Addendum No. 9 to the Work Plan for the RI/FS and IA for the Solid Wood Incorporated Site

Supplemental Sediment Bioassay Field Sampling and Analysis Plan

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ACRONYMS AND ABBREVIATIONS

Acronym	Explanation
bgs	below ground surface
City	City of Olympia
Ecology	Washington State Department of Ecology
FSAP	Field Sampling and Analysis Plan
GPS	Global Positioning System
HCID	Hydrocarbon Identification
IA	Interim Action
MDL	Method Detection Limit
mg/kg	Milligrams per kilogram
MTCA	Model Toxics Control Act
NAD	North American Datum
PQL	Practical Quantitation Limit
PSEP	Puget Sound Estuary Program
QC	Quality Control
RI/FS	Remedial Investigation/Feasibility Study
Site	Solid Wood Incorporated Site
SMS	Sediment Management Standards
TPH	Total Petroleum Hydrocarbons
TPH-D	TPH-Diesel Fraction
TPH-HO	TPH-Heavy Oil Fraction
WAC	Washington Administrative Code

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SECTION 1 – INTRODUCTION

A remedial investigation/feasibility study (RI/FS) and interim action (IA) are being conducted at the Solid Wood Incorporated Site (Site; see Figure 1) in Olympia, Washington. The RI/FS and IA are being conducted under Agreed Order No. DE-08-TCPSR-5415 between the City of Olympia (City) and the Washington State Department of Ecology (Ecology) to investigate the nature and extent of Site contamination and to aid in the development of cleanup actions at the Site (Parametrix 2008).

Sediment investigations have been conducted at the Site since 2008 in support of the RI/FS and IA. Concentrations of total petroleum hydrocarbon (TPH) in the heavy oil fraction (TPH-HO) collected during the most recent sampling event (April 2014) exceeded the Ecology-derived screening level of 100 milligrams per kilogram (mg/kg) for surface sediment at some locations. Consequently, further characterization of TPH in Site sediment was required by Ecology.

This Supplemental Sediment Bioassay Field Sampling and Analysis Plan (FSAP) is Addendum No. 9 to the Work Plan for the RI/FS and IA for the Solid Wood Incorporated Site (Parametrix 2008). The purpose of this FSAP is to present specific procedures for the collection and analysis of sediment bioassay samples at the Site to further characterize TPH contamination in sediment.

1.1 Background

Previous sediment investigations, conducted from 2008 to 2011 as part of the RI/FS and IA, characterized concentrations of (1) Sediment Management Standards (SMS) constituents² and (2) TPHs in beach sediment adjacent to the upland area (Parametrix 2008, 2010, 2011a, 2011b). Constituent concentrations were below applicable SMS chemical criteria (i.e., WAC 173-204-320 or WAC 173-204-520); however, concentrations of the TPH in the diesel fraction (TPH-D) and TPH-HO exceeded the Ecology-derived screening level of 100 milligrams per kilogram (mg/kg) for surface sediment. The sediment characterization also included chemical testing and three bioassays: 1) a 10-day amphipod solid phase survival test using *Eohaustorius estuarius*, 2) a sediment larval development test using *Mytilus galloprovincialis*, and 3) a 20-day polychaete solid phase survival and growth test using *Neanthes arenaceodentata*. One sample location (SD-30), where one field duplicate was also collected, failed one of the three bioassays (the sediment larval test) that was performed at this location (Figure 2; Parametrix 2011a). Three additional samples were collected at a later date from the SD-30 location and one of those samples (SD-33C) failed the sediment larval test as well (Figure 2; Parametrix 2011b).

Based on these results, the City was required by Ecology to conduct a subsequent sampling event to further characterize TPH-HO/TPH-D concentrations in sediment. The sampling event was completed in April 2014 and consisted of collecting 27 samples from 14 sample locations within a focus area that was identified based on previous screening level exceedances and failed sediment bioassays (i.e., SD-30, SD-30-DUP, and SD-33C; see Figure 2). Two samples were collected from 13 of the sample locations; one

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¹ Surface sediment (i.e., the biologically active zone) in Puget Sound is typically defined as the uppermost 10 centimeters of sediment. The biologically-active zone can be deeper at sites if receptors (e.g., geoduck [*Panopea generosa*], ghost shrimp [*Callianassa californiensis*]) are present at the site (Ecology 2013).

² See Table 1 of Washington Administrative Code [WAC] 173-204-320.

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was collected from 0-0.5 feet bgs and the other was collected from 2-3 feet bgs. Only sediment from 0-0.5 feet bgs was collected from the 14th location (SD-55). Concentrations of TPH-HO exceeded Ecology's screening level of 100 mg/kg in surface sediment (0-0.5 feet bgs) at multiple locations. TPH-D was not detected in any sample (PIONEER 2014).

1.2 Physical Location

The Site is approximately 17 acres in size and is located at 700 West Bay Drive NW in Olympia, Washington on the West Bay of Budd Inlet (Figure 1). Historically, the Site was used for milling purposes. The most recent commercial/industrial business on the Site was a lumber yard (Solid Wood, Incorporated), which closed in 2002. The Site is currently vacant and a portion has been converted into West Bay Park. Historical railroad tracks run the entire length of the Site (Parametrix 2008).

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SECTION 2 – FIELD SAMPLING PLAN

2.1 Sample Locations

A preliminary surface sediment TPH-HO delineation boundary was identified based on all previously-collected TPH-HO surficial sediment sample results (see Figure 4). Four sample locations within the TPH-HO delineation boundary will undergo further sediment characterization, which will include both chemical and biological testing. Three of the locations (SD-56, SD-57, and SD-58) are spaced evenly between the northern and southern borders of the TPH-HO delineation boundary, and are located near samples that exceeded the Ecology-derived 100 mg/kg screening level. The fourth location (SD-59) is located near SD-55, which was the easternmost sediment sample, and will help to further delineate the eastern edge of contamination.

2.2 Sampling Methods

Sediment samples will be collected during low tide in order to ensure the sample locations are exposed. Any recent surficial gravel will be removed from the sampling location prior to sample collection. All sediment samples will be discrete, and two gallons of sediment will be collected from the top 10 centimeters of soil (i.e., the biologically-active zone). Sediment samples will be collected with a trowel and placed in a stainless steel bowl. The samples will be mixed in the stainless steel bowl prior to being placed in an eight ounce amber jar for chemical testing and a sediment bag with no head space for biological testing. Any unrepresentative material (e.g., large woody debris, shells, and rocks) will be removed at the discretion of the sampler. The trowel and stainless steel bowl will be decontaminated between each sample using Alconox and deionized water.

At each sampling location, descriptions of the sediment will be recorded on the sampler's field note form (Attachment 1). Descriptions will include density, color, consistency, odor, organic matter, shell or wood debris, biological activity, presence of staining or sheens, and any other distinguishing characteristics or features.

The location of each sample will also be recorded using a handheld Global Positioning System (GPS) device in the North American Datum (NAD) 1983 State Plane Washington South FIPS 4602 (US feet) coordinate system.

2.3 Sample Handling

All samples will be placed in a cooler and held at approximately 4 degrees Celsius until they are received by the project laboratories. Chain-of-custody procedures outlined in the work plan will be followed (Parametrix 2008). Upon sample receipt, the laboratories will comply with the storage temperatures and maximum holding times required for the analyses to be performed (Table 1).

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Section 3 – Laboratory Analysis Plan

Chemical analysis will be performed by Anatek Laboratories of Moscow, Idaho. Biological testing will be performed by Environ Global (previously NewFields) in Port Gamble, Washington.

3.1 Chemical Analysis

Sediment samples will be analyzed for TPHs (TPH-HO, TPH-D, and TPH-HCID [hydrocarbon identification]), grain size, total solids, total sulfides, ammonia, total volatile solids, and total organic carbon. Applicable analytical methods, holding times, and practical quantitation limits (PQLs) are presented in Table 1.

Anatek Laboratories will be responsible for conducting laboratory quality control (QC) procedures and reporting laboratory QC results in accordance with laboratory standard operating procedures. The laboratory will perform and report the following laboratory QC once per batch of samples:

- method blank;
- blank spike;
- · matrix spike; and
- matrix spike duplicate.

3.2 Biological Testing

Biological tests will consist of the following bioassays:

- a 10-day amphipod solid phase survival test using *Eohaustorius estuarius*,
- a sediment larval development test using Mytilus galloprovincialis, and
- a 20-day polychaete solid phase survival and growth test using *Neanthes arenaceodentata*.

Bioassays will be conducted using the protocol in the Puget Sound Estuary Program's (PSEP) guidelines (PSEP 1995).

3.2.1 Biological Sample Preparation

Bioassay testing will commence within seven days of receipt of the sediment samples by Environ Global. The samples will be sieved using a 4 millimeter sieve in order to remove large rocks and pebbles that are too large for the test chambers and could potentially impact the survival of the organisms. Additionally, previous testing indicated that ammonia build-up from shells and decaying macro-invertebrates (e.g., mussels and clams) can be a potential contributor to negative biological effects. Therefore, the samples will also be sieved to remove any shells and macro-invertebrates. Chemical testing (see Section 3.2) will be performed prior to the bioassays. If there are elevated sulfide or ammonia levels, the sediment samples will be allowed to acclimate to test conditions for seven to 14 days prior to the addition of test organisms.³

³ The acclimation period allows for the natural biodegradation of ammonia and sulfide. Ammonia and sulfide concentrations will be monitored until they are below threshold levels, at which time the test will be initiated.



3.2.2 Biological Test Design

Each sample will be replicated five times and placed into separate test chambers. Two additional test chambers will be used. One chamber will be sacrificed at test initiation to measure pore water and initial overlying and interstitial (when applicable) ammonia and sulfide concentrations. The other chamber will be used for measuring daily water quality parameters, as well as pore water and overlying and interstitial (when applicable) ammonia and sulfide concentrations at test termination (see Table 2).

Due to the potential presence of polycyclic aromatic hydrocarbons (PAHs), the test chambers will be uncovered and placed under an ultraviolet light for 14-16 hours per day during the entire duration of the study.⁴ The ultraviolet light will be placed 12 inches above the sediment surface.

3.2.3 Biological Testing Quality Control

Numerous QC methods will be utilized in order to ensure that the bioassays are running properly.

3.2.3.1 Water Quality

Overlying and interstitial (when applicable) sulfide and ammonia concentrations will be analyzed prior to and at the termination of the biological tests. Sulfides will be measured using a spectrophotometer and ammonia will be monitored using an ammonium ion-specific probe. Additionally, dissolved oxygen, salinity, pH, and temperature will be monitored using water-quality probes prior to the biological tests, as well as daily. Acceptable ranges for these parameters are listed in Table 2.

3.2.3.2 Reference Samples

Reference samples will be collected by Environ Global within three days of Site sediment sampling event. Two reference samples will be collected in the Carr Inlet or Sequim Bay using a boat and a Van Veen grab sampler, and locations will be chosen based on grain size. The grain size of the reference samples should be similar to the grain sizes (+/- 5 percent) found in Site sediment samples. Reference samples will be analyzed for the same constituents as Site sediment samples and used for biological tests as well.

3.2.3.3 Controls

Two controls will be implemented for each of the bioassays. The first control will be a negative control, which will utilize negative sediment (i.e., sediment free from contaminants) and the test organisms. The negative control will provide evidence regarding the health of the organisms as well as any laboratory issues. The second control will be a positive control, which will utilize negative sediment (i.e., sediment free from contaminants) and a reference toxicant specific to each biological test (e.g., ammonium chloride, cadmium chloride). The positive control exposes test populations to known toxicants in order to calculate an LC_{50}^{5} for the populations.

⁴ Organisms can experience higher toxicity if exposed to certain PAHs and ultraviolet light (Ecology 2013). Ecology recommends that sediments collected from shallow water or an intertidal area be exposed to ultraviolet light during bioassays to account for this interaction (Ecology 2008).

⁵ An LC₅₀ is the concentration at which 50% of the population has died.



3.2.4 Identified Modifications to PSEP Methods

During previous bioassays, Environ Global, the Army Corps of Engineers, and Ecology identified modified test methods that increased the data quality of PSEP methods. These modifications apply to the sediment larval development test and the 20-day polychaete solid phase survival and growth test, and introduce additional steps in the post-processing of the test samples.

3.2.4.1 Polychaete Ash-Free Dry Weight Modification

The purpose of this modification was to account for the weight of sediment contained in the gut of the worms during the drying process. Worms reared under similar conditions and life history, but exposed to different grain size sediment, may express significantly different dry weights due to the contribution of heavier gut material of the worms maintained in sandy (heavier particles) sediment. This discrepancy in dry weight has the potential to lead to Type II errors, where significant differences are found between test treatments, when none actually exist. The procedure described here is a tool to estimate the actual contribution of gut content to the overall weight of the animals. A procedure defined as "ashing" is employed to heat the worm tissue at high temperatures until all that is left behind is solid inorganic material. At the termination of the 20 day survival and growth test, sediment from each test chamber is sieved through a 0.5 millimeter screen and all recovered polychaetes transferred into a plastic cup. Survival is recorded and worms are rinsed with deionized water and placed in pre-ashed, pre-weighed aluminum boats and dried in a gravimetric oven at 60° Celsius for at least 24 hours. Each weigh boat is removed from the oven, cooled in a desiccator for approximately 30 minutes, and then weighed on an analytical microbalance to 0.01 mg. Each weigh boat is then dried in a muffle furnace heated to 550° Celsius for 2 hours in order to determine ashed weights. The ashed boats are again weighed to 0.01 milligrams and the ashed weight was subtracted from the dry weight to calculate the ash-free dry weight. Both the dry weight and the ash-free dry weight are used to determine individual worm weight and growth rates.

3.2.4.2 Larval Resuspension and Enumeration

The purpose of this modification was to address a phenomenon observed in the larval development test where the developing larvae have the potential to become buried or incorporated among the fine silt or flocculent material that settles in the test chamber as an artifact of the test procedure. The standard procedure of decanting and sub-sampling the test chambers (which contains the larvae) may underestimate the number of animals remaining at test termination (a critical endpoint for results interpretation). The test is terminated approximately 48 to 96 hours after initiation (test and species dependent), when 90% of the control larvae have achieved the appropriate life stage. At termination, the overlying seawater of each replicate test chamber (containing 18 grams of sediment and 900 milliliters of seawater) is decanted into a clean 1 liter jar (leaving the sediment behind) and homogenized with a perforated plunger. From this container, a 10 milliliter subsample is transferred to a shell vial and preserved in 5% buffered formalin. Larvae are subsequently stained with a solution of Rose Bengal to enhance visualization of larvae. The numbers of normal and abnormal larvae are enumerated using an inverted microscope. Following the sampling of larvae for the standard PSEP protocol, the sediment in the test chamber will be re-suspended by pouring the decanted water back into the test chamber, mixing

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for 30 seconds with a perforated plunger, and allowing it to settle for approximately 12 hours. This action of pouring the decanted liquid back into the test chamber (containing the remaining sediment) and mixing has been found to be a useful technique in releasing larvae from the sediment matrix that were otherwise unaccounted for under the standard procedure. A second subsample is then collected as described above. At this point there are two sets of sub-samples that may require microscope enumeration. The decision to enumerate the re-suspended subsamples can be based on the results of the standard test samples.



SECTION 4 – DATA ANALYSIS AND REPORTING

The sediment sampling results will be documented in a report that will present the results of the investigation in text, tables and figures. The purpose of the report will be to (1) evaluate bioassay results using SMS criteria and (2) compare surficial sediment concentrations to Ecology's 100 mg/kg TPH screening level. Alternative methods for characterizing the TPH data (that are consistent with the SMS and MTCA) may also be presented.

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SECTION 5 – REFERENCES

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SEPTEMBER 2014 SECTION 5 – REFERENCES

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Tables

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Table 1: Sample Storage Temperatures, Maximum Holding Times and Analytical Limits for Sediment Sample Analyses

Constituent	Method	Storage Temperature	Maximum Holding Time	PQL (mg/kg dry weight)
TPH-D (mg/kg) ¹	NWTPH-DX	4°C	14 days	25
TPH-HO (mg/kg) ¹	NWTPH-DX	4°C	14 days	50
TPH-HCID (mg/kg)	NWTPH-HCID	4°C	14 days	20 for gasoline 50 for diesel 100 for heavy oil
Grain Size	Modified ASTM D-422/PSEP	4°C	6 months	N/A
Total Volatile Solids	PSEP	4°C	7 days	0.1
Total Solids	PSEP	4°C	14 days	0.1
Total Organic Carbon	EPA 9060	4°C	14 days	0.1
Ammonia (mg/kg)	Plumb 1981	4°C	7 Days	1.0
Total Sulfides (mg/kg)	Plumb 1981	4°C (1 N zinc acetate)	7 Days	1.0
Biological Tests	SMS Standards	4°C	7 days	N/A

Notes

N/A: Not Applicable

ASTM: American Society for Testing and Materials

¹ These samples will be prepped via silica gel cleanup prior to analysis.



Table 2: Target Parameters for Bioassays

Bioassay	Dissolved Oxygen (mg/L)	рН	Temperature (°C)	Salinity (%)
10-Day amphipod solid phase survival test	≥5	7.8 ±0.5	15±1	15±1
Sediment larval development test	≥4.8	7.8 ±0.5	16±1	28±1
20-Day polychaete solid phase survival and growth test	≥5.5	7.8 ±0.5	20±1	28±2

Figures

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Site Location

Addendum No. 9 to the Work Plan for the RI/FS and IA for the Solid Wood Incorporated Site

Olympia, Washington

Figure 1

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2008 and 2011 TPH and Bioassay Results Addendum No. 9 to the Work Plan for the RI/FS and IA for the Solid Wood Incorporated Site Olympia, Washington

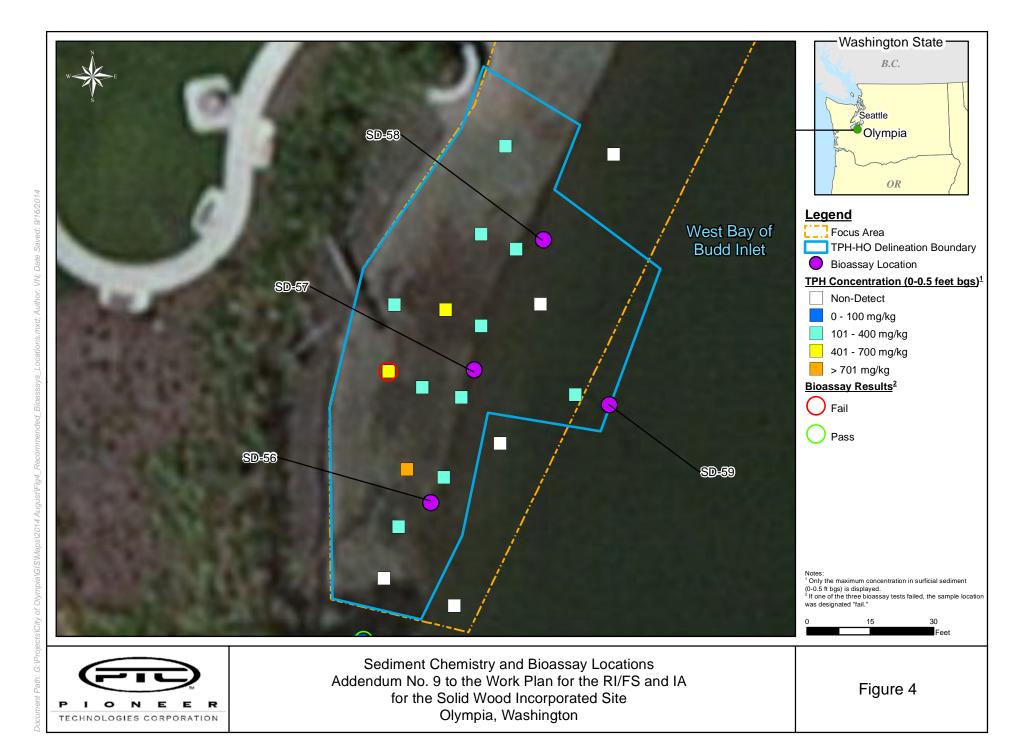
Figure 2

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Olympia, Washington

TECHNOLOGIES CORPORATION

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Attachment 1

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PIONEER DAILY FIELD REPORT

Date:	Site Location:		Site Arrival Time:	Site Γ	Departure Time :	
			One Annyal Time.		one beparture time	
WEATHER	Clear Sun	Overcast	Drizzle	Rain	Snow	
TEMPERATURE	To 32	32-50	50-70	70-85	85 Up	
WIND	Calm	Med.	Strong	Severe		
2						
PEOPLE PRESENT O	N-SITE	NAME	ASSOCIATION	7	TIME ON-SITE AND OFF-SITE	
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