear Aromatic Hydrocarbons, Selected Heterocyclic Compounds, Steranes, Triterpanes and Triaromatic Steroids by GC / MS – SIM*. The GC/MS-SIM method measures concentrations of 72 semivolatile PAH compounds which include the 16 U.S. EPA Priority Pollutant PAHs, alkylated PAHs, and sulfur containing aromatics. Alkyl PAH homologues are quantified using a response factor assigned from the parent PAH compound. The parent and alkylated PAH target analytes, associated response factors and detection limits are listed in Table 1.3.

**Note:** Petroleum biomarkers are not targeted analytes. However, if needed for additional forensic interpretation, the data will be collected.

#### **1.1.3. Ancillary Measurements**

Bulk sediment parameters will be measured as ancillary descriptors of sediment condition.

- Total Organic Carbon (TOC) will be determined by EPA Method 9060A following Alpha SOP 2182, *Total Organic Carbon and Soot in Soil, Sediment*. Results will be reported as percent carbon.
- Grain-size distribution will be determined by ASTM D422-63 following Alpha SOP W-029, *Particle Size of Soils – With/Without Hydrometer and Liquid Limit, Plastic Limit, and Plasticity Index*. Results will be reported as percent gravel, percent sand (coarse, medium, and fine), and percent fines.
- Percent solids will be determined by EPA Method 160.3 following Alpha Analytical SOP 2175, *Percent Solids Determination*. Results will be reported as percent solids.

### **1.2. PTW Characterization**

Principal threat wastes (PTW) are source materials considered to be highly toxic or highly mobile that generally cannot be reliably contained or would present significant risk to human health or the environment should exposure occur (LWG, 2015). The EPA expects to use treatment to address the

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principal threats posed by a site, whenever practical. The PTW chemicals identified in the LWG's FS include polychlorinated biphenyls (PCBs), polychlorinated dioxin and furans (PCDD/Fs), and DDx.

Twenty-seven sediment samples selected from random depth intervals within the remediation envelope area between RM 5 and 5.8 will be analyzed for potential threat waste (PTW) chemicals. The sediment samples will be analyzed for 209 PCB congeners and organochlorine pesticides (DDx) by Alpha Analytical (Mansfield, MA). The sediment samples will also be analyzed for PCDD/Fs by SGS North America (Wilmington, NC). In addition to the PTW characterization, the sediment samples will also be analyzed for the forensic chemical parameters listed above in Section 1.1.

#### **1.2.1. Polychlorinated Biphenyls (PCB congeners)**

The sediment samples will be extracted as described in Alpha Analytical SOP 2172, *Microscale Solvent Extraction (MSE)*. Approximately 30 grams of sediment (less for highly oiled samples) will be spiked with surrogate compounds and serial extracted three times with dichloromethane (DCM) using orbital shaker table techniques. Solvent extracts will be dried over sodium sulfated and exchanged to hexane. The extracts will be purified using silica gel columns, activated copper, and acid. The extracts will be spiked with internal standards (IS) and submitted for instrument analysis.

The sample extracts will be analyzed PCB congeners using a modified EPA Method 8270D following Alpha Analytical SOP 2162, *Determination of PCB Homologs, 136/209 Individual Congeners, and Pesticides Confirmation by GC/MS – SIM*. The low resolution GC/MS-SIM method will be used to measure 209 PCB congeners (including co-elutions). The chemical fingerprint generated by this analysis provides a means to reliably identify and link sources of PCBs in environmental samples. Individual congeners are determined using average response factor (RF) generated from a multi-level calibration. Analytes and target detection limits are listed in Table 1.4.

#### **1.2.2. Organochlorine Pesticides (DDx)**

The sediment samples will be extracted as described in Alpha Analytical SOP 2172, *Microscale Solvent Extraction*. Approximately 30 grams of sediment will be spiked with surrogate compounds and then extracted with dichloromethane (DCM). Solvent extracts will be dried over sodium sulfate and solvent exchanged into hexane. Extracts will be purified using silica gel columns, gel permeation chromatography (GPC), and activated copper. The extracts will be spiked with internal standards (IS) and submitted for instrument analysis.

The sample extracts will be analyzed for organochlorine pesticides using EPA Method 8081B following Alpha Analytical SOP 2158, *Determination of Organochlorine Pesticides by Gas Chromatography/Electron Capture Detection (GC/ECD)*. The DDx concentrations are determined using average response factors (RF) generated from a multi-level calibration curve. Analytes and target detection limits are listed in Table 1.5.

#### **1.2.3. Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans (PCDD/Fs)**

Polychlorinated dibenzo-p-dioxins and furans (PCDD/Fs) will be extracted in hexane using a Soxhlet/Dean Stark extractor. Following extraction, <sup>37</sup>Cl<sub>4</sub>-labeled 2,3,7,8-TCDD is added to each extract to measure the efficiency of the cleanup process. Sample cleanups may include back-extraction with acid and/or base, and gel permeation, alumina, silica gel, Florisil and activated carbon chromatography. High-performance liquid chromatography (HPLC) may be used for further isolation of the 2,3,7,8-isomers or

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other specific isomers or congeners. Extracts are then concentrated, spiked with internal standards (IS) and submitted for instrument analysis.

Sample extracts will be analyzed for PCDD/Fs using EPA method 1613 following SGS SOP 364.6, *Standard Operating Procedure for the Analysis of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans (PCDD/Fs) by high-resolution gas chromatography with high-resolution mass spectrometry (HRGC/HRMS)*. Individual congeners are determined using average response factor (RF) generated from a multi-level calibration. The target analytes and detection limits are listed in Table 1.6.

## **2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES**

This section will discuss the proposed project team and responsibilities for conducting the forensic investigation within the study area.

### **2.1. Project Manager**

Dr. Allen Uhler of NewFields will serve as the Project manager for this investigation. The Project Manager is responsible for all technical aspects of the contract. The Project Manager assures the technical requirements of the contract are met, monitors contract performance, and maintains communication with the ExxonMobil project manager.

#### **Project Manager**

Dr. Allen Uhler  
NewFields – Environmental Forensics Practice  
300 Ledgewood Place, Suite 305  
Rockland, MA 02370  
781-681-5040 x106  
[auhler@newfields.com](mailto:auhler@newfields.com)

### **2.2. Project Coordinator**

Ms. Kerylynn Krahforst of NewFields will serve as the Project Coordinator and will be responsible for all facets of the sampling and analysis program. Her specific responsibilities include the following:

- Coordinate the field and laboratory analyses;
- Ensure that the laboratory capacity is sufficient to undertake the required analyses in a timely manner;
- Ensure adherence to the schedule by tracking sampling, laboratory analysis, validation, and data management tasks; and
- Inform the Project Manager of any decisions that involve changes to the FSP and the analytical QAPP.

#### **Project Coordinator**

Kerylynn Krahforst  
NewFields – Environmental Forensics Practice  
300 Ledgewood Place, Suite 305  
Rockland, MA 02370  
781-681-5040 x123  
[kkrahforst@newfields.com](mailto:kkrahforst@newfields.com)

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## 2.3. Field Coordinator

Dr. Jonathan Nuwer of NewFields will serve as the Field Coordinator, responsible for overall coordination of all field sampling tasks. Specifically, he will be responsible for the following:

- Oversee the planning and coordination for all sampling efforts;
- Coordinate vessel and equipment logistics;
- Oversee all aspects of the sampling to ensure that the appropriate procedures and methods are used; and
- Maintain the field logs.

He will work closely with the Project Coordinator and will immediately notified her if problems occur in the field. If changes to the FSP or analytical QAPP are warranted, he will immediately notify the Project Coordinator.

### Field Coordinator

Dr. Jonathan Nuwer  
NewFields – Sediment Management and Marine Science  
115 2<sup>nd</sup> Ave. N, Suite 100  
Edmonds, WA 98020  
425-967-5285 x104  
[jnuwer@newfields.com](mailto:jnuwer@newfields.com)

## 2.4. Field Crews

Field staff for all sampling events will consist of NewFields employees. The operators of the sampling vessel and equipment, as appropriate, will supply additional staff. Station positioning will be the responsibility of the vessel operator. For all sampling tasks, the field crews will include the following individuals: site safety officer, field task leader, and field crew.

The site safety officer will have the following responsibilities:

- Correct any work practices or conditions that may result in personnel injury or exposure to hazardous materials;
- Determine appropriate personal protection levels and the necessary clothing and equipment, and oversee its proper use;
- Verify that the field crew is aware of the provisions of the HSP and instructed in safe work practices; and
- Verify that the field crew has received the required safety training.

The field task leader will have the following responsibilities:

- Ensure that all activities adhere to the FSP and analytical QAPP;
- Inform the field coordinator of any decisions that involve changes to the FSP and analytical QAPP;
- Mobilize and prepare for field work; and
- Ensure sample custody, including chain-of-custody documentation

Various field staff from NewFields will assist in sample collection, handling, and storage. They may maintain field sampling logs and notebooks and will be responsible for properly labeling sample

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containers. It is the responsibility of the field staff to report any problems or potential changes to the FSP and analytical QAPP to the field task leader.

## **2.5. Database Management**

Ms. Bo Liu of NewFields will have primary responsibility for database management. The project database will allow efficient management of chemical and physical data received from the laboratories. Laboratories conducting analyses for this program will be supplied with specific formats for electronic data deliverables to ensure compatibility with the project database. Qualified personnel will be assigned to conduct QA/QC reviews for each dataset generated. The database will be integrated with a geographical information system (GIS) to allow for presentation of spatial information of the data.

## **2.6. Analytical Laboratories**

The laboratory contracted to perform the Forensic Chemistry analyses is Alpha Analytical (Alpha, Mansfield MA). Alpha will also perform the PCB congener analysis, as well as the organochlorine pesticide analysis. SGE North America (Wilmington NC) will perform the PCDD/F analysis.

The laboratories will assign a project manager responsible for assuring that all analyses performed meet project and measurement quality objectives. It is the Project Manager's responsibility to report any problems to the Project Coordinator. The Project Coordinator will provide technical coordination and oversight to the project managers and laboratory activities. The laboratory Project Managers are:

Susan O'Neil

**Alpha Analytical**

320 Forbes Boulevard

Mansfield, MA 02048

508-844-4117

[soneil@alphalab.com](mailto:soneil@alphalab.com)

Amy Boehm

**SGS North America**

5500 Business Drive

Wilmington, NC 28405

910-350-1903

[Amy.Boehm@sgs.com](mailto:Amy.Boehm@sgs.com)

Copies of SOPs have been submitted to the Project Coordinator for review prior to analysis of any field samples. Precision and reproducibility are key components to a successful analytical program because the data will be critically interpreted using source ratio analysis, principal component analysis, cluster analysis, and possibly other interpretive tools.

### **2.6.1. Laboratory Quality Assurance**

The laboratory's Quality Assurance (QA) Manager (or designee) will be responsible for overseeing the QA activities in the laboratory and ensuring the quality of the data for this project. Specific responsibilities include the following:

- Oversee and implement the laboratory's QA program;
- Maintain QA records for each laboratory facility;

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- Ensure that QA/QC procedures are implemented as required for each method and provide oversight of QA/QC practices and procedures;
  - Review and address or approve non-conformity and corrective action reports; and
  - Coordinate response to any QC issues that affect this project with the Laboratory Project Manager.

### **3.0 SAMPLE HANDLING AND CHAIN OF CUSTODY PROCEDURES**

Chain of custody procedures will be used for all samples throughout the analytical process and for all data and data documentation, whether in hard copy or electronic format. Sampling procedures, including sample collection and documentation, are part of the FSP and as such, are not considered here.

From the time of collection, all samples and sediment cores waiting for processing will be stored on ice or refrigerated at approximately 4°C.

#### **3.1. Sample Containers, Preservation, and Holding Times**

Sample containers, sample size requirements, preservation, and holding times for sediment samples and equipment rinse blanks are summarized in Table 3.1. Sample preservation and field treatment of samples for analyses is described in detail in the FSP.

Based on US EPA guidance the sample holding times prior to analysis and holding times for the extracts presented in Table 3.1 are advisory. These holding times may be extended or preservation guidance changed, as options are assessed.

#### **3.2. Chain of Custody**

All chain of custody records will be made legibly, in indelible ink, and will be signed and dated.

Samples are considered to be in custody if they are:

- in the custodian's actual possession or view;
- in a secured location (under lock) with restricted access; or
- in a container that is secured with an official seal(s) such that the sample cannot be reached without breaking the seal(s).

The principal documents used to identify samples and to document possession are the chain of custody records, field logbooks, and field tracking forms. Chain of custody procedures will be used for all samples at all stages in the analytical or transfer process and for all data and data documentation whether in hard-copy or electronic format.

Samples are kept in the custody of designated sampling and/or field personnel until shipment.

#### **3.3. Sample Shipping**

Sample packing and shipping procedures are detailed in the FSP. Any transfer or movement of samples will use chain of custody procedures. The original signed and dated chain of custody record accompanies the sample(s); a copy is retained by the sample shipper. All shipments will comply with US

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Department of Transportation regulations as specified in 49 Code of Federal Regulations (CFR) 173.6 and 49 CFR 173.24.

### **3.4. Sample Receipt**

Immediately upon receipt of samples, the laboratory sample custodian will review the shipment for consistency with the accompanying chain of custody record and sample condition, before signing and dating the chain of custody record. The custodian will check cooler temperature, and check the original chain of custody records and compare them with the labeled contents of each sample container for correctness and traceability. The sample custodian will sign the chain of custody record and record the date and time received. Sample condition(s) will be noted on the laboratory's sample receipt form and maintained with the chain of custody records. If there are any discrepancies between the chain of custody record and the sample shipment, the sample custodian will contact the sample shipper immediately in an attempt to reconcile these differences. Reconciliation of sample receipt differences will be maintained with the chain of custody records and discussed in the laboratory narrative which accompanies the data report.

**The laboratory will deliver a copy of the chain of custody record and sample receipt form to the Project Coordinator.** Any breaks in the chain of custody or non-conformances will be noted and reported in writing to the Project Coordinator within 24 hours of receipt of samples.

### **3.5. Intra-Laboratory Transfer**

The laboratory custodian or designee will maintain a laboratory sample-tracking record, similar to the chain of custody record that will follow each sample through all of the stages of laboratory processing. The sample-tracking record must contain, at a minimum, the names of individuals responsible for performing the analysis; dates of sample extraction, preparation, and analysis; and the type of analysis being performed.

### **3.6. Inter-Laboratory Transfer**

Transfer of samples from one analytical laboratory to another, e.g., for PCDD/F analysis, will follow chain of custody, sample shipping and receipt procedures described above. Transfer of samples between laboratories will be noted in the laboratory case narrative which accompanies the data report.

### **3.7. Sample Archival**

All remaining samples will be stored frozen at -20°C and sample extracts will be topped with the appropriate solvent (to prevent extracts from evaporating to dryness) and stored frozen at -20°C in glass vials sealed with Teflon lined screw caps and Teflon tape. All samples and extracts will be held by the laboratory in a manner to preserve sample integrity at a secure location with chain of custody procedures for one (1) year after the data package has been validated for that particular set of samples. All archived materials will be accessible for review upon request. At the end of the archival period, the laboratory shall contact the Project Coordinator to obtain directions for handling the remaining samples. The samples will not be disposed of by the laboratory unless provided written approval from the Project Coordinator.

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### **3.8. Data and Data Documentation**

All laboratories will submit electronic data in Excel format and data packages that include all related instrument print-outs (“raw data”) and bench sheets. The laboratories will also provide an electronic data deliverable (EDD) in a flat file format that will be used for database purposes.

The data packages will include the following:

- chain of custody records and sample receipt checklist,
- sample data,
- standard data,
- quality control data,
- case narrative, and
- gas chromatograms and extracted ion profiles.

A copy of the data and data documentation will developed by the laboratory for a given data package will be kept by the laboratory in a secure location using chain of custody procedures for five (5) years after the data packages have been validated. All archived data and documentation will be accessible for review upon request.

## **4.0 LABORATORY OPERATIONS**

All laboratories providing analytical support must have the appropriate facility to store and prepare samples, and appropriate instrumentation and staff to provide analytical data of the required quality within the time period dictated. Laboratories are expected to conduct operations using good laboratory practices including:

- Training and appropriate certified personnel;
- A program of scheduled maintenance of analytical balances, laboratory equipment, and instrumentation;
- Routine checking of analytical balances using a set of standard reference weights (ASTM class, NIST Class S-1, or equivalent);
- Recording all analytical data in secure electronic system with date and associated analyst identification, and/or logbooks with each entry signed and dated by analyst; and
- Monitoring and documenting the temperatures of cold storage areas and freezer units

Personnel in any laboratory performing analyses should be well versed in good laboratory practices, including standard safety protocols. It is the responsibility of the laboratory manager and/or supervisor to ensure that safety training is mandatory for all laboratory personnel. The laboratory is responsible for maintaining a current safety manual in compliance with Occupational Safety and Health Administration (OSHA) or equivalent state or local regulations. Proper procedures for safe storage, handling and disposal of chemicals should be followed at all time; each chemical should be treated as a potential health hazard and/or physical hazard, and good laboratory practices should be implemented accordingly.

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#### **4.1. Quality Assurance Documentation**

All laboratories must have the following documents and information must be current and available to all laboratory personnel:

- Laboratory Quality Assurance Management Plan; and
- Laboratory Standard Operating Procedures (SOPs), providing detailed instructions for performing routine laboratory procedures.

Each laboratory's Quality Assurance staff will monitor the laboratory's component of the project according to their SOPs to ensure the accuracy, integrity, and completeness of the data. Analytical projects staff members will be responsible for ensuring that sample tracking, sample preparation, and the analytical instrument operation all meet the quality control criteria detailed within the applicable SOPs and this QAPP. The SOPs associated with this QAPP or the analytical methods are maintained on file at NewFields.

### **5.0 ASSESSMENT OF DATA QUALITY**

The purpose of this QAPP is to develop and document analytical data of known, acceptable, and defensible quality. The quality of the data is presented as a set of statements that describe in precise quantitative terms the level of uncertainty that can be associated with the data without compromising their intended use. These statements are referred to as Measurement Quality Objectives (MQOs) and are usually expressed in terms of precision, accuracy (or bias), representativeness, completeness, and comparability (PARCC) parameters (U.S. EPA, 2002). PARCC parameters are commonly used to assess the quality of the environmental data.

#### **5.1. Precision**

Precision is the degree of mutual agreement among individual measurements of the same property under prescribed similar conditions, such as replicate measurements of the same sample. Precision is concerned with the "closeness" of the results. Where suitable reference materials (RMs) are available, precision will be expressed as the relative standard deviation (RSD) for the repeated measurements. This use of RMS allows for long-term measurement of precision but does not include homogenization as a source of analytical variability.

In addition to tracking the precision of replicate RM analyses, precision will be expressed as the relative percent difference (RPD) between a pair of replicate data from environmental samples prepared and analyzed in duplicate.

#### **5.2. Accuracy (or Bias)**

Accuracy is the degree of agreement of a measurement with an accepted reference value and may be expressed as the difference between the two measured values or as a percentage of the reference value.

The primary evaluation of bias will be through the use of reference materials (RMs). RMs with certified values (from NIST or a similar source) will be used if they are available. Spiked matrix samples will also be analyzed to assess accuracy for those analytes that are not available in suitable reference material.

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### 5.3. Representativeness

Representativeness is the degree to which data represent a characteristic of an environmental condition. In the field, representativeness will be addressed primarily in the sampling design by the selection of sampling sites and sample collection procedures. In the laboratories, representativeness will be ensured by the proper handling and storage of samples and initiation of analysis within holding times.

### 5.4. Completeness

Completeness is a measure of the proportion of data specified in the sampling plan which is determined to be valid. Completeness will be assessed by comparing the number of valid sample results to the number of potential results planned to be generated. The DQO for completeness is 95%, i.e. no more than 5% of the analytical data missing or qualified as unreliable (rejected).

### 5.5. Comparability

Comparability expresses the confidence with which one data set can be evaluated in relationship to another data set. Comparability of the chemical analytical data is established through the use of:

- Program-defined general analytical methodology (e.g., low resolution MS), detection limits, precision and accuracy requirements and reporting formats;
- NIST-traceable calibration materials;
- Reference material with each sample batch; and
- Analysis of a common “reference oil”.

## 6.0 QUALITY CONTROL PROCEDURES

The analytical methods for this project are described in sections 1.1 through 1.3. The QA/QC requirements for these methods will provide a common foundation for each laboratory’s protocols. This “common foundation” includes:

1. The specification of analytes to be identified and quantified and the minimum sensitivity of the analytical methods;
2. The use of NIST reference materials; and
3. The use of a reference oil. A North Slope Crude Reference oil will be analyzed for PAH, petroleum biomarkers, and total petroleum hydrocarbons. A coal tar reference material will be analyzed for the high molecular weight PAH. Reference oils are not required for the COPC characterization.

Prior to the analysis of samples, each laboratory must provide written protocols for the analytical methods used; calculate detection limits for each analyte in each matrix of interest, and establish an initial calibration curve in the appropriate concentration range for each analyte. The laboratory must demonstrate its continued proficiency by repeated analyses of reference materials, calibration checks, and laboratory method blanks. Laboratories will be expected to take corrective actions promptly if measurement quality objectives described in this plan are not met.

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## 6.1. Standard Operating Procedures for Analytical Methods

Prior to the analysis of field samples, each laboratory is required to submit to the QA Coordinator for review and approval, written Standard Operating Procedures (SOPs) detailing the procedures used in sample receipt and handling, sample preparation and analysis, data reduction and reporting.

## 6.2. Determination of Method Detection Limit and Reporting Limits

The method detection limit (MDL) is defined as the minimum concentration that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The analytical laboratory will establish and report an MDL for each analyte of interest in each matrix, with the exception of oil for which MDLs cannot be accurately determined. The actual MDLs will be established by following the method outlined in the U.S. Federal Register 40CFR Part 136 (US EPA, 1997). The reporting limit (RL) will be based on the low calibration standard analyzed as part of the initial calibration. Results that are less than 5X the MDL or less than the lowest calibration standard will not be required to meet the measurement quality objectives (MQOs) for precision and accuracy (bias), because these results may be outside the “quantitation range”. Thus these results may be flagged by the laboratory with a “J”, to indicate the results are possibly an estimate and have not been required to meet the MQOs. If the analyte is not detected in a sample, the result will be reported as non-detected at the MDL and flagged with a “U”.

Target detection limits for the forensic chemistry analyses are shown in Table 1.2 – Table 1.6. The alkyl homologues are assigned the MDL and reporting limit (RL) from the parent PAH. Reporting limits for the supporting analyses (percent solid and TOC) will be 0.01%. Reporting limit for grain size will be 0.1% or lower. The reporting limit will be demonstrated by the laboratory to be greater than 5X the detection limit.

Target detection limits may not be met due to required dilutions, interferences, and/or limited sample size. If a laboratory MDL does not meet the target detection limit, the reason for the elevated detection limit should be discussed in the laboratory case narrative.

At the discretion of the analytical laboratory, detected analytes at concentrations less than the MDL may be reported, provided that the compound meets the established identification criteria and the peak height is greater than or equal to three times the background noise level ( $S/N > 3X$ ). These results will be “J” flagged by the laboratory.

## 6.3. Quality Control Criteria

Quality control is an integral part of the laboratory activities. It demonstrates the quality operations and analyses, provides analysts with metrics about method performance and aids project managers in identifying and correcting systematic and random problems that can occur in the field and laboratory operations.

The MQOs and the required frequency of analysis for each QC parameter are presented in Table 6-1 through Table 6.6. Where discrepancies exist between this QAPP and laboratory SOPs and QA Manuals, this QAPP will take precedence.

QC procedures will be completed by the laboratories, as required in each method protocol and as indicated in this QAPP. The frequency of analysis for laboratory control samples, matrix spike samples,

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matrix spike duplicates or laboratory duplicates, and method blanks will be one for every 20 samples or one per extraction batch, whichever is more frequent. Surrogate spikes and internal standards will be added to every field sample and QC sample, as required by the method. Calibration procedures will be completed at the frequency specified in each method description. As required for EPA SW-846 methods (EPA 2004), performance-based control limits have been established by each laboratory.

The analytical laboratory will determine when the MQOs have not been met, and perform appropriate corrective actions before continuing the analyses or reporting of the data. If the “Corrective Action” in the Method Performance Criteria table states “Resolve before proceeding”, the laboratory must perform an adjustment to the analytical process and subsequently demonstrate the criteria will be met before proceeding with analysis for project samples. In addition, if the results associated with a non-compliant QC element have been obtained, the laboratory must repeat those analyses until acceptable QC results are obtained. If the laboratory determines the non-compliance does not affect the quality of the data, the laboratory will discuss the non-compliance and rationale used to conclude the data are not affected in the case narrative which accompanies the data report. If the laboratory determines the non-compliance is due to interferences or circumstances outside the laboratory’s control, the laboratory will discuss the reason for the non-compliance in the case narrative and the results reported.

#### **6.3.1. Initial Calibration**

Acceptable calibration (initial and continuing) must be established and documented prior to sample analysis. NIST-traceable calibration materials must be used where available in establishing calibration. Initial calibration will be established according to the criteria in Table 6.1 through Table 6.6. A specific requirement for this project is to use methodology (and tune instrumentation) for low detection limits, therefore, samples with analytes above the calibration range will be diluted and reanalyzed. If samples require a dilution, results from the initial analytical run that were within the calibration range should be reported. Results from the diluted analyses should be reported only for those analytes that exceeded the calibration range.

#### **6.3.2. Initial Calibration Verification**

As part of the initial calibration procedure, an initial calibration verification (ICV) standard is analyzed immediately following the initial calibration. The ICV consists of a second source material. The ICV must meet the specified criteria prior to sample analysis. A second source material is not available for the high molecular weight PAHs, therefore an ICV will not be analyzed.

#### **6.3.3. Continuing Calibration Verification**

Continuing calibration verification (CCV) standards will be run at the beginning (opening) and end (closing) of each analytical sequence, and at the frequencies indicated in Table 6.1 through Table 6.4. If the CCV results do not meet the specified criteria, injection maintenance should be performed and another CCV reanalyzed along with the affected samples. If the CCV still does not meet the specified criteria, then the instrument must be recalibrated and all samples analyzed since the last acceptable CCV must be reanalyzed.

#### **6.3.4. Reference Materials**

Reference materials of a matrix appropriate to the samples being analyzed will be analyzed every 20 samples throughout the analytical program, if available. The data resulting from the analysis of these samples will be reported in the same manner as that from the field samples. These data will be the

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prime materials used to determine and document accuracy and precision of the associated field sample data. The reference materials to be used are listed in the criteria tables.

Accuracy is computed by comparing the laboratory's value for each analyte against either end of the range of values reported by the certifying agency. The laboratory's value must be within 30% of either the upper or lower end of the NIST's 95% uncertainty range for SRM 1941b, except the low end of fluorene for SRM 1941b is extended to 40%.

A North Slope Crude reference oil will be analyzed for n-alkanes and TPH and parent and alkylated PAH. The results from these analyses will be compared to an established laboratory mean.

#### **6.3.5. Method Blanks**

Method (procedural) blanks are laboratory derived samples which have been subjected to the same preparation or extraction procedures and analytical protocols as project samples. A method blank will be prepared and analyzed with every 20 field samples analyzed. Acceptance criteria are provided in Table 6.1 through Table 6.6. Failure to meet acceptance criteria requires definitive corrective action to identify and eliminate the source (s) of the contamination before the subsequent reanalysis and re-extraction of the blank and affected samples. Sample results will not be blank corrected.

#### **6.3.6. Rinsate Blanks**

Introduction of chemical contaminants during sampling and analytical activities will be assessed by the analysis of blanks. Rinsate blanks, consisting of sampling equipment rinsates, will be generated for all chemical parameter groups for approximately 5% of the sediment samples collected and submitted to the laboratory for sample analysis.

#### **6.3.7. Laboratory Control Sample/Laboratory Control Sample Duplicates**

Laboratory control sample/laboratory control sample duplicates (LCS/LCSDs) will be prepared and analyzed with every 20 field samples. Samples will be spiked prior to extraction and will be processed identically to the field samples to assess analyte recoveries. Acceptance criteria are provided in Table 6.1 through Table 6.4.

#### **6.3.8. Sample Duplicate**

A duplicate sample (DUP) aliquot from a representative matrix will be prepared and analyzed with every 20 field samples, except for water samples. Water samples will not be analyzed in duplicate because of the difficulty in sub-sampling representative aliquots. Acceptance criteria are provided in Table 6.1 through Table 6.4.

#### **6.3.9. Matrix Spike/Matrix Spike Duplicate**

Matrix spike/matrix spike duplicates (MS/MSDs) will be analyzed every 20 field samples, except for water samples (i.e., rinsate blanks). MS/MSDs will not be analyzed with water samples and inert sorbent materials sample batches because of the difficulty in sub-sampling representative aliquots. Spike solution concentrations for the MS/MSD must be appropriate to the matrix and anticipated range of contaminants in the sample; that is 5 to 10 times analyte concentration. However, because it not possible to know the concentration of contaminants prior to analysis, professional judgment should be exercised in choosing concentrations that are reasonable under the circumstances. The MS/MSDs acceptance criteria are provided in Table 6.1 through Table 6.6.

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### **6.3.10. Surrogates**

All field and QC samples will be spiked with surrogates prior to extraction, as required by the analytical methods. Control criteria for the surrogate recovery are listed in Table 6.1 through Table 6.6. Sample data will not be corrected for surrogate recovery, with the exception of the reference material (i.e., SRM 1941b). The certified values of SRM 1941b were determined based on surrogate corrected data.

### **6.3.11. Internal Standards**

All sample extracts will be spiked with internal standards prior to analysis, when required by the analytical method. Control criteria for internal standard recovery are listed in Table 6.1 through Table 6.6.

## **7.0 DATA REDUCTION, VALIDATION AND REPORTING**

### **7.1. Data Reduction**

Data reduction is the process whereby raw data (analytical measurements) are converted or reduced into meaningful results (analyte concentrations). This process may be either manual or electronic. Primary data reduction requires accounting for specific sample preparations, sample volume (or weight) analyzed, and any concentrations or dilutions required.

Primary data reduction is the responsibility of the analyst conducting the analytical measurement and is subject to further review by laboratory staff, the Laboratory Project Manager, and finally, independent reviewers. All data reduction procedures will be described in the laboratory SOPs. Any deviations from the laboratory SOPs will be discussed in the laboratory case narratives.

The following data reduction processes should be considered:

- Data generated from the analysis of blank samples will not be utilized for correction of analyte data;
- Surrogate compounds, matrix spikes and spike blanks will be evaluated as %R;
- Reference materials will be reported in units as that from the field samples;
- Continuing calibration factors will be presented as %D; and
- Duplicate sample results will be expressed as RPD.

### **7.2. Data Review and Data Validation**

Data review is an internal review process where data are reviewed and evaluated by personnel within the laboratory. Data validation is an independent review process conducted by personnel not associated with data collection and generation activities.

Data review is initiated at the bench level by the analyst, who is responsible for ensuring that the analytical data are correct and complete, the appropriate SOPs have been followed, and the QC results are within the acceptable limits. The Laboratory Project Manager has final review authority. It is the Laboratory Project Manager's responsibility to ensure that all analyses performed by that laboratory are correct, complete, and meet project data quality objectives.

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External and independent data validation may be performed for all samples using a full data package containing sufficient information to allow the independent validation of the sample identity and integrity, the laboratory measurement system, and resulting quantitative and qualitative data. The required information with associated print-outs is listed in Table 7.1.

Full validation will consist of a review of the entire data package for compliance with documentation and quality control criteria for all of the following items, plus recalculations of instrument calibration curves, sample and QC results:

- Package completeness
- Holding times from extraction to analysis
- Instrument calibration, initial and continuing
- Blank results
- Instrument performance
- Spike recoveries
- Standard reference materials results
- Laboratory duplicate results
- Reported detection limits
- Compound quantitation
- Compound identification
- Verification of electronic data deliverable (EDD) against hardcopy (10% verification)

## **8.0 CORRECTIVE ACTION/PROCEDURE ALTERATION**

When the data from the analyses of any quality control sample exceeds the project specified control limits or indicates that the analytical method is drifting out of control, it is the immediate responsibility of the analyst to identify and correct the situation before continuing with sample analysis.

A narrative describing the problem noted, the steps taken to identify and correct the problem and the treatment of the relevant sample batches must be prepared and submitted with the relevant data package. If the action indicates a revision to a current SOP is warranted, the laboratory will revise the SOP and submit the SOP to the Project Coordinator within 30 working days after the problem was noted. Until a revised SOP is approved, any data sets reported with the revised method will have the changes to the method noted in the laboratory's case narrative.

## **9.0 REFERENCES**

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National Oceanic and Atmospheric Administration. (2011). Analytical Quality Assurance Plan. Mississippi Canyon 252 (Deepwater Horizon) Natural Resource Damage Assessment.

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U.S. EPA, (2002). Guidance for Quality Assurance Project Plans EPA QA/G-5. EPA/240/R-02/009. US Environmental Protection Agency, Office of Environmental Information, Washington, DC.

**Table 1.1. Standard Operating Procedures**

<b>SOP Number</b>	<b>SOP Title</b>	<b>Procedure</b>
2165.13	Extraction of Water Samples by Separatory Funnel	Extraction
2172.11	Microscale Solvent Extraction (MSE)	Extraction
2261.5	Shaker Table Extraction	Extraction
2263.5	Gravimetric Determination	Screening
2167.3	Gel Permeation Chromatography	Cleanup
2168.4	Sulfur Cleanup	Cleanup
2260.4	Alumina Column Cleanup of Organic Extracts	Cleanup
2267.2	Silica Gel Cleanup Preparation (Manual)	Cleanup
2158.1	Determination of Organochlorine Pesticides By Gas Chromatography/Electron Capture Detection (GC/ECD)	Analysis
2162.12	Determination of PCB Homologs, 136/209 Individual Congeners, and Pesticide Confirmation by GC/MS-SIM	Analysis
2175.4	Percent Solids Determination	Analysis
2182.19	Total Organic Carbon and Soot in Soil, Sediment	Analysis
2246.7	Total Petroleum and Saturated Hydrocarbons by Gas Chromatography/Flame Ionization Detector	Analysis
2247.8	Analysis of Parent and Alkylated Polynuclear Aromatic Hydrocarbons , Selected Heterocyclic Compounds, Steranes, Triterpanes, and Triaromatic Steroids by GC/MS-SIM	Analysis
364.6	Standard Operating Procedure for the Analysis of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans (PCDD/Fs)	Extraction/Analysis
W-029.1	Particle Size Analysis of Soils - With/Without Hydrometer and Liquid Limit, Plastic Limit, and Plasticity Index	Analysis

**Table 1.2. n-Alkanes and Isoprenoid Target Analytes**

Abbr.	Compound
C9	n-Nonane
C10	n-Decane
C11	n-Undecane
C12	n-Dodecane
C13	n-Tridecane
1380	2,6,10 Trimethyldodecane
C14	n-Tetradecane
1470	2,6,10 Trimethyltridecane
C15	n-Pentadecane
C16	n-Hexadecane
nPr	Norpristane
C17	n-Heptadecane
Pr	Pristane
C18	n-Octadecane
Ph	Phytane
C19	n-Nonadecane
C20	n-Eicosane
C21	n-Heneicosane
C22	n-Docosane
C23	n-Tricosane
C24	n-Tetracosane
C25	n-Pentacosane
C26	n-Hexacosane
C27	n-Heptacosane
C28	n-Octacosane
C29	n-Nonacosane
C30	n-Triacontane
C31	n-Hentriacontane
C32	n-Dotriacontane
C33	n-Tritriacontane
C34	n-Tetratriacontane
C35	n-Pentatriacontane
C36	n-Hexatriacontane
C37	n-Heptatriacontane
C38	n-Octatriacontane
C39	n-Nonatriacontane
C40	n-Tetracontane
TSH	Total Saturated Hydrocarbons ( $\Sigma$ C9 –C40)
TPH	Total Petroleum Hydrocarbons (C9-C44)

**Target Method Detection Limit**

Sediments/Soils: 0.1 – 0.5 µg/Kg, dry weight  
Waters: 1.0 – 5.0 ng/L

**Target Reporting Limit**

Sediments/Soils: 2 µg/Kg, dry weight  
Waters: 10 ng/L  
Oil: 2 mg/Kg

**Table 1.3. Polycyclic Aromatic and Related Heterocyclic Target Analytes<sup>a</sup>**

Abbr.	Compound	RF Source <sup>b</sup>
D0	cis/trans-Decalin	
D1	C1-Decalins	tD0 <sup>c</sup>
D2	C2-Decalins	tD0
D3	C3-Decalins	tD0
D4	C4-Decalins	tD0
BT0	Benzothiophene	
BT1	C1-Benzo(b)thiophenes	BT0
BT2	C2-Benzo(b)thiophenes	BT0
BT3	C3-Benzo(b)thiophenes	BT0
BT4	C4-Benzo(b)thiophenes	BT0
<b>N0</b>	<b>Naphthalene</b>	
N1	C1-Naphthalenes	N0
N2	C2-Naphthalenes	N0
N3	C3-Naphthalenes	N0
N4	C4-Naphthalenes	N0
B	Biphenyl	
DF	Dibenzofuran	
<b>AY</b>	<b>Acenaphthylene</b>	
<b>AE</b>	<b>Acenaphthene</b>	
<b>F0</b>	<b>Fluorene</b>	
F1	C1-Fluorenes	F0
F2	C2-Fluorenes	F0
F3	C3-Fluorenes	F0
<b>A0</b>	<b>Anthracene</b>	
<b>P0</b>	<b>Phenanthrene</b>	
PA1	C1-Phenanthrenes/Anthracenes	P0
PA2	C2-Phenanthrenes/Anthracenes	P0
PA3	C3-Phenanthrenes/Anthracenes	P0
PA4	C4-Phenanthrenes/Anthracenes	P0
RET	Retene	
DBT0	Dibenzothiophene	
DBT1	C1-Dibenzothiophenes	DBT0
DBT2	C2-Dibenzothiophenes	DBT0
DBT3	C3-Dibenzothiophenes	DBT0
DBT4	C4-Dibenzothiophenes	DBT0
BF	Benzo(b)fluorene	

Abbr.	Compound	RF Source <sup>b</sup>
<b>FLO</b>	<b>Fluoranthene</b>	
<b>PY0</b>	<b>Pyrene</b>	
FP1	C1-Fluoranthenes/Pyrenes	PY0
FP2	C2-Fluoranthenes/Pyrenes	PY0
FP3	C3-Fluoranthenes/Pyrenes	PY0
FP4	C4-Fluoranthenes/Pyrenes	PY0
NBT0	Naphthobenzothiophenes	
NBT1	C1-Naphthobenzothiophenes	NBT0
NBT2	C2-Naphthobenzothiophenes	NBT0
NBT3	C3-Naphthobenzothiophenes	NBT0
NBT4	C4-Naphthobenzothiophenes	NBT0
<b>BA0</b>	<b>Benzo[a]anthracene</b>	
<b>C0</b>	<b>Chrysene/Triphenylene</b>	
BC1	C1-Chrysenes	C0
BC2	C2-Chrysenes	C0
BC3	C3-Chrysenes	C0
BC4	C4-Chrysenes	C0
<b>BBF</b>	<b>Benzo[b]fluoranthene</b>	
<b>BJKF</b>	<b>Benzo[j]+[k]fluoranthene</b>	BKF <sup>d</sup>
BAF	Benzo[a]fluoranthene	BKF or BAF
<b>BEP</b>	<b>Benzo[e]pyrene</b>	
<b>BAP</b>	<b>Benzo[a]pyrene</b>	
PER	Perylene	
<b>IND</b>	<b>Indeno[1,2,3-cd]pyrene</b>	
<b>DA</b>	<b>Dibenz[a,h]anthracene</b>	
<b>GHI</b>	<b>Benzo[g,h,i]perylene</b>	
CAR	Carbazole	
2MN	2-Methylnaphthalene	
1MN	1-Methylnaphthalene	
26DMN	2,6-Dimethylnaphthalene	
167TMN	1,6,7-Trimethylnaphthalene	
4MDT	4-Methyldibenzothiophene	DBT0
2MDT	2/3-Methyldibenzothiophene	DBT0
1MDT	1-Methyldibenzothiophene	DBT0
3MP	3-Methylphenanthrene	P0
2MP	2/4-Methylphenanthrene	P0
2MA	2-Methylantracene	P0
9MP	9-Methylphenanthrene	P0
1MP	1-Methylphenanthrene	P0

<sup>a</sup>US EPA Priority Pollutant PAH compounds listed in bold.

<sup>b</sup>Response factor (RF to be used for quantitation of alkyl PAH compounds and alkyl sulfur heterocyclics. If blank, compound is included in the calibration mix.)

<sup>c</sup>tD0 = transD0 (used if cis/trans in separate standards.)

<sup>d</sup>BKF = Benzo(k)fluoranthene. Benzo(j)fluoranthene and benzo(k)fluoranthene co-elute and will be reported as Benzo[j]+[k]fluoranthene (BJKF).

#### **Target Method Detection Limit**

Sediments/Soils: 0.1 – 0.5 µg/Kg, dry weight  
Waters: 1.0 – 5.0 ng/L

#### **Target Reporting Limit**

Sediments/Soils: 2 µg/Kg, dry weight  
Waters: 10 ng/L  
Oil: 2 mg/Kg

**Table 1.4. Polychlorinated Biphenyl Target Analytes<sup>a</sup>**

<b>PCB Congeners (Ballschmiter and Zell numbers)</b>					
CL1-BZ#1	CL4-BZ#69	CL4-BZ#66	CL6-BZ#140	CL6-BZ#162	Monochlorobiphenyls
CL1-BZ#2	CL4-BZ#43	CL4-BZ#80	CL5-BZ#108	CL7-BZ#174	Dichlorobiphenyls
CL1-BZ#3	CL3-BZ#36	CL4-BZ#55	CL5-BZ#107/#123	CL6-BZ#128	Trichlorobiphenyls
CL2-BZ#4/#10	CL4-BZ#52	CL5-BZ#92	CL7-BZ#188	CL8-BZ#202	Tetrachlorobiphenyls
CL2-BZ#9	CL4-BZ#48	CL5-BZ#89/#84	CL6-BZ#134	<b>CL6-BZ#167</b>	Pentachlorobiphenyls
CL2-BZ#7	CL4-BZ#49	CL5-BZ#101/#90	CL5-BZ#106	CL7-BZ#181	Hexachlorobiphenyls
CL2-BZ#6	CL5-BZ#104	CL4-BZ#56	CL6-BZ#133	CL7-BZ#177	Heptachlorobiphenyls
CL2-BZ#5	CL4-BZ#47	CL5-BZ#113	CL6-BZ#142	CL8-BZ#204/#200	Octachlorobiphenyls
CL2-BZ#8	CL4-BZ#65/#75/#62	CL5-BZ#99	<b>CL5-BZ#118</b>	CL7-BZ#171	Nonachlorobiphenyls
CL3-BZ#19	CL3-BZ#39	CL6-BZ#150	CL6-BZ#131	CL7-BZ#173	Decachlorobiphenyl
CL2-BZ#14	CL3-BZ#38	CL4-BZ#60	CL7-BZ#184	CL8-BZ#197	Total PCB
CL3-BZ#30	CL4-BZ#44	CL6-BZ#152	CL6-BZ#165	CL7-BZ#172	
CL3-BZ#18	CL4-BZ#59	CL5-BZ#119	CL6-BZ#146	CL7-BZ#192	
CL2-BZ#11	CL4-BZ#42	CL5-BZ#83/#125/#112	CL6-BZ#161	<b>CL6-BZ#156</b>	
CL3-BZ#17	CL4-BZ#71	CL5-BZ#86/#109	CL5-BZ#122	<b>CL6-BZ#157</b>	
CL2-BZ#12	CL3-BZ#35	CL6-BZ#145	CL6-BZ#168	<b>CL7-BZ#180</b>	
CL3-BZ#27	CL4-BZ#41	CL5-BZ#97	<b>CL5-BZ#114</b>	CL7-BZ#193	
CL2-BZ#13	CL4-BZ#72	CL6-BZ#148	CL6-BZ#153	CL8-BZ#199	
CL3-BZ#24	CL5-BZ#96	CL4-BZ#79	CL6-BZ#132	CL7-BZ#191	
CL3-BZ#16	CL5-BZ#103	CL5-BZ#116	CL7-BZ#179	CL8-BZ#198	
CL3-BZ#32	CL4-BZ#68/#64	CL6-BZ#154	CL6-BZ#141	CL8-BZ#201	
CL2-BZ#15	CL4-BZ#40	CL4-BZ#78	CL7-BZ#176	<b>CL7-BZ#170</b>	
CL3-BZ#34	CL3-BZ#37	CL5-BZ#87/#111	<b>CL5-BZ#105</b>	CL7-BZ#190	
CL3-BZ#23	CL5-BZ#100	CL6-BZ#136	CL6-BZ#137	CL8-BZ#196	
CL4-BZ#54	CL5-BZ#94	CL5-BZ#117	CL5-BZ#127	CL8-BZ#203	
CL3-BZ#29	CL4-BZ#57	CL5-BZ#115	CL7-BZ#186	<b>CL6-BZ#169</b>	
CL4-BZ#50	CL4-BZ#67/#58	CL5-BZ#85	CL6-BZ#130/#164	CL9-BZ#208	
CL3-BZ#26	CL5-BZ#102	CL5-BZ#120	CL7-BZ#178	CL9-BZ#207	
CL3-BZ#25	CL4-BZ#61	CL5-BZ#110	CL6-BZ#138	<b>CL7-BZ#189</b>	
CL4-BZ#53	CL5-BZ#98	<b>CL4-BZ#81</b>	CL6-BZ#163/#160	CL8-BZ#195	
CL3-BZ#31	CL4-BZ#76	CL6-BZ#151	CL6-BZ#129/#158	CL8-BZ#194	
CL3-BZ#28	CL5-BZ#93	CL6-BZ#135	CL7-BZ#182/#175	CL8-BZ#205	
CL3-BZ#33	CL4-BZ#63	CL5-BZ#82	CL7-BZ#187	CL9-BZ#206	
CL4-BZ#51	CL5-BZ#121/#95/#88	CL6-BZ#144	CL7-BZ#183	CL10-BZ#209	
CL3-BZ#21/#20	CL4-BZ#74	CL6-BZ#147/#149	CL6-BZ#166		
CL4-BZ#45	CL6-BZ#155	<b>CL4-BZ#77</b>	CL6-BZ#159		
CL3-BZ#22	CL4-BZ#70	CL6-BZ#143/#139	<b>CL5-BZ#126</b>		
CL4-BZ#73/#46	CL5-BZ#91	CL5-BZ#124	CL7-BZ#185		

<sup>a</sup>World Health Organization (WHO) PCB congeners listed in bold.

**Target Method Detection Limit**

Sediments/Soils: 0.2 – 0.4 µg/Kg, dry weight  
Waters: 0.25 – 0.75 ng/L

**Target Reporting Limit**

Sediments/Soils: 0.4 – 0.8 µg/Kg, dry weight  
Waters: 0.5 – 1.5 ng/L

Note: The RL information provided is calculated using a 100% Solids factor.

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**Table 1.5. Organochlorine Pesticide Target Analytes**

Compound	Sediment		Water	
	RL (µg/Kg)	MDL (µg/Kg)	RL (ng/L)	MDL (ng/L)
4,4'-DDD	0.4	0.4	0.5	0.5
4,4'-DDE	0.4	0.4	0.5	0.5
4,4'-DDT	0.4	0.4	0.5	0.5
2,4'-DDE	0.4	0.4	0.5	0.5
2,4'-DDD	0.4	0.4	0.5	0.5
2,4'-DDT	0.4	0.4	0.5	0.5

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**Table 1.6. Polychlorinated Dioxin and Furan Target Analytes**

Compound	Sediment		Water	
	RL (pg/g)	MDL (pg/g)	RL (pg/L)	MDL (pg/L)
2378-TCDD	0.5	0.07	5	1.16
12378-PeCDD	2.5	0.21	25	3.66
123478-HxCDD	2.5	0.26	25	4.15
123678-HxCDD	2.5	0.29	25	4.10
123789-HxCDD	2.5	0.25	25	4.10
1234678-HpCDD	2.5	0.28	25	5.14
OCDD	5	0.4	50	10.2
2378-TCDF	0.5	0.08	5	1.19
12378-PeCDF	2.5	0.18	25	4.12
23478-PeCDF	2.5	0.24	25	4.38
123478-HxCDF	2.5	0.22	25	3.79
123678-HxCDF	2.5	0.21	25	3.72
234678-HxCDF	2.5	0.25	25	4.32
123789-HxCDF	2.5	0.24	25	4.36
1234678-HpCDF	2.5	0.33	25	4.53
1234789-HpCDF	2.5	0.30	25	4.18
OCDF	5	0.6	50	9.0

**Table 3.1. Sample Containers, Preservation, and Holding Times.**

Analysis Group	Analysis	Method	Sample Size	Preservation	Holding Times	Containers	Laboratory/Contact
<b>Sediment</b>							
Forensic Chemical Analysis	PAHs and alkyl PAHs	EPA 8270 Modified	30 g	4 ± 2°C; store in the dark	14 days to extraction	8 ounce (oz) wide-mouth glass jar	Alpha Susan O'Neil <a href="mailto:soneil@alphalab.com">soneil@alphalab.com</a> 508 844-4117
				< -10°C; store in the dark	1 year to extraction		
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
	TPH and n-alkanes	EPA 8015 Modified	30 g	4 ± 2°C; store in the dark	14 days to extraction		
				< -10°C; store in the dark	1 year to extraction		
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
Ancillary Measurements	TOC	EPA 9060 Modified	20 g	4 ± 2°C; store in dark	28 days	8 oz plastic jar	
	Grain Size	ASTM D422	100 g	Ambient	None established		
PTW Characterization	Organochlorine Pesticides	EPA 8081A	30 g	4 ± 2°C; store in the dark	14 days to extraction	8 oz wide-mouth glass jar	Alpha Susan O'Neil <a href="mailto:soneil@alphalab.com">soneil@alphalab.com</a> 508 844-4117
				< -10°C; store in the dark	1 year to extraction		
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
	PCB Congeners	EPA 8270 Modified	30 g	4 ± 2°C; store in the dark	14 days to extraction		
				< -10°C; store in the dark	1 year to extraction		
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
	Dioxin/Furan Congeners	EPA 1613B	10g	0-4°C; store in the dark	45 days to extraction	4 oz amber wide-mouth glass jar	SGS <a href="mailto:amy.boehm@sgs.com">amy.boehm@sgs.com</a> 910 350-1903
				< -10°C; store in the dark	1 year to extraction		
Archive				< -20°C; store in the dark	1 year to extraction	8 oz wide-mouth glass jar	Alpha Susan O'Neil <a href="mailto:soneil@alphalab.com">soneil@alphalab.com</a> 508 844-4117
<b>Water (Rinsate Blanks)</b>							
Forensic Chemical Analysis	PAHs and alkyl PAHs	EPA 8270 Modified	1 L	4 ± 2°C; store in the dark	14 days to extraction	2 1-liter (L) amber bottle	Alpha Susan O'Neil <a href="mailto:soneil@alphalab.com">soneil@alphalab.com</a> 508 844-4117
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
	TPH and n-alkanes	EPA 8015 Modified	1 L	4 ± 2°C; store in the dark	14 days to extraction		
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
PTW Characterization	Organochlorine Pesticides	EPA 8081A	1 L	4 ± 2°C; store in the dark	14 days to extraction	1 1-L amber bottle	Alpha Susan O'Neil <a href="mailto:soneil@alphalab.com">soneil@alphalab.com</a> 508 844-4117
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
	PCB Congeners	EPA 8270 Modified	1 L	4 ± 2°C; store in the dark	14 days to extraction	1 1-L amber bottle	Alpha Susan O'Neil <a href="mailto:soneil@alphalab.com">soneil@alphalab.com</a> 508 844-4117
				4 ± 2°C; store in the dark	40 days from extraction to analysis		
	Dioxin/Furan Congeners	EPA 1613B	1 L	0-4°C; store in the dark	45 days to extraction	2 1-L amber bottle	SGS <a href="mailto:amy.boehm@sgs.com">amy.boehm@sgs.com</a> 910 350-1903

**Table 6.1. Measurement Quality Objectives for n-Alkanes/Isoprenoids and TPH.**

Element or Sample Type	Minimum Frequency	Measurement Quality Objective/ Acceptance Criteria	Corrective Action
Initial Calibration (Standard solution - all target analytes, except nC <sub>39</sub> )	Prior to every sequence, or as needed based on continuing calibration/verification check.	5-point calibration curve %RSD ≤ 20	Resolve before proceeding.
Continuing Calibration (CCAL)	Every 12 field samples not to exceed 24 hours	%D ≤ 15 for 90% of analytes %D ≤ 20 for 10% of analytes	Perform Instrument Maintenance. Re-analyze affected samples.
Initial Calibration Verification (Second Source or can be met if CCAL is second source)	Per initial calibration	%R target analytes 80-120%	Resolve before proceeding.
SRMs - no SRMs for SHC or TPH are available at this time			
North Slope Crude Reference Oil	Prior to every sequence, reported per analytical batch	± 35% difference from established laboratory mean	Resolve before proceeding.
Matrix Spike/Matrix Spike Duplicate (Sediments/Soils)	One per batch/every 20 field samples	%R 50% - 125% for target analytes detected at >5X the spiked amount; RPD ≤ 30%.	Evaluate impact to data, discuss with manager, determine if corrective action is needed.
Laboratory Control Sample/ Laboratory Control Sample Duplicate	One per batch/every 20 field samples	%R 50% - 125% for target analytes, RPD ≤ 30%.	Resolve before proceeding.
Procedural Blank	One per batch/every 20 field samples	No more than 2 analytes to exceed 5x target MDL unless analyte not detected in associated samples(s) or analyte concentration >10x blank value	Resolve before proceeding. QA coordinator may be contacted to resolve issues surrounding 'minor exceedances'.
Duplicate Sample Analysis (not required for water matrix)	One per batch/every 20 field samples	RPD ≤ 30% if analyte concentration is greater than QL	Evaluate impact to data, discuss with manager, determine if corrective action is needed.
Mass Discrimination	Initial calibration and CCVs (mid-level)	Ratio for the raw areas of nC <sub>36</sub> /nC <sub>20</sub> ≥ 0.70	Resolve before proceeding.
Surrogates	Every sample	%R 40-125%	Re-extract affected samples. Evaluate impact to data, discuss with manager, determine if corrective action is needed.

**Table 6.2. Measurement Quality Objectives for Parent PAH and alkyl PAH**

Element or Sample Type	Minimum Frequency	Measurement Quality Objective/ Acceptance Criteria	Corrective Action
Tuning	Prior to every sequence	Tune as specified in laboratory SOP	Resolve before proceeding.
Initial Calibration (All parent PAH and selected alkyl homologue PAH isomers)	Prior to every sequence, or as needed based on continuing calibration/ verification check.	5-point calibration curve over two orders of magnitude %RSD $\leq$ 20	Resolve before proceeding.
Continuing Calibration (CCAL)	Every 12 field samples not to exceed 24 hours	%D $\leq$ 25 for 90% of the analytes; %D $\leq$ 35 for 10% of the analytes	Perform instrument maintenance. Re-analyze affected samples.
Initial Calibration Verification (Second Source or can be met if CCAL is second source)	Per initial calibration	%R target analytes 80-120%	Resolve before proceeding.
SRM 1941b for sediment	One per batch/every 20 field samples	Within $\pm$ 30% of NIST 95% uncertainty range for analytes within the quantitation range. 2 analytes may be greater than 30% outside, however average %D must be $<35\%$ <sup>1</sup>	Resolve before proceeding.
North Slope Crude Reference Oil	Prior to every sequence, reported per analytical batch	$\pm 20\%$ of established laboratory mean for the selected diagnostic compounds listed in Table 1.2 when $>10\times$ the RL; and $\pm 35\%$ difference from the established mean for all other analytes in Table 1.2 greater than $10\times$ the RL	Resolve before proceeding.
Matrix Spike/Matrix Spike Duplicate (Sediments/Soils)	One per batch/every 20 field samples	%R 50% - 125% for target analytes detected at $>5\times$ the spiked amount; RPD $\leq 30\%$ , except biphenyl (40%-140%) and decalin (25%-125%)	Evaluate impact to data, discuss with manager, determine if corrective action is needed.
Laboratory Control Sample/ Laboratory Control Sample Duplicate	One per batch/every 20 field samples	%R 50% - 125% for target analytes, RPD $\leq 30\%$ , except biphenyl (40%-140%) and decalin (25%-125%)	Resolve before proceeding.
Procedural Blank	One per batch/every 20 field samples	No more than 2 analytes to exceed $5\times$ target MDL unless analyte not detected in associated samples(s) or analyte concentration $>10\times$ blank value	Resolve before proceeding. QA coordinator may be contacted to resolve issues surrounding 'minor exceedance'.
Sample Duplicate (not required for water matrix)	One per batch/every 20 field samples	RPD $\leq 30\%$ if analyte concentration is greater than QL	Evaluate impact to data, discuss with manager, and determine if corrective action is needed.
Mass Discrimination	Initial calibration and CCVs (mid-level)	Ratio for the concentration of Benzo[g,h,i]perylene to phenanthrene $\geq 0.70$	Resolve before proceeding.
Internal Standard (IS)	Every sample	50% - 200% of the area of the IS in the associated calibration standard	Resolve before proceeding.

<sup>1</sup> Except for fluorene in SRM 1941b extend the low end to 40%.

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Element or Sample Type	Minimum Frequency	Measurement Quality Objective/ Acceptance Criteria	Corrective Action
Surrogates	Every sample	%R 40-120%	Re-extract affected samples. Evaluate impact to data, discuss with manager, if corrective action is needed.

**Table 6.3. Measurement Quality Objectives for TOC, Grain Size, and Total Solids.**

Element or Sample Type	Minimum Frequency	Acceptance Criteria	Relevant Parameter(s) Reference Methods*
Initial Calibration	Prior to analysis (method and instrument specific procedures & number of standards)	For multipoint calibration, correlation coefficient $r > 0.995$	TOC
Continuing Calibration (CCAL)	Must start and end analytical sequence and every 10 samples	%R 90% - 110%	TOC
Method Blank	One per batch/every 20 field samples	Not to exceed QL	TOC
Laboratory Control Sample	One per batch/every 20 field samples	%R 75% - 125%	TOC
Matrix Spike/Matrix Spike Duplicate	One per batch/every 20 field samples	%R 75% - 125% RPD $\leq 25$	TOC
Replicate Analyses <sup>a</sup>	Each sample must be analyzed at least in duplicate. The average of the replicates shall be reported.	RPD or RSD $< 20\%$ for concentrations $> QL$	TOC
Sample Duplicates <sup>b</sup>	One per batch/ every 20 field samples	RPD $\leq 25\%$ for analyte concentrations $> QL$	TOC
Reference Materials	One per batch/ every 20 field samples	Values must be within $\pm 20\%$ of NIST uncertainty range	TOC

<sup>a</sup> Method SW9060 requires quadruplicate analyses, however duplicate or triplicate analyses are acceptable. Standard Method 5310C requires that injections be repeated until consecutive measurements within 10% are obtained for a water matrix, however, duplicate analyses  $< 20\%$  RPD are acceptable based on a sediment matrix.

<sup>b</sup> Method SW9060 requires a duplicate spike. A matrix spike and sample supuplicate are acceptable in lieu of matrix spike/matrix spike duplicates. For grain size, RPD criteria only applied if fraction is greater than 5%.

**\*Reference Methods:**

TOC: Plumb 1981/SW 846 Method 9060A  
 Grain Size: ASTM D422. If using sieve analysis only, report as percent gravel, coarse sand, medium sand, fine sand, very fine sand, and silt/clay. If using sieve and hydrometer, report as percent gravel, coarse sand, medium sand, fine sand, very fine sand, silt, and clay.  
 Total Solids (percent): EPA 160.3

**Table 6.4. Measurement Quality Objectives for PCB Congeners**

Element or Sample Type	Minimum Frequency	Measurement Quality Objective/ Acceptance Criteria	Corrective Action
Tuning	Prior to every calibration Checked prior to every sequence	Tune as specified in laboratory SOP	Resolve before proceeding.
Initial Calibration	Prior to every sequence, or as needed based on continuing calibration/verification check.	5 point curve. %RSD $\leq$ 20% for 90% of the analytes; no analyte >30%	Resolve before proceeding.
Continuing Calibration (CCAL)	Every 12 field samples not to exceed 24 hrs	%D $\leq$ 20% for 90% of the analytes; no analyte >30%.	Perform instrument maintenance. Re-analyze affected samples.
Initial Calibration Verification (Second Source or can be met if CCAL is second source)	Per initial calibration	%R target analytes 80-120%	Resolve before proceeding.
Matrix Spike/Matrix Spike Duplicate (Sediments, Soils only). Waters if available aliquots.	One per batch/every 20 field samples	%R 40% - 140% for target analytes detected at >5X background; RPD $\leq$ 30%	Evaluate impact to data, discuss with manager, determine if corrective action is needed.
SRM 1941b for sediment	One per batch/every 20 field samples	40 – 140%	Resolve before proceeding.
LCS/LCSD	One per batch/every 20 field samples	%R 40% - 140% for target analytes, RPD $\leq$ 30%	Resolve before proceeding.
Procedural Blank	One per batch/every 20 field samples	Less than RL unless analyte not detected in associated samples(s) or analyte concentration >5x blank value	Resolve before proceeding. QA coordinator may be contacted to resolve issues surrounding 'minor exceedance'.
Sample Duplicate	One per batch/every 20 field samples	RPD $\leq$ 30% if analyte concentration is greater than RL	Evaluate impact to data, discuss with manager, and determine if corrective action is needed.
Internal Standard (IS)	Every sample	50% - 200% of the area of the IS in the associated calibration standard	Resolve before proceeding.
Surrogates	Every sample	%R 50-125%	Re-extract affected samples. Evaluate impact to data, discuss with manager, if corrective action is needed.

**Table 6.5. Measurement Quality Objectives for Chlorinated Pesticides**

Element or Sample Type	Minimum Frequency	Measurement Quality Objective/ Acceptance Criteria	Corrective Action
Initial Calibration	Prior to every sequence, or as needed based on continuing calibration/verification check.	Up to 10 levels may be analyzed; a minimum of 5-point calibration curve. %RSD $\leq$ 20, r is $\geq$ 0.99, the COD or $r^2$ is $\geq$ 0.99	Resolve before proceeding.
Continuing Calibration (CCAL)	Every 12 hours	%D $\leq$ 20	Perform instrument maintenance. Re-analyze affected samples.
Initial Calibration Verification (Second Source or can be met if CCAL is second source)	Per initial calibration	%R target analytes 80-120%	Resolve before proceeding.
SRM 1941b for sediment	One per batch/every 20 field samples	40 – 140%	Resolve before proceeding.
Matrix Spike/Matrix Spike Duplicate (Sediments/Soils)	One per batch/every 20 field samples	%R 40% - 140% for target analytes detected at $>5X$ the spiked amount; RPD $\leq$ 30%	Evaluate impact to data, discuss with manager, determine if corrective action is needed.
Laboratory Control Sample/Duplicate	One per batch/every 20 field samples	%R 40% - 140% for target analytes, RPD $\leq$ 30%.	
Method Blank	One per batch/every 20 field samples	No more than 2 analytes to exceed 5x target MDL unless analyte not detected in associated samples(s) or analyte concentration $>5x$ blank value	Resolve before proceeding. QA coordinator may be contacted to resolve issues surrounding 'minor exceedance'.
Sample Duplicate	One per batch/every 20 field samples	RPD $\leq$ 30% if analyte concentration is greater than QL	Evaluate impact to data, discuss with manager, and determine if corrective action is needed.
Internal Standard (IS)	Every sample	50% - 200% of the area of the IS in the associated calibration standard	Resolve before proceeding.
Surrogate	Every sample	%R 30-150%	Evaluate impact to data, discuss with manager, if corrective action is needed.

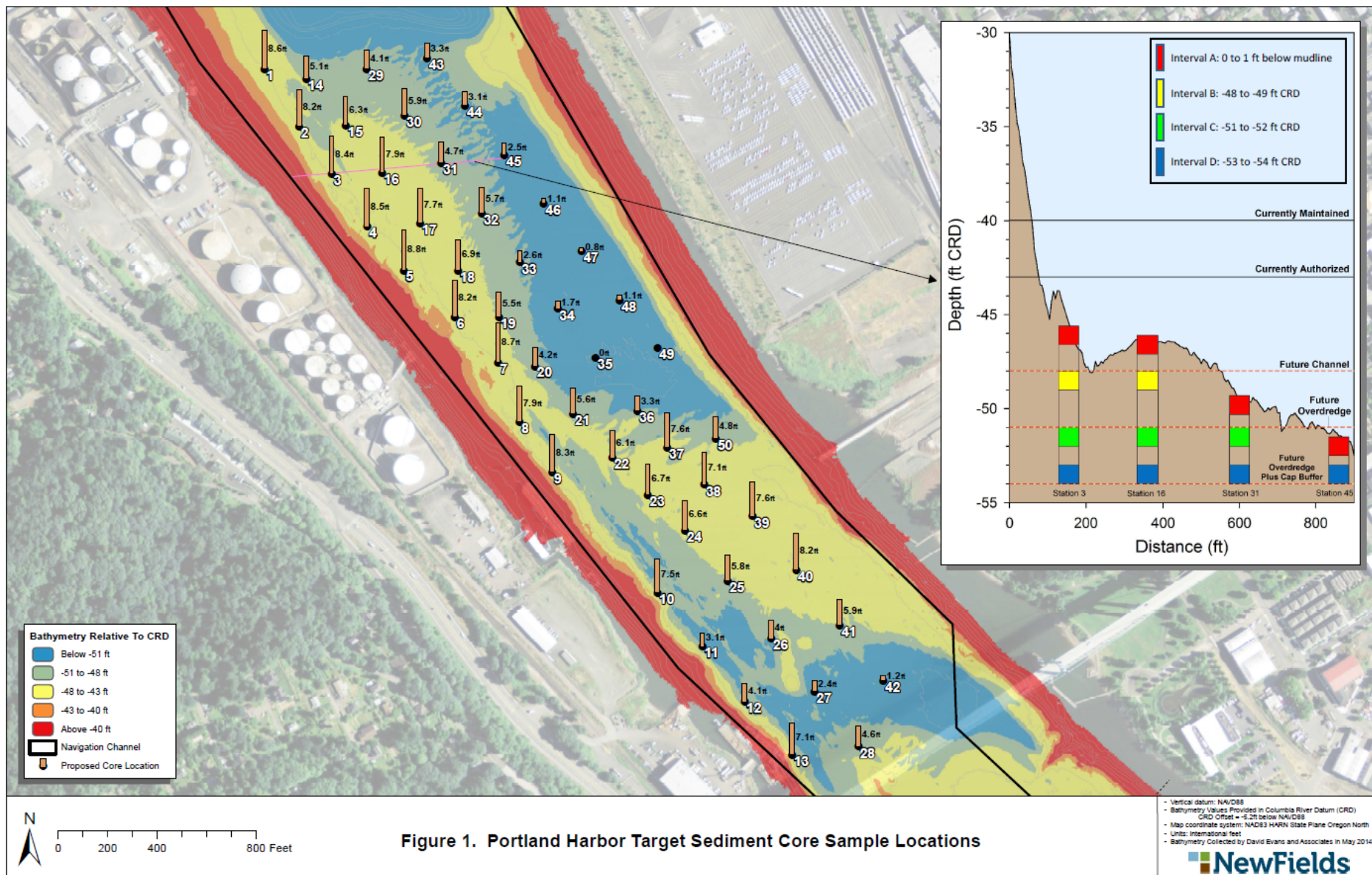
**Table 6.6. Measurement Quality Objectives for PCDD/Fs.**

<b>Element or Sample Type</b>	<b>Minimum Frequency</b>	<b>Measurement Quality Objective/ Acceptance Criteria</b>	<b>Corrective Action</b>
Initial Calibration	Six-point calibration curve - Initially and as required	< 20% RSD for native standards < 30% RSD for internal standards S/N ratio > 2.5 Isotope ratios and retention times within control limits	Resolve before proceeding.
Continuing Calibration Verification (CCV/VER)	Once per 12-hours, prior to sample analysis	Per table 6 of Method 1613B	Perform instrument maintenance. Re-analyze affected samples.
RT Window Defining Standard Mixture (CPSM)	Once per 12-hours, prior to sample analysis.	Valley between 2,3,7,8-TCDD and closest eluting isomer <25%. Valley between 2,3,7,8-TCDF and closest eluting isomer <40%. First and last eluting isomers identified.	Resolve before proceeding. Criteria must be met before any samples, blanks, or QC samples are analyzed.
Matrix Spike/Matrix Spike Duplicate	One per batch/every 20 field samples	%R 80% - 120% for target analytes detected at >5X background; RPD ≤30%	Evaluate impact to data, discuss with manager, determine if corrective action is needed.
LCS/LCSD (OPR/OPRD)	One per batch/every 20 field samples	%R 60% - 140% for target analytes, RPD ≤30%	Resolve before proceeding.
Laboratory Blank	One per batch/every 20 field samples	Less than RL unless analyte not detected in associated samples(s) or analyte concentration >5x blank value	Resolve before proceeding. QA coordinator may be contacted to resolve issues surrounding 'minor exceedance'.
Sample Duplicate	One per batch/every 20 field samples	RPD ≤ 30% if analyte concentration is greater than RL	Evaluate impact to data, discuss with manager, and determine if corrective action is needed.
Internal Standard (IS) and Surrogate standards (SS)	Every sample	Per table 7 of Method 1613B	Resolve before proceeding.

---

**Table 7.1. Laboratory Data Deliverables**

<b>Chain-of-Custody/Sample Receipt Checklist</b>	
Sample Data:	Result summaries including surrogate recoveries, percent total solids, dilutions, etc.
Standards Data:	Target MDL data based on the method in 40CFR,136 Calibration summaries: Initial calibration data, standard curve equation, correlation coefficient or %RSD, continuing calibration %D.
Quality Control Data (Method Blanks, CRMs, Duplicates, Matrix Spike, Laboratory Control Spikes):	Result summaries including surrogate recoveries, plus %R and RPD as applicable.
Case Narrative :	Special handling or analysis conditions. Any circumstance that requires special explanation such as an exception to QA/QC conditions or control criteria, dilutions, reanalysis, etc. Corrective actions/procedure alterations
Chromatograms and Extracted Ion Profiles:	Appropriately scaled (1) GC/FID chromatograms for samples and associated QC analyzed for extractable hydrocarbons; (2) GC/MS EIPs for samples and associated QC analyzed for biomarkers
Electronic Data Deliverable	As specified in laboratory contract.



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## **APPENDIX B: HEALTH AND SAFETY PLAN**

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**PORTLAND HARBOR SUPPLEMENTAL SEDIMENT STUDY**  
**Portland, Oregon**

***SITE SPECIFIC HEALTH AND SAFETY PLAN***

***OCTOBER 2015***

*Prepared for:*

**ExxonMobil**

*Prepared by:*



115 2<sup>nd</sup> Avenue N, Suite 100  
Edmonds, WA 98020

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## **1. GENERAL**

### **1.1 Introduction**

This Site-Specific Health and Safety Plan (SSHASP) has been developed for ExxonMobil as part of the Portland Harbor Supplemental Sediment Study. The purpose of this SSHASP is to define precautions to be taken to protect project personnel, the public and the environment. Because this project takes place on a hazardous waste site, work activities will be guided by the Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) regulations as outlined in 29 CFR 1910.120 and 1926.65.

This SSHASP addresses NewFields site activities for the Supplemental Sediment Study, as described in Section 2.2 Scope of Work. Work activities conducted off-site or by other companies are not included in this plan.

The plan will be made available to and must be reviewed by NewFields site personnel. NewFields personnel who have not read and signed this SSHASP will be restricted to the Support Zone. Personnel covered by this SSHASP who do not comply with the requirements will be excluded from site activities.

The content of this SSHASP may change or undergo revision based on field monitoring results, modifications to the technical scope of work, or additional information made available to NewFields personnel. Any proposed changes must be reviewed and approved by the Project Manager. Site personnel will be notified of changes prior to resuming work activities and will review the modified portions of the plan.

### **1.2 Project Personnel Including Responsibilities**

The following describes the project personnel and their health and safety roles and responsibilities for this project. This section will be updated as necessary to reflect the current project personnel and responsibilities for this project. All field personnel will receive ExxonMobil Loss Prevention System (LPS) training.

#### ***1.2.1 Project Manager***

The NewFields Project Manager (PM) for this site is Dr. Allen Uhler. The PM has responsibility and authority to direct NewFields' work operations. The PM coordinates safety and health functions with the Site Health and Safety Officer (HSO), has the authority to oversee and monitor the performance of the HSO, and bears ultimate responsibility for the proper implementation of this SSHASP. The specific duties of the PM are:

- Preparing and coordinating the site work plan
- Providing site supervisor(s) with work assignments and overseeing their performance
- Coordinating safety and health efforts with the HSO

- 
- Coordinating emergency response procedures with the Site Emergency Response Coordinator (ERC)
  - Serving as primary site liaison with public agencies, officials and other site contractors

### ***1.2.2 Site Health and Safety Officer (HSO)***

The NewFields Site Health and Safety Officer (HSO) for this site is Mr. Jasper Boas. The HSO has responsibility and authority to implement this SSHASP and to verify compliance with its requirements. The HSO reports to the Project Manager. The HSO is on site or readily accessible to the site during NewFields work operations and has the authority to stop work of NewFields personnel and subcontractors if unsafe conditions are detected. The specific responsibilities of the HSO are:

- Managing the NewFields safety and health functions on the site
- Serving as the point of contact for NewFields safety and health matters
- Ensuring all field staff have received LPS training
- Conducting or confirming the site monitoring, worker training, and effective use of PPE
- Assessing site conditions for unsafe acts and conditions and providing corrective action
- Assisting with the preparation of and/or reviewing this SSHASP
- Maintaining safety and health records as required by this SSHASP
- Communicating safety and health matters with the Emergency Response Coordinator (ERC), Site Supervisor(s), and others as necessary

### ***1.2.3 Site Supervisor***

The NewFields Site Supervisor for this site is Dr. Jonathan Nuwer. The Site Supervisor is responsible for field operations and reports to the Project Manager (PM). The Site Supervisor enforces the implementation of the SSHASP requirements and procedures in the field. The specific responsibilities of the Site Supervisor are:

- Executing the work plan and schedule as detailed by the PM
- Coordination with the HSO on safety and health matters
- Verifying and enforcing compliance with the requirements of this SSHASP

### ***1.2.4 Site Personnel***

Site personnel are responsible for complying with this SSHASP, using the proper PPE, reporting unsafe acts and conditions, attending safety training, and following the work and safety and health instructions of the PM, HSO, and Site Supervisor. All field personnel will have received LPS training prior to fieldwork. Field personnel will perform safe performance self-assessments (SPSAs), as taught in LPS training, throughout the course of the fieldwork.

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### 1.3 Emergency Contact Information

Facility/Agency	Emergency Phone Number	Non- Emergency Phone Number
<b>Police Department: Multnomah County Sheriff</b>	911	503-243-7952
<b>Fire Department</b>	911	
<b>U.S. Coast Guard: Portland – Search &amp; Rescue</b>	503-240-9301	
<b>Ambulance/Emergency Medical Assistance</b>	911	
<b>Hospital: Legacy Good Samaritan Hospital</b>	360-413-4128	360-695-1281
<b>Poison Control Center</b>	800-522-6337	
<b>National Response Center (NRC) for Oil/Chemical Spills</b>	800-424-8802	
<b>Site Emergency Coordinator</b>	206-335-4666	

## 2. SITE AND PROJECT DESCRIPTION

### 2.1 Physical Description

The study area for the Portland Harbor Superfund Site is located within the Willamette River, extending from the southern tip of Sauvie Island at RM 3.5 to the southern end of Swan Island at RM 9.2. Most industrial development along the banks of the Willamette River in Portland has occurred in this area, and the shoreline and channel have been significantly altered (e.g., bulkheads, piers) to accommodate industrial activities and urban growth. Sampling will be focused between RM 5 and RM 6 of the Superfund site, both in proximity to the shoreline and in the central channel.

### 2.2 Scope of Work

Sediment core will be collected from a vessel on the Willamette River. Sediment cores will be samples will be processed at Analytical Resources Inc. (ARI) in Tukwila, Washington. Once samples are processed at ARI, they will be shipped to laboratories for analysis.

### 2.3 Results of Past Investigations

Problem chemicals identified in previous Portland Harbor sediment investigations include heavy metals, dioxins, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), tributyltin (TBT), and pesticides.

## 3. HAZARD ASSESSMENTS

The following is a general discussion of the hazards that may be encountered on site. Required controls and PPE are included in Appendix B-2 – Job Safety Analysis Forms.

### 3.1 Chemical Hazards

Based on a review of available site history, including available sampling data, the chemicals listed in Table 3.1 are known or suspected to be present on the project site. Table 3.1 also includes information on the permissible exposure limits (PEL), immediately dangerous to life and health (IDLH) levels, exposure routes, and symptoms associated with each chemical. The samples to be collected will be wet, limiting inhalation exposure to vapors or particulates.

Table 3.1: Chemical Hazards						
Chemical of Concern	PEL - TWA <sup>1</sup> mg/m <sup>3</sup>	TLV - TWA <sup>2</sup> mg/m <sup>3</sup>	STEL mg/m <sup>3</sup>	Ceiling Limit mg/m <sup>3</sup>	Exposure Routes	Symptoms
2,3,7,8-TCDD	-	-	-	-	Inh, Abs, Ing, Con	Irritation to eyes, chloracne, dermatitis
Arsenic (organic)	0.5	-	-	-	Inh, Ing, Con	Irritation to skin, respiratory distress, diarrhea
Arsenic (inorganic)	0.002	0.01	-	-	Inh, Abs, Con, Ing	Ulceration of nasal septum, dermatitis, respiratory irritation
Bis(2-ethylhexyl)phthalate	5	-	10	-	Inh, Ing, Con	Irritation to eyes and mucus membranes
Coal Tar Pitch Volatiles <sup>3</sup>	0.2	0.2	-	-	Inh, Abs, Con	Dermatitis, bronchitis
Copper (dusts and mists)	1	1	-	-	Inh, Ing, Con	Irritation of eyes, nose, throat
Copper (fume)	0.1	0.2	-	-	Inh, Con	Fever, chills, muscle ache, nausea, dry throat, weakness
DDT	0.5	1	-	-	Inh, Abs, Ing, Con	Irritation to eyes and skin, tremors, dizziness, confusion, headache, vomiting
Hexane (89.6%)	180	176	-	-	nh, Ing, Con	Irritation to eyes, skin, respiratory system; head, giddiness, dizziness, light-head; nausea; chemical pneumonia, dermatitis
Hydrogen Sulfide	14	-	21	20 (ppm)	Ing, Ing, Con	Convulsions, eye and respiratory irritation, headache, dizziness
Lead	0.1	0.15	-	-	Inh, Ing, Con	Weakness, insomnia, constipation, abdominal pain, anemia, tremors
Methanol (99.8%)	260	260	325	-	Inh, Ing, Con, Abs	Headache, drowsiness, coughing, skin and eye irritation or burning, blindness
Naphthalene*	50	52	75	79	Inh, Abs, Ing, Con	Headache, nausea, vomiting, abdominal. pain, sweating, eye irritation
Nitric Acid (10% or 1.6N)	5	5	10	-	Ing, Ing, Con, Abs	Severe burns to eye or skin, breathing difficulties
PCB (42% chlorine)	0.001	1	-	-	Inh, Abs, Ing, Con	Irritation to eyes, chloracne
PCB (54% chlorine)	0.001	0.5	-	-	Inh, Abs, Ing, Con	Irritation to eyes, chloracne

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#### Table Notes

1. PEL-TWA values from NIOSH Pocket Guide to Chemical Hazards (1997).
2. TLV-TWA values from American Conference of Governmental Industrial Hygienists (1996).
3. PEL and TLV values for coal tar pitch volatiles include anthracene, benzo(a)pyrene, chrysene, and pyrene.

\*Naphthalene:  $50\text{mg}/\text{m}^3 = 10\text{ppm}$  (NIOSH 1997).

Inh = Inhalation, Abs = Absorption, Con = Contact, Ing = Ingestion

NA = Not applicable

NE = Not established

### **3.2 Physical Hazards**

NewFields site personnel may be exposed to the following physical hazards while conducting work activities on this site:

- Extreme weather
- Heat stress
- Cold stress
- Slips/trips/falls
- Cuts/abrasions
- Struck by heavy equipment
- Struck by vehicle
- Excessive noise
- Electricity
- Violence

### **3.3 Biological Hazards**

Biological hazards that may be encountered during this project include:

- Ticks and associated diseases
- Mosquitoes and associated diseases
- Spiders
- Stinging insects

### **3.4 Vessel Operational Hazards**

General vessel operational hazards and mitigating actions are included in Appendix B-2 – Job Safety Analysis Forms. Additionally, procedures for fuel spill response and the avoidance of collisions are discussed below.

#### **3.4.1 Fuel Spills**

Fueling of the sampling vessel will take place at Fred's Marina fuel dock at 12800 NW Marina Way. If a spill occurs during the fueling process, the following response actions will be followed:

- 
- Identify and stop the source of the leak
  - Notify the marina for assistance
  - Call the U.S. Coast Guard National Response Center (800) 424-8802
  - Contain spill with oil absorbent pads or booms
  - Properly dispose of used or saturated absorbents

### **3.4.2 Collisions**

Collision of the sampling vessel with other boats or objects will be avoided by following the actions below:

- The vessel will maintain a proper look-out by sight and hearing
- The sampling vessel will give way to sailing vessels, vessels engaged in fishing, and vessels with restricted maneuverability
- When moving at a speed of 10 knots or more a minimum distance of 30 meter will be kept from other vessels
- The vessel's course will be altered to the starboard when approaching a vessel head-on
- Using light and/or sound signaling
- Maintaining radio watch on VHF channel 16

## **3.5 Job Safety Analysis**

A job safety analysis (JSA) has been completed for each task described in Section 2.2 Scope of Work. Because the potential for exposure to hazards during this project depends primarily on the work activity being conducted and the location where the activity is performed, separate assessments will be completed for a task that is performed in multiple locations unless the hazards, potential exposures, and controls are the same.

JSAs will be updated as required if work procedures, site conditions, or identified hazards change. Affected site personnel will be notified of revisions to hazard assessments prior to re-commencement of work activities. Completed JSAs are located in Appendix B-2 – Job Safety Analysis Forms.

## **4. SITE CONTROL**

This site control plan is designed to reduce the spread of hazardous substances, identify and isolate contaminated areas, facilitate emergency evacuation and medical care, prevent unauthorized entry, and deter vandalism and theft. Because the site is a river that will be accessed by a vessel, the vessel is treated as the site.

The site control plan includes the following elements:

- Vessel description
- Vessel access and security
- Authorizations

- 
- Work zones
  - Buddy system information
  - Site communications

#### **4.1 Vessel Description**

The 36-foot vessel *R/V Nancy Anne*, owned and operated by Mr. Bill Jaworski of Marine Sampling Systems Inc. (MSS) will be used for all sediment collection activities. The *Nancy Anne* is a welded aluminum catamaran with a large forward working deck, purpose-built by MSS in 1994 to deploy sediment sampling equipment. Sediments will be collected from the platform using MSS's sampling equipment, including a power grab sampler and proprietary 'Marine Sampling Systems Vibracore'. MSS has more than 20 years of experience sediment sampling, including numerous Portland Harbor sediment investigations.

#### **4.2 Vessel Access and Security**

Access to the vessel is controlled in an effort to reduce potential for exposure to site hazards. MSS (subcontractor to NewFields) is responsible for vessel access control and security.

During working hours, access to the vessel is controlled by MSS with entry and egress at docking facilities. When no work activities are being conducted on the vessel, access will be controlled by MSS.

#### **4.3 Authorizations**

The PM or designee must approve NewFields personnel authorized to enter the site. To be authorized, personnel must meet the training and medical surveillance requirements as outlined by this SSHASP, including the signature of the individual on the Pre-Entry Briefing Sign-Off Form (see Appendix B-3).

##### **4.2.1 Visitors**

Visitors must be authorized by the PM or designee. Visitors will be escorted at all times and will be restricted to the Support Zone. Prior to accessing the vessel, visitors will receive and sign off on a pre-entry briefing as detailed in Section 5.6 Pre-Entry Briefing – Visitors.

#### **4.4 Work Zones**

Work zones are established to restrict access to areas where potential hazards exist and to implement appropriate controls for NewFields personnel working in each work zone. Different zones may be used aboard the vessel, as determined by PM or designee, based on

---

the presence or absence of hazards and the work tasks to be performed. These zones can be identified as:

1. Support Zone (SZ) – The SZ is the portion of the vessel where there is minimal potential for exposure to contamination.
2. Contamination Reduction Zone (CRZ) – The primary purpose of the CRZ is for decontamination of site personnel and equipment. The CRZ also serves as a buffer between the Exclusion Zone and the SZ to limit the potential spread of contamination to the SZ and the surrounding area. The boundaries of the CRZ will be marked with colored tape.
3. Exclusion Zone (EZ) – The EZ is the area of the site where hazardous substance(s) are known or suspected to be present. The boundaries of the EZ will be marked with colored tape.

Work zone classifications and boundaries may change as circumstances warrant. Whenever boundaries are adjusted, zone markings will also be changed and personnel will be notified.

#### **4.5 Buddy System**

NewFields personnel working in the EZ will utilize the buddy system. The buddy system consists of:

- Working in pairs
- Maintaining visual contact with one another
- Observing buddy for signs of distress
- Periodically checking each other's PPE for damage
- Notifying appropriate site personnel of emergencies

#### **4.6 Site Communication Plan**

Site personnel must be able to communicate with others on and off the site, including emergency coordinators, emergency response personnel, and NewFields Management site personnel and with off-site personnel and agencies. The primary means of communication for this project will be cell phones unless cell service is inadequate on the site.

### **5. TRAINING**

Site personnel will receive required training prior to accessing the site. Training requirements for this project include:

- HAZWOPER training
  - Initial
  - Manager/Supervisor
  - Refresher
- LPS training
- Pre-Entry Briefing, including SSHASP review

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Initial training will be conducted by a qualified and knowledgeable instructor. Refresher training can be completed by classroom, online, or computer-based training.

### **5.1 Initial HAZWOPER Training**

Initial training requirements are based on site personnel's potential for exposure to hazardous materials and compliance with the requirements of the HAZWOPER Standard Section (e)(3).

Personnel working intermittently on the site, whose exposure levels are expected to be below the permissible exposure limits, and who are not expected to be exposed to health hazards, will complete 24-hour initial HAZWOPER training consistent with the requirements of 29 CFR 1910.120(e)(3)(iii). In addition, such personnel will have received one day of supervised field experience under a trained and experienced supervisor.

Personnel who work regularly on site and have potential exposure at or above the permissible exposure limits, and who may be exposed to health hazards, must successfully complete 40-hour initial HAZWOPER training consistent with the requirements of 29 CFR 1910.120(e)(3)(i). In addition, such personnel will have received 3 days of supervised field experience under a trained and experienced supervisor.

### **5.2 HAZWOPER Manager/Supervisor Training**

On-site managers and supervisors who are directly responsible for and/or who supervise workers engaged in hazardous waste operations will have completed an additional eight (8) hours of specialized supervisory training.

### **5.3 HAZWOPER Refresher Training**

Site personnel will receive annual 8-hour HAZWOPER refresher training consistent with the requirements of the HAZWOPER Standard. This training will review health and safety topics relevant to this site.

### **5.4 HAZWOPER Equivalent Training**

For this project, prior academic training or job experience will not be accepted in lieu of HAZWOPER initial training.

For this project, prior academic training or job experience will be accepted in lieu of HAZWOPER initial training, as described in paragraphs (e)(1)-(e)(4) of the standard. In accordance with HAZWOPER (e)(9), NewFields will certify that such personnel have equivalent training and will include a copy of that certification with other training records.

---

## **5.5 LPS Training**

All site personnel will receive general LPS training. Additionally, the Site Supervisor will receive manager/supervisor LPS training. This training introduces such tools as:

- Safe Performance Self-Assessment (SPSA)
- Job Safety Analysis (JSA)
- Root Cause Analysis
- Loss Prevention Observation (LPO)

## **5.6 Pre-Entry Briefing – Site Personnel**

Site personnel will review this SSHASP and will attend a pre-entry briefing prior to commencement of work, to familiarize them with the project safety requirements and the contents of the SSHASP. Additional briefings will be provided to notify personnel of changes in site conditions and/or the requirements. This may include but is not limited to:

- Changes in the work schedule or plan
- Newly identified hazards and required controls
- Work zone boundary adjustments
- Work related incidents

Site workers must acknowledge that they have attended the pre-entry briefing by signing the Site Personnel Pre-Entry Briefing Form in Appendix B-3.

## **5.7 Pre-Entry Briefing – Visitors**

A pre-entry briefing is required for all site visitors. The pre-entry briefing for visitors will include but is not limited to:

- Brief site history
- Site hazards and controls, including required PPE
- Site lay-out including work zones
- Emergency response procedures
- Other pertinent safety and health requirements

Visitors must acknowledge that they have attended the pre-entry briefing by signing the Visitor's Pre-Entry Briefing Form in Appendix B-3.

## **5.8 Additional Training**

No additional training is required for site personnel on this project.

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## **5.9 Training Recordkeeping**

Training records for this project will be maintained by the PM or designee. Records will be kept in NewFields' Edmonds, Washington office.

## **6. MEDICAL SURVEILLANCE**

Medical surveillance is provided to medically monitor the health of personnel who:

- May be or are exposed to hazardous substances at or above the established PEL for 30 or more days a year;
- Are required to wear a respirator; **or**
- Are injured, become ill, or develop signs or symptoms due to possible overexposure to hazardous substances.

When medical examinations and procedures are required for medical surveillance, they will be conducted by or under the supervision of a licensed physician and provided to personnel free of cost, without loss of pay, and at a reasonable time and place.

Currently none of the above situations applies to NewFields site personnel on this project. As such, medical surveillance is not required for NewFields site personnel at this time.

## **7. PERSONAL PROTECTIVE EQUIPMENT (PPE)**

Safety and health hazards will be eliminated or reduced to the greatest extent possible through engineering controls and work practices. Where hazards cannot be adequately controlled, a combination of work practices, and personal protective equipment (PPE) will be used to protect site personnel. PPE selection and use will comply with the following NewFields SOPs:

- SOP #15 *Hearing Conservation Program*
- SOP #20 *Personal Protective Equipment*
- SOP #21 *Respiratory Protection*

### **7.1 PPE Level**

The initial PPE level for each work task is provided in Appendix B-2 – Job Safety Analysis Forms. Initial PPE level was determined based on the work activity, the anticipated route(s) of entry of hazardous substances on site, and contaminant concentration(s).

PPE level may be upgraded or downgraded depending on changing site conditions and work procedures. Criteria for PPE changes are included in Appendix B-2.

### **7.2 Respiratory Protection**

When respiratory protection is required for this project, it is indicated in the hazard analysis.

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Use of respiratory protection will comply with the NewFields Respiratory Protection Program. Please refer to NewFields SOP # 21 *Respiratory Protection Program*.

### **7.3 Hearing Conservation**

Site personnel with potential exposure to noise levels exceeding the OSHA Action Level of 85 dBA as an 8-hour time-weighted average will be included in the NewFields Hearing Conservation Program. Where noise exposure meets or exceeds this level, noise is listed as a physical hazard in the hazard analysis for the tasks/operation, and hearing protection is required PPE. Based on current site conditions and planned operations, there are no site tasks which have noise exposure that equals or exceeds 85 dBA.

For the requirements of the NewFields Hearing Conservation Program, please see NewFields SOP #15 *Hearing Conservation*.

## **8. EXPOSURE MONITORING PLAN**

This exposure monitoring plan is designed to determine the potential exposure for site personnel to hazardous substances on the site. Exposure monitoring will be conducted and interpreted by qualified personnel. This section specifies exposure monitoring to be conducted by NewFields site personnel.

### **8.1 Air Monitoring**

Air monitoring may be used to determine the potential exposure for site personnel to hazardous airborne substances. NewFields site personnel are not required to conduct air monitoring for this project.

## **9. DECONTAMINATION**

Decontamination procedures are designed to achieve an orderly, controlled removal or neutralization of contaminants that may accumulate on personnel or equipment. They minimize personnel contact with contaminants and the transfer of contamination to clean areas. These procedures also address how to properly dispose of residual waste from the decontamination process.

Decontamination procedures will be conducted in the CRZ. The location and design of the decontamination stations will minimize the spread of contamination.

NewFields site personnel required to enter the EZ will comply with the decontamination procedures detailed below. These procedures are designed to meet the requirements of 1910.120(k) and include site-specific information about the decontamination process for

- Personnel and PPE
- Equipment
- Emergencies
- Proper disposal of residual waste

- 
- Monitoring the effectiveness of decontamination procedures

### **9.1 Decontamination Procedures**

NewFields personnel will follow the decontamination procedures detailed below:

- *Personnel*

Gloves will be replaced or thoroughly washed using Liquinox or other phosphate-free detergent and rinsed with distilled water before and after handling sediment samples. Personal non-disposable field equipment (e.g., boots, waterproof gloves, garments) will be rinsed with water and brushed clean prior to leaving the CRZ. Special attention will be given to removing mud and sediments that may have adhere to boot treads.

- *Equipment*

Site water will be used to rinse sediments from vibracore sampler. The sampler will then be thoroughly washed using Liquinox or other phosphate-free detergent and rinsed with site water. If a residual creosote or petroleum sheen remains on the sampling equipment or is difficult to remove using the standard decontamination procedure above, a final hexane rinse may be added.

- *Emergencies*

In the event of an emergency involving personal injury, care will be taken to spread contamination beyond the CRZ.

### **9.2 Disposal of Residual Waste**

Residual waste will be handled and disposed of in compliance with local, state, and federal regulations. Clean Harbors, of Kent, Washington will handle all waste sediments from this project, including but not limited to transportation, waste hazard classification and disposal.

Used PPE such as disposable gloves and supplies (e.g., paper towels and packaging) will be placed in plastic storage bags and disposed of as municipal waste. If PPE contains residual sediments, the PPE will be decontaminated using the procedures outlined above and will be disposed of as non-hazardous material. Recyclable waste material (e.g., cardboard, aluminum) will be recycled as feasible.

### **9.3 Monitoring the Effectiveness of Decontamination Procedures**

The effectiveness of decontamination procedures will be evaluated and monitored. Visual examination is used to verify that decontamination procedures are implemented as required by this SSHASP and to evaluate whether contamination is controlled by these procedures. Equipment rinsate blanks will be collected and analyzed for chemical parameters as a means of determining the effectiveness of decontamination procedures.

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## **10. EMERGENCY RESPONSE PROCEDURES**

Emergency situations at this site may include:

- Fire/Explosion
- Accidental releases
- Injury or illness
- Man Overboard

All emergency situations must be reported immediately to the PM.

Emergency contact information is provided in Section 1.3. In addition, emergency contact information, basic emergency response procedures, and the location and directions to the nearest medical facility are included in Appendix B-1 of this SSHASP.

### **10.1 Emergency Evacuation Procedures**

NewFields personnel will stop work and assemble at the designated muster location on the vessel. The HSO will account for NewFields personnel at the muster point. The U.S. Coast Guard will be notified immediately if any NewFields personnel cannot be accounted for.

### **10.2 Fire/Explosion**

In case of a fire or explosion, NewFields personnel will follow emergency evacuation procedures. If NewFields personnel discover a fire or witness an explosion, they will notify the HSO and Emergency Coordinator of the location of the fire/explosion and any related injuries/illnesses as soon as it is safe to do so.

### **10.3 Accidental Hazardous Chemical Releases**

NewFields personnel are not qualified to respond to accidental emergency releases of hazardous chemicals. If an emergency release occurs, NewFields personnel will follow emergency evacuation procedures.

As soon as it is safe to do so, NewFields personnel who discover a hazardous substance release will contact the HSO and the U.S. Coast Guard and provide as much of the following information as possible:

- Identification of hazardous substance
- Location of the release
- Estimated quantity of substance released
- Direction/flow of release
- Known related fire or explosion incidents
- Injuries or illnesses resulting from the release

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## **10.4 Injuries/Illnesses**

If an injury or illness occurs, immediately notify the U.S. Coast Guard. NewFields personnel with current first aid/CPR certification may render aid as appropriate.

## **10.5 Man Overboard**

If a man-overboard situation occurs, all vessel engines will be stopped immediately. Floatation devices (e.g., life rings) attached to lines will be throw to the victim from the vessel. The victim will then be brought aboard the vessel; wet clothing will be removed and replaced with dry clothing. The victim may need to be treated for cold stress. No other person should enter the water unless the victim is unconscious or seriously injured. Rescuers must wear life vests and be tethered to the sampling vessel or shore.

## **11. CONFINED SPACES**

The purpose of this section is to identify permit-required confined spaces (PRCS) on site and to describe the procedures that have been developed to safeguard site personnel working in PRCS. In compliance with the requirements of 29 CFR 1910.120(b)(4)(ii)(I), this section is included even when no PRCS are present to indicate that a site-specific evaluation for permit spaces has been made. All identified confined spaces are treated as PRCS until it is determined otherwise.

No confined spaces were identified during the site-specific evaluation.

## **12. RECORDKEEPING**

The PM or designee will maintain health and safety records for this project. Records will be maintained by the Site Supervisor.

## **13. APPLICABLE SOPS**

Other NewFields SOPs will be practiced that are applicable to this project:

- SOP #8 Emergency Action Plan
- SOP #11 Fire Prevention
- SOP #12 First Aid and CPR
- SOP #13 Hazardous Chemical Communication
- SOP #14 HAZWOPER
- SOP #15 Hearing Conservation
- SOP #16 Heat/Cold Stress
- SOP #17 Incident/Accident Investigation and Reporting
- SOP #20 Personal Protective Equipment

- 
- SOP #23 Subcontractor Safety
  - SOP #24 Vehicle Safety
  - SOP #26 Stop Work Procedure

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## **Appendix B-1 – Emergency Contact and Response Information**

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## Emergency Services and Contacts

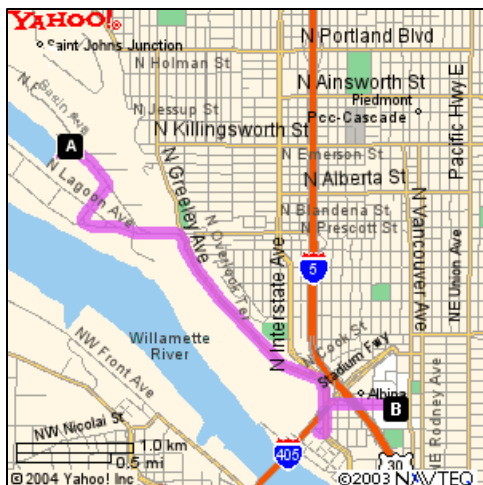
Service	Name/Location	Phone Number
Ambulance	--/--	911
Fire	--/--	911
Police	Emergency	911
	Multnomah County Sheriff River Patrol	503-243-7952
Coast Guard	Portland Search & Rescue	503-240-9301
Hospital	Legacy Emanuel Hospital & Health Center 2801 N Gantenbein Ave Portland, OR 97227	360-413-4128
	Legacy Good Samaritan Hospital 1015 NW 22 <sup>nd</sup> Ave Portland, OR 97210	503-413-7711
NewFields PM	Allen Uhler	781-681-5040 x106
ExxonMobil PM	Deborah Edwards	281-639-2505
		908-403-3140

## Emergency Response Procedures

1. Call 911
  - Give your name and location
  - Give location of accident/incident
  - Give name and affiliation of injured person
  - Describe injuries/spill/accident
  - Describe type of medical aid provided so far
  - Alert if injured person is contaminated
  - Give a summary of accident and suspected cause
  - Give time of occurrence
  - Describe temporary measures taken to minimize risk
  - Establish meeting location with ambulance
2. Notify SSHO
3. Notify NewFields PM
4. Notify ExxonMobil PM
5. Site Supervisor to complete a report within 24 hours

## Directions to Legacy Emanuel Hospital

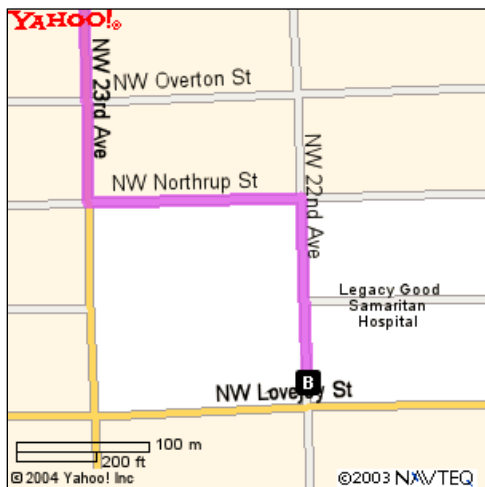
1. Start at the **Swan Island Lagoon** boat ramp parking lot
2. Turn **Right** on **N BASIN AVE** - go **0.3** mi
3. Turn **Right** on **N ANCHOR ST** - go **0.2** mi
4. Continue on a local road - go **0.1** mi
5. Continue on **N CHANNEL AVE** - go **0.1** mi
6. Continue on **N GOING ST** - go **0.4** mi
8. Continue on **N GREELEY AVE** - go **0.9** mi
9. Bear **Right** on **N INTERSTATE AVE** - go **0.3** mi
10. Continue on a local road - go **< 0.1** mi
11. Turn **Right** on **N LARRABEE AVE** - go **< 0.1** mi
12. Turn **Right** on **N RANDOLPH AVE** - go **< 0.1** mi
13. Turn **Right** on **N RUSSELL ST** - go **< 0.1** mi
14. Turn **Left** on **N INTERSTATE AVE** - go **0.1** mi
15. Turn **Right** on **N GRAHAM ST** - go **0.4** mi
16. Turn **Right** on **N GANTENBEIN AVE** - go **< 0.1** mi
17. Arrive at **2801 N GANTENBEIN AVE, PORTLAND**



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## Directions to Legacy Good Samaritan Hospital

1. Start at **Fred's Marina** on **NW MARINA WAY** - go **0.4** mi
2. Turn **Left** on **NW ST HELENS RD** - go **6.4** mi
3. Continue on **NW NICOLAI ST** - go **0.1** mi
4. Bear **Right** on **NW WARDWAY ST** - go **0.1** mi
5. **NW WARDWAY ST** becomes **NW VAUGHN ST** - go **0.5** mi
6. Turn **Right** on **NW 23RD AVE** - go **0.4** mi
7. Turn **Left** on **NW NORTHRUP ST** - go **0.1** mi
8. Turn **Right** on **NW 22ND AVE** - go **0.1** mi
9. Arrive at **1015 NW 22ND AVE, PORTLAND**



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## **Appendix B-2 – Job Safety Analysis Forms**

Job Safety Analysis Form		
<b>Company Name</b>		<b>Date Revised</b>
NewFields		6/12/2014
<b>Task/Operation</b>		<b>Location(s)</b>
General Vessel Operations		Portland Harbor, Willamette River
Job Safety Analysis		
Job Steps	Potential Hazards	Critical Actions to Mitigate Hazards
1) Vessel orientation	Not knowing proper emergency procedures	-Boat operator to conduct safety brief. -Keep boat clear of trash/debris/tripping hazards.
2) Transfers: Boat to shore. Shore to boat	Falling overboard; Slips, Trips, and Falls; Pinch Points; Poor dock/pier condition; Abrupt boat handling; Bumping dock too hard on approach; Dangerous wave height.	-Approved PFD must be worn at all times -Follow direction of vessel Captain and crew -Maintain three points of contact at all times -Keep hands and feet clear of pinch points, motors, and other moving equipment.
3) Transits	Falling overboard; Slips, Trips, and Falls; Pinch Points; Weather - storms, fog; Striking submerged objects.	-Approved PFD must be worn at all times -Follow direction of vessel Captain and crew -Maintain three points of contact at all times -Keep hands and feet clear of pinch points, motors, and other moving equipment. -When working outside cabin wear foul-weather PPE. -Remain seated while vessel is in transit.
Physical Hazards		
Name of Physical Hazard	Source	Exposure Potential
Pinch Points	boat deck, machinery	Likely
Heat (ambient)	weather	Unlikely
Cold Weather Operations	weather	Likely
Heavy Manual Lifting/Moving	sampling device	Likely
Slips/Trips/Falls	cluttered/wet boat deck area	Likely
Inclement Weather – Snow, rain,fog	weather	Likely
Biological Hazards		
Name of Biological Hazard	Source	Exposure Potential
Insect bites and stings	Insects in gear or near piers	Likely
Control Measures Used		
Level of PPE: D		

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<b>Location:</b> on boat deck	<b>PPE Equipment:</b> Chemical-resistant steel toe boots, PVC Bib-style overalls and jacket with hood, splash-proof safety goggles, nitrile gloves, hardhat, PFD Type III.
<b>Work Practices:</b>	Always wear PDF. Keep boat deck clear of debris. Keep clear of pinch points. Maintain three points of contact at all times. Be seated while vessel is in motion.
<b>Hazard Assessment Conducted by Jon Nuwer on 6/12/2014</b>	
<i>Signature</i>	

Job Safety Analysis Form		
Company Name		Date Revised
NewFields		6/12/2014
Task/Operation		Location(s)
Sediment and Water Sampling		Portland Harbor, Willamette River
Job Safety Analysis		
Job Steps	Potential Hazards	Critical Actions to Mitigate Hazards
4) Deploy sampler over side of vessel	Pinched by sampler	-Keep hands away from pinch points -Don't hold onto sampler while suspended
	Slip or fall on deck	-Keep work area clear of clutter -Keep deck free of sediment -Wear boots with tread
	Struck by sampler	-Only deploy in calm water -Use tag lines to allow distance from sampler -Wear hard hat at all times
	Fall into river	-Wear PFD at all times -Life-rings on deck -Use buddy system
5) Recover sampler	--Same as above--	--Same as above--
6) Move sampler on deck	Back strain – not using legs to lift or twisting with load	-Bend and lift with legs/arms, keep back straight and do not twist -Use two people to lift
7) Remove sediment/water samples from sampler	Cut or abrasion while opening sample	-Wear cut-resistant gloves while opening sampler
	Exposure to hazardous chemicals	-Wear chemical resistant gloves -Wear splash-resistant safety glasses -Wear long-sleeved coat and overalls
8) Package samples	Cut from broken glass	-Inspect glassware for cracks -Wear gloves -Handle carefully
9) Onloading and Offloading gear from vessel	Slip or fall on ramp or dock	-Wear boots with tread -Ensure dock is free of debris

	Back strain – not using legs to lift or twisting with load				-Bend and lift with legs/arms, keep back straight and do not twist -Use two people to lift	
	Fall into river				-Wear PFD at all times -Use buddy system	
Chemical Hazards						
Chemical of Concern	PEL - TWA <sup>1</sup> mg/m <sup>3</sup>	TLV - TWA <sup>2</sup> mg/m <sup>3</sup>	STEL mg/m <sup>3</sup>	Ceiling Limit mg/m <sup>3</sup>	Exposure Routes	Symptoms
2,3,7,8-TCDD	-	-	-	-	Inh, Abs, Ing, Con	Irritation to eyes, chloracne, dermatitis
Arsenic (organic)	0.5	-	-	-	Inh, Ing, Con	Irritation to skin, respiratory distress, diarrhea
Arsenic (inorganic)	0.002	0.01	-	-	Inh, Abs, Con, Ing	Ulceration of nasal septum, dermatitis, respiratory irritation
Bis(2-ethylhexyl)phthalate	5	-	10	-	Inh, Ing, Con	Irritation to eyes and mucus membranes
Coal Tar Pitch Volatiles <sup>3</sup>	0.2	0.2	-	-	Inh, Abs, Con	Dermatitis, bronchitis
Copper (dusts and mists)	1	1	-	-	Inh, Ing, Con	Irritation of eyes, nose, throat
Copper (fume)	0.1	0.2	-	-	Inh, Con	Fever, chills, muscle ache, nausea, dry throat, weakness
DDT	0.5	1	-	-	Inh, Abs, Ing, Con	Irritation to eyes and skin, tremors, dizziness, confusion, headache, vomiting
Hexane (89.6%)	180	176	-	-	nh, Ing, Con	Irritation to eyes, skin, respiratory system; head, giddiness, dizziness, light-head; nausea; chemical pneumonia, dermatitis
Hydrogen Sulfide	14	-	21	20 (ppm)	Ing, Ing, Con	Convulsions, eye and respiratory irritation, headache, dizziness
Lead	0.1	0.15	-	-	Inh, Ing, Con	Weakness, insomnia, constipation, abdominal pain, anemia, tremors
Methanol (99.8%)	260	260	325	-	Inh, Ing, Con, Abs	Headache, drowsiness, coughing, skin and eye irritation or burning, blindness
Naphthalene*	50	52	75	79	Inh, Abs, Ing, Con	Headache, nausea, vomiting, abdominal. pain, sweating, eye irritation
Nitric Acid (10% or 1.6N)	5	5	10	-	Ing, Ing, Con, Abs	Severe burns to eye or skin, breathing difficulties
PCB (42% chlorine)	0.001	1	-	-	Inh, Abs, Ing, Con	Irritation to eyes, chloracne
PCB (54% chlorine)	0.001	0.5	-	-	Inh, Abs, Ing, Con	Irritation to eyes, chloracne

Physical Hazards		
Name of Physical Hazard	Source	Exposure Potential
Pinch Points	boat deck, machinery, samplers	Likely
Heat (ambient)	weather	Unlikely
Cold Weather Operations	weather	Likely
Heavy Manual Lifting/Moving	sampling device	Likely
Oxidizers	decontamination solution	Likely
Slips/Trips/Falls	cluttered/wet deck	Likely
Inclement Weather – Snow, rain,fog	weather	Likely
Chemicals	sediment	Likely
Biological Hazards		
Name of Biological Hazard	Source	Exposure Potential
Insect bites and stings	Insects in gear or near piers	Likely
Control Measures Used		
Level of PPE: D		
Location: on boat deck	PPE Equipment: Chemical-resistant steel toe boots, PVC Bib-style overalls and jacket with hood, splash-proof safety goggles, nitrile gloves, hardhat, PFD Type III.	
Work Practices:	Frequent changes of disposable nitrile gloves Wash hands and face with soap and water after each sampling event Take shower at end of workday Check extension cords are intact and connections are not in contact with wet surfaces.	
Hazard Assessment Conducted by Jon Nuwer on 6/12/2014		
Signature		

#### Notes

1. PEL-TWA values from NIOSH Pocket Guide to Chemical Hazards (1997).
2. TLV-TWA values from American Conference of Governmental Industrial Hygienists (1996).
3. PEL and TLV values for coal tar pitch volatiles include anthracene, benzo(a)pyrene, chrysene, and pyrene.

\*Naphthalene:  $50\text{mg}/\text{m}^3 = 10\text{ppm}$  (NIOSH 1997).

Inh = Inhalation, Abs = Absorption, Con = Contact, Ing = Ingestion

Job Safety Analysis Form		
Company Name		Date Revised
NewFields		10/14/2015
Task/Operation		Location(s)
Sediment Core Transport and Handling		Portland Harbor, Willamette River
Job Safety Analysis		
Job Steps	Potential Hazards	Critical Actions to Mitigate Hazards
1) Position truck at boat launch to receive cores	-Hit dock with truck or back truck into river	-Person on dock will communicate with driver -Voice and hand signals will be used to inform driver of position and actions to be taken
	Slip or fall on dock	-Keep dock area clear of clutter -Wear boots with tread
	Fall into river	-Wear PFD at all times -Life-rings on dock -Use buddy system
2) Move cores from boat to truck	Back strain – not using legs to lift or twisting with load	-Bend and lift with legs/arms, keep back straight and do not twist -Use two people to lift -Use lifting straps to ensure secure hold
	Cut or abrasion grasping core barrel	-Wear cut-resistant gloves -Inspect core for sharp edges
	Exposure to hazardous chemicals	-Wear chemical resistant gloves under work gloves -Wear splash-resistant safety glasses -Wear long-sleeved coat and overalls
	Slip or fall on dock	-Keep dock area clear of clutter -Wear boots with tread
	Fall into river	-Wear PFD at all times -Life-rings on dock -Use buddy system
3) Secure cores within truck	Fall/slip while climbing in/out of truck	-Remove PFD for better mobility -Use handles on side of truck to pull yourself up -Maintain three points of contact with fixed objects -Wear boots with tread

	Injured by falling core while securing	-Work in teams of two with one person always having secure hold of the core -Fit ratchet straps securely around core and tighten completely -Test security of strapping
	Slip or fall in back of truck	-Wear boots with tread -Ensure truck is free of debris -Wipe up any spills
4) Offloading cores at processing facility	Back strain – not using legs to lift or twisting with load	-Bend and lift with legs/arms, keep back straight and do not twist -Use two people to lift -Use lifting straps to ensure secure hold
	Fall/slip while climbing in/out of truck	-Use handles on side of truck to pull yourself up -Maintain three points of contact with fixed objects -Wear boots with tread
	Cut or abrasion grasping core barrel	-Wear cut-resistant gloves -Inspect core for sharp edges
<b>Physical Hazards</b>		
<b>Name of Physical Hazard</b>	<b>Source</b>	<b>Exposure Potential</b>
Pinch Points	truck door deck	Likely
Heat (ambient)	weather	Unlikely
Cold Weather Operations	weather	Likely
Slips/Trips/Falls	cluttered/wet dock or truck bed	Likely
Inclement Weather – Snow, rain, fog	weather	Likely
Chemicals	sediment	Likely
<b>Biological Hazards</b>		
<b>Name of Biological Hazard</b>	<b>Source</b>	<b>Exposure Potential</b>
Insect bites and stings	Insects in gear or near piers	Likely
<b>Control Measures Used</b>		
<b>Level of PPE: D</b>		
<b>Location:</b> on dock	<b>PPE Equipment:</b> Chemical-resistant steel toe boots, splash-proof safety goggles, heavy work gloves, hardhat, PFD Type III.	
<b>Location:</b> on pavement	<b>PPE Equipment:</b> Chemical-resistant steel toe boots, splash-proof safety goggles, heavy work gloves, hardhat.	

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<b>Work Practices:</b>	Always wear PDF when working over water. Keep dock and truck bed clear of debris. Keep clear of pinch points. Maintain three points of contact at all times.
<b>Hazard Assessment Conducted by Jon Nuwer on 10/14/2015</b>	
<i>Signature</i>	

Job Safety Analysis Form		
<b>Company Name</b>		<b>Date Revised</b>
NewFields		10/14/2015
<b>Task/Operation</b>		<b>Location(s)</b>
Sediment Core Processing		Analytical Resources, Inc., Tukwila, WA
Job Safety Analysis		
Job Steps	Potential Hazards	Critical Actions to Mitigate Hazards
1) Position core on processing table	Back strain – not using legs to lift or twisting with load	-Bend and lift with legs/arms, keep back straight and do not twist -Use two people to lift -Use lifting straps to ensure secure hold
	Slip or fall	-Keep lab area clear of clutter -Wear boots with tread
	Cut or abrasion grasping core barrel	-Wear cut-resistant gloves -Inspect core for sharp edges
2) Slice open core barrel	Injury from circular saw	-Wear eye and ear protection -Wear heavy work gloves -Ensure all personnel and electrical cords are clear from saw path -Use both hands to operate saw -Push along cutting path, slowing if resistance is reached -Unplug saw when not using
	Cut or abrasion grasping core barrel	-Wear cut-resistant gloves -Inspect core for sharp edges
	Exposure to hazardous chemicals	-Wear chemical resistant gloves under work gloves -Wear splash-resistant safety glasses -Wear long-sleeved coat and overalls
	Slip or fall	-Keep dock area clear of clutter -Wear boots with tread
3) Remove sediment samples from core	Cut or abrasion grasping core barrel	-Wear cut-resistant gloves -Inspect core for sharp edges

	Exposure to hazardous chemicals	-Wear chemical resistant gloves under work gloves -Wear splash-resistant safety glasses -Wear long-sleeved coat and overalls
4) Packaging and shipping samples	Cut from broken glass	-Inspect glassware for cracks -Wear gloves -Handle carefully
	Back strain - not using legs to lift or twisting with load	-Bend and lift with legs/arms, keep back straight and do not twist -Use two people to lift
<b>Physical Hazards</b>		
<b>Name of Physical Hazard</b>	<b>Source</b>	<b>Exposure Potential</b>
Pinch Points	processing table	Likely
Cuts	circular saw	Likely
Cold Weather Operations	weather	Likely
Slips/Trips/Falls	cluttered/wet floor	Likely
Chemicals	sediment	Likely
<b>Biological Hazards</b>		
<b>Name of Biological Hazard</b>	<b>Source</b>	<b>Exposure Potential</b>
None		
<b>Control Measures Used</b>		
<b>Level of PPE: D</b>		
<b>Location:</b> in lab	<b>PPE Equipment:</b> Chemical-resistant steel toe boots, Long-sleeve shirt, long pants, splash-proof safety goggles, nitrile gloves.	
<b>Work Practices:</b>	Frequent changes of disposable nitrile gloves Keep lab floor clear of debris. Keep clear of saw path Wash hands and face with soap and water after each sampling event Take shower at end of workday Check extension cords are intact and connections are not in contact with wet surfaces	
<b>Hazard Assessment Conducted by Jon Nuwer on 10/14/2015</b>		
<i>Signature</i>		

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## **Appendix B-3 – Pre-Entry Briefing Sign Off Forms**

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### Portland Harbor Site Personnel Pre-Entry Briefing Form

Personnel will receive site-specific training and will read the project SSHASP prior to entering the site. Upon completing these tasks, personnel must indicate their understanding and agreement with the requirements of the SSHASP by signing below.

I have received site-specific training for Portland Harbor sampling activities. I have also read and understand the Site-Specific Health and Safety Plan. I will comply with all of its provisions.

Name (Print)	Signature	Date

---

## Portland Harbor Site Visitor Pre-Entry Briefing Form

Site visitors will attend a pre-entry briefing prior to accessing the site. The briefing will provide an overview of the site and project and the requirements with which visitors must comply. Visitors will indicate their understanding and agreement with the requirements explained in this briefing by signing below.

I have attended the pre-entry briefing for Portland Harbor sampling activities. The briefing included information regarding:

- Brief site history and description
- Site hazards and controls, including required PPE
- Site lay-out including work zones
- Emergency response procedures
- Other pertinent safety and health requirements

I understand and will comply with the requirements explained during the briefing.

Name (Print)	Signature	Date

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## **Appendix B-4 – Vessel Operations Checklist**

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## Vessel Operations Checklist

### Items Required by Federal Law

- ☐ Wearable Life Jackets
- ☐ Throwable PFD
- ☐ Fire Extinguisher
- ☐ Backfire Flame Arrester
- ☐ Ventilation System
- ☐ Whistle or Horn
- ☐ Navigational Lights
- ☐ Registration Documents
- ☐ Boat Number, permanent decal

### Emergency Gear

- ☐ Throw Rope/Bag
- ☐ Signaling Mirror
- ☐ Whistle (on life jacket)
- ☐ Rescue Blanket
- ☐ First Aid Kit
- ☐ VHF Radio

### Personal Items

- ☐ Sun Screen
- ☐ Sunglasses
- ☐ Prescription Medication
- ☐ Hard Hat
- ☐ PFD
- ☐ Steeltoe Boots
- ☐ Rain Gear
- ☐ Food
- ☐ Water
- ☐ Insect Repellant
- ☐ Nitrile Gloves
- ☐ Work Gloves
- ☐ Safety Glasses

### Miscellaneous Items

- ☐ Dewatering Device (bucket, bailer, bilge pump)
- ☐ Anchor and Line
- ☐ Moring Lines
- ☐ Toolkit
- ☐ Navigational Equipment (maps, compass, GPS)
- ☐ Spare Parts (as appropriate)
- ☐ Flashlight (with working batteries)
- ☐ Communications (VHF radio, CB radio, cell phone)
- ☐ Knife
- ☐ Repair Kit/Duct Tape
- ☐ Boat Hook
- ☐ Extra Fuel/Oil

Continued on next page.

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## Vessel Operations Checklist (continued)

### Sampling Gear

- ☐ Vibracore
- ☐ Core Barrels
- ☐ Cutter Heads
- ☐ Power Grab
- ☐ Winch
- ☐ Stainless Steel Pans
- ☐ Stainless Steel Spoons
- ☐ Sample Jars
- ☐ Sample Labels
- ☐ Chain of Custody Forms
- ☐ Field Sampling Plan
- ☐ Health and Safety Plan
- ☐ Zipties
- ☐ Liquinox soap
- ☐ Nitrile gloves
- ☐ Earplugs
- ☐ Cleaning brushes
- ☐ Electrical tape
- ☐ Bungee Cords, pkg
- ☐ Notebooks
- ☐ Writing pads
- ☐ Pens
- ☐ Markers
- ☐ Paper towels
- ☐ Distilled Water
- ☐ Coolers
- ☐ Packing Tape
- ☐ Camera (with working batteries)

### Pre-Launch Checklist

- ☐ Complete check for leaks
- ☐ Motor in good operating condition
- ☐ Propeller in good condition
- ☐ Fuel tanks full
- ☐ Suitable weather and water conditions
- ☐ Passengers briefed on emergency procedures
- ☐ Life jackets checked for fit
- ☐ Float plan left with responsible person on shore

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# APPENDIX C: FIELD FORMS

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## SEDIMENT CORING LOG (Vibracore)

Core Number \_\_\_\_\_

PROJECT NAME: Exxon Mobil 2015 Portland Harbor Sediment Sampling  
DATE SAMPLED: \_\_\_\_\_  
LOCATION: \_\_\_\_\_  
TIME: \_\_\_\_\_  
UNCORRECTED DEPTH (ft): \_\_\_\_\_  
SURFACE ELEVATION (ft MLLW): \_\_\_\_\_  
MUDLINE ELEVATION (ft MLLW): \_\_\_\_\_  
SAMPLED BY: \_\_\_\_\_

CORE PENETRATION: \_\_\_\_\_  
CORE RECOVERY: \_\_\_\_\_  
% RECOVERY: \_\_\_\_\_

### NOTES:

Surface Sample Bottom:

$$MD = [E_{ML} - E_D] \times (PR/100)$$

MD = Max sample depth (feet)

$E_{ML}$  = Mudline elevation (feet MLLW)

$E_D$  = Sample bottom elevation (feet MLLW)

PR = Percent recovery

DEPTH		SAMPLE ID	SEDIMENT DESCRIPTION	COMMENTS
Recovered Core Length	Feet Below Mudline Based on Actual % Recovery			
1				
2				
3				
4				
5				
6				
7				

REVIEWED BY: \_\_\_\_\_ PAGE \_\_\_\_\_ OF \_\_\_\_\_

**Project:** 2015 ExxonMobil Portland  
Harbor Sediment Sampling

**Station:** \_\_\_\_\_

**Location:** \_\_\_\_\_

**Date/Time:** \_\_\_\_\_

**Crew:** \_\_\_\_\_

Grab #	Bottom Depth	Penetration Depth	Time
<i>Sediment Type:</i>	<i>Sediment Color:</i>	<i>Sediment Odor:</i>	<i>Comments:</i>
Cobble Gravel Sand C M F Silt / Clay Organic matter Woody debris Shell debris	Drab olive Brown Brown surface Gray Black Other:	None Slight Moderate Strong	
Grab #	Bottom Depth	Penetration Depth	Time
<i>Sediment Type:</i>	<i>Sediment Color:</i>	<i>Sediment Odor:</i>	<i>Comments:</i>
Cobble Gravel Sand C M F Silt / Clay Organic matter Woody debris Shell debris	Drab olive Brown Brown surface Gray Black Other:	None Slight Moderate Strong	
Grab #	Bottom Depth	Penetration Depth	Time
<i>Sediment Type:</i>	<i>Sediment Color:</i>	<i>Sediment Odor:</i>	<i>Comments:</i>
Cobble Gravel Sand C M F Silt / Clay Organic matter Woody debris Shell debris	Drab olive Brown Brown surface Gray Black Other:	None Slight Moderate Strong	
Grab #	Bottom Depth	Penetration Depth	Time
<i>Sediment Type:</i>	<i>Sediment Color:</i>	<i>Sediment Odor:</i>	<i>Comments:</i>
Cobble Gravel Sand C M F Silt / Clay Organic matter Woody debris Shell debris	Drab olive Brown Brown surface Gray Black Other:	None Slight Moderate Strong	

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Sample Container Logbook

<b>Client:</b> ExxonMobil	<b>Location ID:</b>
<b>Project:</b> 2015 Portland Harbor Sediment Sampling	<b>Time/Date Collected:</b>
<b>Crew:</b>	<b>Time/Date Processed:</b>
<b>Comments:</b>	

Sample Container Tag Number	Sample ID	Analysis	Laboratory

**Notes:**

Report Originator: \_\_\_\_\_ Responsible for Response: \_\_\_\_\_

**DESCRIPTION OF PROBLEM:**

Date and Time \_\_\_\_\_

Problem Recognized: \_\_\_\_\_ By: \_\_\_\_\_

Date of \_\_\_\_\_

Actual Occurrence: \_\_\_\_\_ By: \_\_\_\_\_

Analyte: \_\_\_\_\_ Analytical Method: \_\_\_\_\_

Cause of Problem: \_\_\_\_\_

**CORRECTIVE ACTION PLANNED:**

Person Responsible  
for Corrective Action: \_\_\_\_\_

Corrective Action  
Plan Approval: \_\_\_\_\_

Date of  
Corrective  
Action: \_\_\_\_\_

Date: \_\_\_\_\_

**DESCRIPTION OF FOLLOW-UP ACTIVITIES:**

Person Responsible for Follow-up Activities: \_\_\_\_\_ Date of Follow-up Activity: \_\_\_\_\_

Final Corrective Action Approval: \_\_\_\_\_ Date: \_\_\_\_\_

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## **APPENDIX D: STANDARD OPERATING PROCEDURES**

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## **SEDIMENT CORE COLLECTION AND PROCESSING**

Sediment cores are collected to evaluate chemical and/or biological characteristics of surface and subsurface sediments at depths that greatly exceed those achieved by grab or other surface samplers. The purpose of this standard operating procedure (SOP) is to define and standardize procedures for the collection of samples from surface and subsurface sediment cores. Additionally, this SOP will help ensure that the highest quality, most representative data are collected, and that these data are comparable to data from other programs. This SOP is based on the procedures outlined in Puget Sound Estuary Program guidelines (PSEP 1996).

### **SUMMARY OF METHOD**

Sediment cores are collected using some type of coring device, including gravity corers, piston corers, vibracorers and diver-driven cores. Actual operations will vary depending on the equipment selected. Selection of the most appropriate corer usually depends on many factors, including but not limited to:

- The quantity of sample required
- The penetration depth required
- The sediment type
- Vessel availability and capability

Regardless of the coring method, the core tube should be constructed of a non-contaminating material such as stainless steel or aluminum, or should use a liner constructed of a non-contaminating material (e.g., polycarbonate).

Once the sediment core is collected, it is extruded or split so that the sediment can be sampled, processed, and transported to the analytical laboratory.

### **SUPPLIES AND EQUIPMENT**

A generalized supply and equipment list is provided below. Additional equipment may be required depending on project requirements.

#### Sampling Device

- Corer
- Core tubes
- Core tube liners (optional)
- Core tube caps

#### Field Equipment

- Aluminum foil
  - Decontamination supplies
  - Duct tape
  - Hack saw
  - Indelible ink pen
  - Pipe cutter
  - Circular saw (if splitting tube longitudinally)
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- Plunger (if necessary)
  - Table or tray
  - Ice (if storing cores)
  - Stainless-steel bowls
  - Stainless-steel spoons, spatulas, and/or mixer
  - Personal protective equipment for field team
  - First Aid kit
  - Cell phone
  - Sample containers
  - Bubble wrap
  - Clear tape
  - Permanent markers
  - Pencils
  - Coolers

#### Documentation

- Core description forms or log book
- Waterproof field logbook
- Field sampling plan
- Health and safety plan
- Correction forms
- Request for change forms
- Waterproof sample description forms

### **CORE COLLECTION PROCEDURE**

#### **Corer Deployment**

The vibracorer uses a hydraulic system that vibrates and drives a length of aluminum tubing into the sediment. The vibracorer will use aluminum core tubes approximately 4 inches in diameter. A stainless core nose with fingers will be pushed into the aluminum tube and riveted in place. The core tube will then be clamped tightly to the chuck on the vibracore head. The vibracorer is then lowered to the bottom, where the unit is energized and allowed to penetrate. The core will be driven to target depth or refusal.

#### **Corer Retrieval and Extrusion**

Once the sampler is brought back onboard the survey vessel, the tube will be disconnected from the vibracore head and laid at an angle more than horizontal (not to let material come out top of tube). The core is then capped on both ends to preserve the sediment sample. Extrusion is done by splitting the aluminum core tube in half using a circular saw with a blade depth just enough to cut cleanly through the aluminum core tube without disturbing the sediment core inside. Two passes with the circular saw are made on opposite sides of the core tube, splitting the core tube directly in half down the centerline. The aluminum core tube is then removed and the sediment core is rolled out onto a clean, aluminum-foil-covered processing table.

A new clean aluminum core tube will be used for every sediment core collected. Care will be taken during sampling to avoid contact of the sample tube with potentially contaminated surfaces. Extra

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sample tubes will be available during sampling operations for uninterrupted sampling in the event of a potential core tube breakage or contamination. Core tubes suspected to have been accidentally contaminated will not be used. Logs and field notes of all core samples will be maintained as samples are collected and correlated to the sampling location map. The following information will be included in this log:

- Elevation of each station sampled
- Location of each station as determined by DGPS
- Date and time of collection of each sediment core sample
- Names of field supervisor and person(s) collecting and handling the sample
- Observations made during sample collection including: weather conditions, complications, ship traffic, and other details associated with the sampling effort
- The sample station identification
- Length and depth intervals of each core and estimated recovery for each sediment sample
- Qualitative notation of apparent resistance of sediment column to coring
- Any deviation from the approved sampling plan

### **SEDIMENT SAMPLE PROCESSING**

Sediment processing and sampling techniques are as follows:

- Immediately following core extrusion and splitting, collect samples for volatile samples (either organics or sulfides) using a decontaminated, stainless-steel spoon. The volatile organics sample jar should be tightly packed (to eliminate obvious air pockets) and filled so that there is no head-space remaining in the jar. Alternatively, if there is adequate water in the sediment, the container may be filled to overflowing so that a convex meniscus forms at the top, and the cap carefully placed on the jar. Once sealed, there should be no air bubbles. The sulfides sample is preserved with 0.2 N zinc acetate.
- Record core sediment characteristics on a core description form. Observations should include stratification of color and sediment composition, odor, biological organisms, foreign objects etc.
- Place remaining core sediment in a decontaminated, stainless-steel bowl (see SOP for Surface Sediment Sampling) and mix thoroughly with a stainless-steel spoon, spatula or mixer until uniform color and texture are achieved. Large rocks or wood pieces may be omitted from the final laboratory sample, but should be noted in the log or description form.
- If sediment from multiple core sections will be composited, cover the bowl with clean foil and set the bowl aside (refrigerate or keep cool on ice) while handling additional cores. Once all the required sediment has been placed in the bowl, thoroughly mix until uniform color and texture are achieved.
- Transfer aliquots of homogenized sediment to labeled sample containers provided by the analytical laboratory. Labels should include, at minimum, the company name, project name, sample identifier, date and time of collection, and the initials of sampling personnel.
- Pack and transport samples as described in the SOP for Surface Sediment Sampling. If samples will be stored, follow procedures specified in the project sampling plan.

### **QUALITY CONTROL PROCEDURES**

Field quality control (QC) samples that may be collected during sediment coring are the same as for any field sampling program. The types and frequency of field QC sample collection are project-specific and

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will be described in the field sampling plan. The most commonly collected field QC sample are described below (PSEP 1996):

- Field Blank. A field blank is a sample of analyte-free water that is supplied by the laboratory. The field blank is generated by transferring the analyte-free water to another laboratory-supplied sample container while at the field sampling location. Field blank results are used to measure and document any possible onsite contamination.
- Field Split Sample. A field split sample consists of aliquots of the same homogenized sediment sample that are equally distributed in two sets of sample containers. These samples may be analyzed identically or analyzed by different laboratories to evaluate repeatability of sample handling and analytical procedures, sample heterogeneity, and analytical procedures.
- Field Replicate. A field replicate consists of a second sample that is collected using the same sampling methodology used to obtain the first sample. It is collected at the same sampling location and as soon after the original sample as possible. Analysis of the field replicate allows evaluation of the repeatability of field sampling methodologies, as well as the heterogeneity of the sample matrix. Statistical analysis of multiple replicates may also be used to calculate the likely range of an analyte concentration at a given sampling location.

## **REFERENCES**

PSEP. 1996. Puget Sound Estuary Program: Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound. Prepared for U.S. Environmental Protection Agency, Region 10, and Puget Sound Estuary Program Seattle, WA. Tetra Tech and HRA, Inc., Bellevue, WA.

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## **SURFACE SEDIMENT SAMPLING AND PROCESSING**

The purpose of this standard operating procedure (SOP) is to define and standardize the methods for collecting surface sediment samples from freshwater or marine environments. For the purpose of this SOP, surface sediments are defined as those from 0 to at most 30 cm below the sediment-water interface. The actual definition of surface sediments is typically program-specific and is dependent on the purpose of the study and the regulatory criteria (if any) to which the data will be compared.

This SOP utilizes and augments the procedures outlined in Puget Sound Estuary Program (PSEP 1996) guidelines. A goal of this SOP is to ensure that the highest quality, most representative data be collected, and that these data are comparable to data collected by different programs that follow PSEP guidelines.

### **SUMMARY OF METHOD**

Sediment samples for chemical and toxicity analysis are collected using a surface sediment sampling device (e.g., grab sampler). If a sample meets acceptability guidelines, overlying water is siphoned off the surface and the sediment is described in the field log. Sediment samples for chemical analysis may be collected directly from the sampler (e.g., volatile organic compounds and sulfides) or sediment from the sampler may be homogenized using decontaminated, stainless-steel containers and utensils prior to being placed in sample jars. Sediment from several sampler casts may also be composited.

### **SUPPLIES AND EQUIPMENT**

A generalized supply and equipment list is provided below. Additional equipment may be required depending on project requirements.

#### Sampling Device

- Grab sampler or box corer

#### Field Equipment

- Siphoning hose
  - Aluminum foil
  - Decontamination supplies
  - Indelible ink pen
  - Table or tray
  - Ice (if storing cores)
  - Stainless-steel bowls
  - Stainless-steel spoons, spatulas, and/or mixer
  - Personal protective equipment for field team
  - First Aid kit
  - Cell phone
  - Sample containers
  - Bubble wrap
  - Clear tape
  - Permanent markers
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- Pencils
  - Coolers

#### Documentation

- Grab sample description forms or log book
- Waterproof field logbook
- Field sampling plan
- Health and safety plan
- Correction forms
- Request for change forms
- Waterproof sample description forms

### **PROCEDURES**

#### **Equipment Decontamination**

The basic procedure used most commonly in field projects to decontaminate field sampling equipment is as follows:

- Rinse with tap or vessel water
- Wash with brush and Liquinox detergent
- Double rinse with distilled water
- Rinse with 0.1 N nitric acid (optional – if metals analysis is being performed)
- Rinse with methanol or hexane (optional – if organics analysis is being performed or adhering petroleum residue present)
- Rinse with distilled water
- Cover with aluminum foil

This procedure may be modified depending on site-specific requirements, as described in PSEP (1986). For example, if sampling is in areas known to be uncontaminated or only slightly contaminated, the solvent and/or acid rinses may be eliminated. Conversely, if creosote or other petroleum-based residue is encountered, a hexane rinse may be added.

Decontamination with acid or solvents should always be performed outdoors using appropriate protective equipment, including, at a minimum, chemical-resistant gloves (e.g., nitrile) and goggles. All decontamination liquids that include solvents or acids should be contained in tightly sealed buckets or other containers for disposal in an approved onshore facility. Alternatively, low-vapor pressure solvents may be evaporated in a well-ventilated open area away from the work zone.

#### **Sediment Sample Collection**

To collect sediment for chemical and biological analyses, a sampler that obtains a quantifiable volume of sediment with minimal disturbance of the sediments must be employed. Additionally, the sampler should be composed of a material such as stainless steel or aluminum, or have a non-contaminating coating such as Teflon™. Samplers capable of providing high-quality sediment samples include grab-type samplers (van Veen, Smith-McIntyres, Young grab, power-grab and ponar grab) and box cores (Soutar, mini-Soutar, Gray-O'Hara, spade core). Some programs require a sampler that collects from a

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specific area (e.g., 0.1 m<sup>2</sup>). Most sampling devices are typically a standard size; however, some non-standard sizes are available to meet the requirements of specific programs. Grab samplers, especially the van Veen grab, are the most commonly used samplers to collect surface sediment. Power grab samplers are often used for programs requiring collection of sediment deeper than 10 cm or in areas with debris.

A hydraulic winch system should be used to deploy the sampler at a rate not exceeding 1 m/sec to minimize the bow wake associated with sampler descent. Once the sampler hits the bottom, the jaws are slowly closed and the sampler is brought to the deck of the vessel at a rate not exceeding 1 m/sec to minimize any washing and disturbance of the sediment within the sampler. At the moment the sampler hits the bottom, the time, depth, and location of sample acquisition are recorded in the field logbook.

Once onboard, the sampler is secured, any overlying water is carefully siphoned off, and the sample is inspected to determine acceptability. Criteria used to determine acceptability are those detailed in PSEP (1986), except when noted in the project-specific field sampling plan (FSP). These criteria include but are not limited to:

- There is minimal or no excessive water leakage from the jaws of the sampler.
- There is no excessive turbidity in the water overlying the sample.
- The sampler is not over-penetrated.
- The sediment surface appears to be intact with minimal disturbance.
- The program-specified penetration depths are attained.

If the sample meets acceptability criteria, the sample is recorded and observations entered into a sample description form or log. Once the sample has been characterized, the sediment is then subsampled for chemical and biological analyses.

### **Sample Processing**

Sediment for chemical and/or toxicity analyses is removed from the sampler using a stainless-steel spoon. Depending on programmatic goals, the upper 2 to 30 cm of sediment are removed. To prevent possible cross contamination, sediments touching the margins of the sampler are not used.

Samples for volatile compounds (either organics or sulfides) are collected using a decontaminated stainless-steel spoon while sediment is still in the sampler. These sediments are not homogenized. The volatile organics sample jar should be tightly packed with sediment (to eliminate obvious air pockets) and filled so that there is no headspace remaining in the jar. Alternatively, if there is adequate water in the sediment, the container may be filled to overflowing so that a convex meniscus forms at the top, and the cap carefully placed on the jar. Once sealed, there should be no air bubbles. The sulfides sample is preserved with 0.2 N zinc acetate.

The remaining sediment is then placed into a pre-cleaned, stainless-steel bowl. Once a sufficient amount of sediment has been collected, the sediment is homogenized until it is of uniform color and has obtained a smooth consistency. It is then dispensed into pre-cleaned sample jars for the various chemical or biological analyses. Sample jars for biological analyses should be filled to the top with sediment to minimize available headspace. This procedure will minimize any oxidation reactions within the sediment. Sample jars for chemical analysis may be frozen for storage, leaving enough headspace left in the container to allow for expansion of the sediment upon freezing. After dispensing the sediment, the containers are then placed into coolers with ice and are either shipped directly to the analytical laboratories or transported to a storage facility.

### **Chain-of-Custody**

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## Field

The cruise leader or other designated field sample custodian is responsible for all sample tracking and chain-of-custody procedures until sample custody is transferred to the laboratory. Custody procedures in the field are as follows:

- Record all field and sample collection activities (including sample identification number, collection time and date) in the field logbook. While being used in the field, the logbook remains with the field team at all times. Upon completion of the sampling effort, the logbook should be reproduced and then kept in a secure area.
- Complete a chain-of-custody form whenever samples are being transferred or removed from the custody of field sampling personnel. Record each individual sample on the form. Include additional information to assist in sample tracking such as collection date and time, number of containers, and sample matrix. The chain-of-custody may also serve as the sample analysis request form, with the required analysis indicated for each individual sample.
- Sign the form and ensure that the samples are not left unattended unless secured.
- Store, pack, or ship samples as described in the following section. Place the original completed chain-of-custody form in a sealed plastic bag inside the shipping container. A copy is retained by the shipping party.
- Complete a separate custody form for each individual shipping container or a single form for all samples in multiple shipping containers in a single shipment, with the number of containers noted on the custody form.
- Attach completed custody seals to any shipping container that will be sent to the laboratory by delivery service or courier. Delivery personnel are not required to sign the custody form if custody seals are used. Custody seals are used to detect unauthorized tampering with the samples. Gummed paper or tape should be used so that the seal must be broken when the container is opened. The laboratory sample custodian (or other sample recipient) will establish the integrity of the seals.
- The laboratory custodian (or other sample recipient) acknowledges receipt of the samples by signing, dating, and noting the time of transfer on the chain-of-custody form. The condition of the samples and any problems or irregularities (e.g., cracked or broken jars, loose lids, evidence of tampering) should also be recorded. Return a copy of the completed custody form to the project manager or designated sample coordinator.

## Laboratory

The laboratory will designate a sample custodian who is responsible for receiving samples and documenting their progress through the laboratory analytical process. Each custodian will ensure that the chain-of-custody and sample tracking forms are properly completed, signed, and initialed on transfer of the samples. Specific laboratory chain-of-custody procedures should be in writing, included in the laboratory QA plan, and approved prior to beginning sampling and analysis. Laboratory custody procedures should include the following:

- A designated laboratory person initiates and maintains a sample tracking log that will follow each sample through all stages of laboratory processing and analysis.
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- The laboratory tracking log includes, at a minimum, the sample number, location and type of storage, date and time of each removal, and signature of the person removing or returning the sample.
  - The final disposition of the sample is recorded.

### **Chain-of-Custody Quality Control Procedures**

Complete and correct chain-of-custody is essential to ensure and demonstrate sample integrity. Errors in entering information or transferring custody can result in analytical or data reporting errors. Inaccuracies or errors in sample tracking and custody records can compromise data usability, particularly as legal evidence.

Quality control procedures include the following:

- Allow adequate time to take accurate and complete field records and to carefully complete chain-of-custody forms.
- When possible, work in pairs or more to complete the chain-of-custody form and check for accurate information entry.
- Complete all custody records in ink; errors should be neatly crossed out and corrected and initialed by the person making the change.
- Immediately notify the project manager of any deviation from required custody procedures.

### **Packing and Shipping Samples**

Environmental samples are packed in a manner to reduce the chance of sample breakage, ensure sample integrity, and prevent material leakage and potential exposure to hazardous materials in the event of breakage. Samples are placed in sealed plastic bags and packed in a sturdy container with adequate packing material to prevent breakage. Ice or dry ice may be included to maintain sample storage conditions. Samples are transported by field personnel or shipped via courier or common carrier. Shipping procedures are in accordance with U.S. Department of Transportation regulations (49 CFR 173.6 and 49 CFR 173.24).

All preserved samples should be shipped as soon as possible after completion of sampling. This minimizes the number of people handling samples and protects sample quality and security.

#### Sample Packing

Upon completion of final sample inventory by the field sample custodian and completion of chain-of-custody, samples are packed as follows:

- If not already done after sample collection, wipe the outside of each sample container and lid with a disposable cloth to remove any soil or sediment adhering to the outside of the jar and place each container in a sealed plastic bag (e.g., ziplock).
  - Wrap each glass sample container in bubble wrap or place it in a bubble wrap plastic bag. [Note: When samples are being transported by field personnel directly from the field site to the laboratory (thereby ensuring careful handling), this step is recommended but may be omitted. However, this step is required when a courier or delivery service is transporting the samples.]
  - Line the shipping container with heavy-duty plastic bags (e.g., garbage bags) and bubble wrap. Use a leak-proof, sturdy container that can withstand rough treatment during shipping. If ice chests or coolers are used, the drain should be securely plugged and sealed with duct tape.
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- Place the samples tightly in the shipping container.
  - Place the original completed chain-of-custody form in a sealed plastic bag and place it inside the shipping container. If using a cooler or ice chest, the form should be securely taped to the inside of lid.
  - Tie or seal the bag lining the shipping container.
  - If required to meet sample storage requirements, fill the ice chest with crushed or block ice, blue ice (refrigerated samples, 4°C) or dry ice (frozen samples). A temperature blank (provided by the laboratory) should be packed in each cooler.
  - If samples for volatile organics analysis (VOA) are included in the shipping container, a VOA trip blank (provided by the analytical laboratory) should also be packed in the cooler.
  - Seal shipping container securely with packing or duct tape.
  - If the shipping container will be transported by anyone other than the person who completed and signed the chain-of-custody form, attach completed custody seals so that the shipping container cannot be opened without breaking the seal.
  - Attach a This End Up label to each side of the shipping container to ensure that jars are transported in an upright position. A Fragile label may also be attached to reduce rough handling of the samples.
  - Label the shipping container with all appropriate information (name of project, time and date, responsible person and company name, address and phone) to enable positive identification.

#### Sample Shipping

Packed containers may be delivered to the laboratory or storage facility by field personnel, courier, or common carrier (FedEx, UPS). However, any outside carrier or courier service must provide a delivery receipt. The carrier or courier must also ensure delivery time if holding time and storage conditions are critical.

Unless arranged in advance, shipping charges should be prepaid by sender to avoid confusion and possible rejection of the package by the laboratory.

The adequacy of handling and shipping procedures is reflected in the condition of the samples upon receipt by the laboratory:

- No jars are cracked or broken.
- There is no evidence of sample leakage.
- Measuring the temperature of the temperature blank indicates that correct storage conditions have been maintained.

The sample custodian or other designated person is responsible for confirming that copies of all shipping documents, completed in full and correctly, are on file.

#### **QUALITY CONTROL PROCEDURES**

Field quality control (QC) samples that may be collected during sediment coring are the same as for any field sampling program. The types and frequency of field QC sample collection are project-specific and will be described in the field sampling plan. The most commonly collected field QC sample are described below (PSEP 1996):

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- Field Blank. A field blank is a sample of analyte-free water that is supplied by the laboratory. The field blank is generated by transferring the analyte-free water to another laboratory-supplied sample container while at the field sampling location. Field blank results are used to measure and document any possible onsite contamination.
  - Field Split Sample. A field split sample consists of aliquots of the same homogenized sediment sample that are equally distributed in two sets of sample containers. These samples may be analyzed identically or analyzed by different laboratories to evaluate repeatability of sample handling and analytical procedures, sample heterogeneity, and analytical procedures.
  - Field Replicate. A field replicate consists of a second sample that is collected using the same sampling methodology used to obtain the first sample. It is collected at the same sampling location and as soon after the original sample as possible. Analysis of the field replicate allows evaluation of the repeatability of field sampling methodologies, as well as the heterogeneity of the sample matrix. Statistical analysis of multiple replicates may also be used to calculate the likely range of an analyte concentration at a given sampling location.

## **REFERENCES**

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## **Appendix B**

### **Field Sampling Report**