# Supplemental Work Plan
# Tru-Grit Site
# 1110 Alexander Avenue
# Tacoma, Washington

January 25, 2018

Prepared for

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# Supplemental Work Plan
# Tru-Grit Site
# 1110 Alexander Avenue
# Tacoma, Washington

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| | |
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| Project Coordinator: | Juliann Cooley |

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# LIST OF ABBREVIATIONS AND ACRONYMS

| | |
|---|---|
| CanAm | CanAm Minerals, Inc. |
| cm | centimeter |
| Ecology | Washington State Department of Ecology |
| EIM | Electronic Information Management |
| EPA | US Environmental Protection Agency |
| FS | feasibility study |
| ft | feet/foot |
| LAI | Landau Associates, Inc. |
| m | meter |
| µm | micrometer |
| mg/kg | milligrams per kilometer |
| mL | milliliter |
| Port | Port of Tacoma |
| PSEP | Puget Sound Estuary Program Guidance |
| PSL | preliminary screening level |
| QA | quality assurance |
| QC | quality control |
| RI | remedial investigation |
| SAP | sampling and analysis plan |
| SCUM | Sediment Cleanup Users Manual |
| SMS | Sediment Cleanup Users Manual |
| Tru-Grit | Tru-Grit Abrasives, Inc. |
| WAC | Washington Administrative Code |

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# 1.0 INTRODUCTION AND BACKGROUND INFORMATION

This document presents a Supplemental Work Plan to address a data gap identified in the Remedial Investigation (RI) report prepared for the CanAm Minerals, Inc. (CanAm) Tru-Grit Abrasives, Inc. (Tru-Grit) Site located at 1110 Alexander Avenue adjacent to the Blair Waterway in Tacoma, Washington (Figure 1). Landau Associates, Inc. (LAI) prepared an RI report (LAI 2014) under Agreed Order (No. DE-8978). The RI report identified several data gaps. CanAm subsequently sent a letter (CanAm 2016) to Washington State Department of Ecology (Ecology) proposing a plan to fill data gaps. Ecology responded to the letter with approval for the additional work (Ecology 2017). The data collected during implementation of this work plan will be included in the final RI report. This Supplemental Work Plan was prepared consistent with the requirements of Washington Administrative Code (WAC) 173-340-820, the Sediment Management Standards (SMS), WAC 173-204 (Ecology 2013), and the Sediment Cleanup User's Manual II (SCUM II; Ecology 2015b).

Can-Am operates the Tru-Grit facility. The facility is a handling, storage, and distribution facility of a granular abrasive material used in composite roofing and sandblast grit. Can Am leases the property where the facility is located, which is owned by the Port of Tacoma (Port). For the purposes of this document, the granular material is referred to as grit or grit material. Handling of the grit material included receipt by barge; offloading via a ship-to-shore conveyance system; and storage, processing, and packaging for resale. The offloading operation was conducted starting in 1990 and continued until offloading at the facility ceased in 2013. The upland portion of the site is still used for some processing and packaging operations. As indicated in the RI report, grit material was released to the Blair Waterway during the course of Tru-Grit operations and appears to have resulted in copper and zinc concentrations exceeding preliminary screening levels (PSLs) in surface sediment at the Site, located in the vicinity of the ship-to-shore offloading conveyance system (Figure 2). However, the potential bioavailability of elevated copper and zinc concentrations in surface sediment has not been fully evaluated. Arsenic is also present at concentrations above PSLs, but is not found in grit material offloaded and processed at this facility and is not considered an impact related to operations at the Tru-Grit facility. This Supplemental Work Plan provides a detailed approach to evaluating the potential bioavailability of the copper and zinc identified in surface sediments in the RI report.

As part of the RI, soil and groundwater in the Site uplands were also evaluated. The Site is primarily paved, but a narrow strip of vegetated land is present just above the shoreline. Elevated copper and zinc concentrations were identified in the unpaved portion of the site in areas of Tru Grit operations; however, copper and zinc concentrations may also be the result of and/or augmented by former activities at the site, which included boat building and maintenance (both associated with releases of copper and zinc). Dissolved metals concentrations in samples collected from seeps along the shoreline exceeded PSLs for copper. The seep samples exhibited elevated conductivity indicating they are representative of a mixture of marine water and groundwater. Copper concentrations in marine water may be elevated in areas of heavy boat usage due to the presence of copper bottom paints on marine vessels. The influence of marine water and the Site's former history as a boat yard confound interpretation of the groundwater data and the soil to groundwater pathway. CanAm proposes to conduct leachate testing on raw grit material to determine the leaching potential of the material that was handled at the site. This data will be used to evaluate whether the material handled at the site has the potential to leach into groundwater. A sample of marine water (surface water) will also be collected and analyzed for dissolved copper to evaluate potential contribution to groundwater concentrations from marine water. Sampling and analysis procedures for leachate testing and surface water analysis are further described in this Supplemental Work Plan.

The scope of work presented in this Supplemental Work Plan includes surface sediment sampling, bioassay testing, raw material leachate testing, surface water testing, and reporting of results. In order to compare current chemical data with bioassay testing results at each sample location, chemical analysis will also be performed at each location where bioassay testing samples are collected.

# 2.0 SAMPLE LOCATIONS

Sample locations of grit material, surface water, and sediment are described below.

## 2.1 Grit Material Sample Location

Three samples of raw, unprocessed grit material will be collected from separate areas of the storage piles for leaching test purposes. The raw material will be collected from stockpiled material that originated from the same location in Anyox, British Columbia as the material that was released to the waterway. Recent chemical testing of the grit material for total metals in 2016 indicates that the concentrations of metal in the grit material has remained consistent over time (CanAm 2016). Raw grit material is currently stored inside a building at a nearby property on Taylor Way. A sample of the grit material will be collected from the storage pile inside the building.

## 2.2 Surface Water Sample Location

Two surface water samples will be collected from the sampling vessel one approximately 60 feet (ft) from the base of the riprap on the shoreline and another approximately 120 ft from the base of the riprap on the shoreline. Samples will be collected from a depth of approximately 1 meter (m) below the water surface.

## 2.3 Sediment Sample Locations

Sediment samples will be collected to evaluate the potential bioavailability of elevated copper and zinc concentrations resulting from potential Tru-Grit operations in surface sediment. On-site sample locations were selected from existing RI sample locations that best represent sediment potentially impacted by Tru-Grit's operations, as indicated by data collected during the RI. Indications of potential Tru-Grit impact include the following:

- Proximity to ship-to-shore offloading conveyance system and west of the offloading area
- High percentages of grit material in surface sediments
- Elevated concentrations of copper and/or zinc in surface sediments
- Relatively lower concentrations of arsenic in surface sediments.

Reference sediment samples will also be collected for quality assurance (QA) and quality control (QC) purposes for the bioassay testing; these sample locations were selected in accordance with sediment bioassay reference area guidelines in Puget Sound Estuary Program Guidance (PSEP; Ecology 2015a).

### 2.3.1 Bioassay/Chemical Testing Site Sample Locations

Based on the criteria listed above, on-site sediment sample locations will be collected from locations G-5, SG-14, SG-16, and SG-17. These locations are shown on both Figure 2 and Figure 3, and data collected during the RI indicate the greatest potential impact by Tru-Grit operations at these locations. Table 1 includes coordinates for the proposed sample locations.

As shown on these figures, the proposed sample locations are all near the former ship-to shore conveyance system location or the approximate location of the barge and sediment at these locations contain elevated grit percentages. Both G-5 and SG-14 are located directly below the former ship-to-shore offloading conveyance system; G-5 is below the transition point between a conveyor belt and a hopper and SG-14 is located at the transition point from the barge to the conveyor belt; SG-16 and SG-17 are located northwest of the former approximate barge location. Observed grit percentages at these locations were relatively high in the surface sediment; greater than 15 percent grit at G-5, SG-16, and SG-17, and between 5 percent and 15 percent grit at SG-14 (Figure 3).

Analytical data shown on Figure 2 indicate elevated concentrations of copper (ranging from 200 to 1,390 milligrams per kilogram [mg/kg]) and zinc (ranging from 650 mg/kg to 3,400 mg/kg) at G-5, SG-14, SG-16, and SG-17. Concentrations of arsenic were also below the SMS sediment cleanup objective (ranging from 6.6 mg/kg to 31 mg/kg), though arsenic concentrations at SG-17 are significantly greater (31 mg/kg) than at other proposed locations. Based on existing analytical data of grit material utilized by Tru-Grit at the site, elevated copper and zinc concentrations are consistent with impacts directly related to Tru-Grit operations at the site; elevated arsenic concentrations (principally at SG-17) may be the result of sediment impacts from sources other than Tru-Grit.

### 2.3.2 Bioassay Reference Sample Location

Reference samples to be used during bioassay testing will be collected from Carr Inlet, which meets sediment chemistry, sediment bioassay, and general habitat criteria for reference sample location selection (Ecology 2015a). Reference samples collected from Carr Inlet will be processed using field grain size sieving procedures (further discussed in Section 3.2.2) to select the appropriate reference sediments to be utilized during bioassay testing.

# 3.0 FIELD INVESTIGATION PROCEDURES

This section presents investigation methods and procedures to be used in the field that specifically apply to the sediment bioassay investigation and collection of raw grit material. This information is intended to be supplemental to procedures outline in the RI Work plan (LAI 2013) and the Sampling and Analysis Plan (SAP; Appendix A).

## 3.1 Sediment Bioassay Field Procedures

General sediment sampling field investigation procedures, including shallow sediment sample collection procedures, decontamination, and sampling documentation and handling, will be conducted in accordance with the existing RI/FS Work Plan and SAP.

### 3.1.1 Station Positioning Methods

The objective of the station positioning is to accurately establish and record the positions of all sampling locations within ±2 m (6.56 ft). The northing and easting coordinates of the proposed sediment sampling station locations in State Plane Coordinates are provided in Table 1. Station locations will be surveyed in the field using a Trimble NT300D differential GPS or equivalent with the use of a known survey control point. Sampling station coordinates will be reported relative to the North American Datum of 1983 (NAD83). Planned sampling location coordinates (Table 1) will be entered into the sampling vessel's onboard GPS unit.

### 3.1.2 Sediment Sample Collection

Sediment samples will be collected from on-site and reference sample locations for toxicity (bioassay) testing and chemical analytical testing. Sediment surface grab samples will be collected with a pneumatic power grab type sampler. Once the adequacy of the grab sample has been established according to the procedures outlined in the SAP (Appendix A), overlying water will be removed with a siphon and the top 10 centimeters (cm) of the sediment sample will be transferred to a clean, stainless steel bowl for homogenization using a stainless steel spoon. Approximately 10 liters (L) of sediment are required for bioassay testing, chemical analysis, and sieve analysis. If less than 10L of sediment are obtained in the initial grab, a second grab will be collected immediately adjacent (not overlapping but less than 5 ft) to the initial grab. The additional sample volume will be added to the clean container and homogenized with the initial grab sample before segregating aliquots for the bioassay testing, chemical analysis, and sieve analysis. As described in the SAP, the sediment will be characterized in the field at the time of sample collection. Field characterization will include determining color, odor, sheen, approximate grain size, Unified Soil Classification System (USCS) soil classification, and a determination of whether grit material is present, and if so, the approximate percentage of grit material. Sample collection procedures are further described in the following sections.

#### 3.1.2.1 Sediment Sample Collection for Chemical Testing

Sample collection procedures for chemical analytical testing will be consistent with procedures utilized during RI sampling; these procedures are included in the SAP (Appendix A).

#### 3.1.2.2 Sediment Sample Collection for Bioassay Toxicity Testing

Samples for bioassay toxicity testing, including reference samples, will be collected from the upper 10 cm using the procedures described in the SAP (Appendix A) for collection of surface sediment for chemical analysis. A minimum sample volume of 4L is required for each bioassay sample; twice the necessary volume of material will be collected at each location and the excess material will be archived. The sample aliquot for bioassay testing will be transferred to a clean bucket and sealed for transport to the lab.

Field grain size wet sieving will be conducted at the time of sampling to determine the fines content of each sample for the purpose of selecting an appropriate reference sediment samples to utilize during bioassay testing. This process separates the sediment sample into size fractions greater than 62.5 micrometers (µm; i.e., sand and gravel) and less than 62.5 µm (i.e., silt and clay) for classification of sand and silt/clay fractions. This process helps determine appropriate reference stations with similar grain size fractions (by volume) during field operations. This procedure requires a 62.5-µm sieve, a funnel with a diameter slightly greater than that of the sieve frame, a 100-milliliter (mL) graduated cylinder, a squirt bottle, a supply of distilled water, and a bowl for collecting rinse water. Procedures for field grain size sieving are as follows:

- Place a 62.5 µm (4-phi or 0.0025-inch mesh or #230 mesh size) sieve in a funnel with a bowl underneath.
- Moisten the sieve using a light spray of distilled water.
- Place exactly 50 mL of sample in the 100-mL graduated cylinder, add 20 to 30 mL of distilled water, and stir to fluidize the sample.
- Pour the sample into the sieve and thoroughly rinse any residue from the 100-mL graduated cylinder and stir into the sieve.
- Wash the sediment onto the sieve with distilled water using a water pick or squirt bottle having low water pressure.
- Continue wet sieving until only clear water passes through the sieve. This is accomplished by sieving an appropriate sample quantity (i.e., a sample volume that is not too large) and by efficient use of rinse water. Both of these techniques may require experimentation before routine wet sieving is started. Upon completion of sieving, carefully return the contents (i.e., sand and gravel fraction) of the sieve to the 100-mL graduated cylinder.
- Tap the graduated cylinder gently to settle the solid material.
- Read the volume of solid material from the scale on the side of the graduated cylinder and record the value. The fraction of sample with grain size greater than 62.5 µm is the ratio of the volume of material retained in the sieve to the original volume (50 mL).

### 3.1.3 Sample Containers, Preservation, and Storage

Samples submitted to the laboratory for chemical and toxicity analysis will be placed in the appropriate sample container provided by the laboratory. The samples will be preserved by cooling to a temperature of 4 degrees Celsius or frozen as required by the analytical method. Maximum holding and extraction times until analysis will be strictly adhered to by field personnel and the analytical laboratory. Analytical methods, sample containers, and holding times for chemical and toxicity testing to be performed are provided in Table 2.

### 3.1.4 Management of Residual Wastes

Excess sediment generated during sediment sampling will be returned to the water at the station where it was collected.

## 3.2 Raw Material Sampling Procedure

Sampling of raw grit material will be conducted using hand implements. Stockpiled material is expected to be easily accessible. The sample will be collected using a decontaminated stainless steel spoon. Approximately equal amounts of material will be collected from three separate locations on the pile and placed into a clean, laboratory-supplied, sampling container. The container will be labeled, placed under chain-of-custody protocol, and transported to the laboratory in accordance with the procedures outlined in the SAP (Appendix A).

## 3.3 Surface Water Sampling Procedure

Surface water samples for dissolved copper will be collected using a peristaltic pump and dedicated sample tubing. Tubing will be connected to the peristaltic pump and will be extended to the recommended sampling depth (1m) by tying it to a pole. Sample tubing will be fitted with an inline 0.45 micron filter to remove suspended particles. Samples will be collected directly into laboratory provided, appropriately preserved sample containers.

# 4.0 LABORATORY ANALYTICAL METHODS

Surface sediment samples collected from on-site locations will undergo chemical and toxicity testing, and the reference sample will undergo toxicity testing only. The proposed analyses are presented in the following sections.

Additionally, the raw grit material will undergo leaching tests. The leaching test consists of subjecting the sample to leaching conditions and analyzing the leachate. Leaching test procedures are described in Section 4.3.

## 4.1 Sediment Chemical Analysis

Surface sediment samples G-5, SG-14, SG-16, and SG-17 will be analyzed for the full SMS list of chemical analyses, which include metals, semi-volatiles, total organic carbon (TOC), grain size, and polychlorinated biphenyls. A full list of proposed chemical analyses is presented on Table 2, and the associated sample preparation methods, cleanup methods, and practical quantitation limits are included in Table A-3 of the SAP (Appendix A).

## 4.2 Toxicity (Bioassay) Testing

Surface sediment samples G-5, SG-14, SG-16, and SG-17 will undergo toxicity tests (bioassay), including two acute effects tests and one chronic effects tests. Also, a reference sample will be analyzed during the toxicity tests. Concurrent tests on reference sediment are conducted to control for possible sediment grain size effects on bioassay organisms. Reference sediment samples will have a grain size that is similar to the project sediment sample used for toxicity testing (the percent fines for the reference sample will be within 20 percent of the project sample percent fines). As described in Section 2.2, the reference samples will be collected from Carr Inlet.

Three sediment toxicity tests (bioassay) will be conducted on each sample:

- Acute 10-day amphipod mortality
- Acute larval mortality/abnormality
- Chronic 20-day juvenile polychaete growth rate.

Toxicity testing will be completed in compliance with the procedures and QA/QC performance standards described in the PSEP (Ecology 2015a) as revised by subsequent agency-approved updates and as described in Chapter 4 and Appendix C of SCUM II (Ecology 2015b). A brief summary of each test is presented in sections 4.2.1, 4.2.2, and 4.2.3; specific bioassay analysis procedures are presented in Appendix B.

### 4.2.1 Acute Effects Test: Amphipod

The acute 10-day amphipod mortality test evaluates the survival of benthic-dwelling amphipods exposed to project sediment. The test will be conducted using adult amphipods (*Ampelisca abdita* or

*Eohaustorius estuarius*). The species will be selected by the lab based on the interstitial water salinity (greater than or equal to [≥] 25 parts per trillion and percentage of fine-grain sediments (≥60 percent fines or <60 percent fines) as recommended by Ecology in SCUM II (Ecology 2015b). If the project sample contains greater than 60 percent fines, the selected species will be *Ampelisca abdita*, if the sample contains less than 60 percent fines, the selected species will be *Eohaustorius estuaries*.

### 4.2.2 Acute Effects Test: Larval

The acute larval mortality/abnormality test assesses larval embryo development while being exposed to project sediment. The test will be conducted using either a mollusk or echinoderm species [e.g. *Mytilus galloprovincialis* (blue mussel) or *Crassostrea gigas* (pacific oyster); or, if mollusks are not available, *Dendraster excentricus* (sand dollar), the *Strongylocentrotus purpuratus* (purple sea urchin) or *Strongylocentrotus droebachiensis* (green sea urchin)]. Modifications to the acute larval mortality/abnormality test were conducted using these species. Selection of an appropriate test species for the acute larval mortality/abnormality is dependent on the seasonal availability of adult organisms that can produce viable gametes. Consequently, for this project, the laboratory will select the best available echinoderm larvae, during the week preceding delivery of the initial sediment samples. For termination of the bivalve development test, the resuspension method will be employed to liberate any larvae trapped in fine sediments or the flocculent layer at the sediment/water interface.

### 4.2.3 Chronic Effects Test: Juvenile Polychaete

A chronic effects test will be conducted using the 20-day juvenile polychaete survival and growth rate. The test is conducted using a species of juvenile polychaete worms (*Neanthes arenoceodentat*). The test measures survival and mean individual growth rate expressed as milligrams per individual per day while exposed to project sediment. Prior to conducting this test, the lab will verify that interstitial salinity is greater than 20 percent. Both dry weight and ash-free dry weight (AFDW) values will be measured at the test termination for evaluation of growth and survival rates.

## 4.3 Leaching Test Procedures and Surface Water Analytical Procedures

The raw grit sample will be subjected to synthetic precipitation leaching procedure (SPLP) by using US Environmental Protection Agency (EPA) SW-846 Method 1312. This method is designed to simulate exposure to precipitation by exposing the sample to liquid with a similar pH to rainfall. The liquid (leachate) is then extracted and analyzed. The extraction fluid to be utilized during this test will be water with a pH of approximately 5, which is intended to simulate the pH of acidified rainfall. Leachate will be analyzed for dissolved copper by EPA Method 6020. Surface water samples will also be analyzed for dissolved copper by EPA Method 6020. Reporting limits for EPA Method 6020, which is the most sensitive method available from the analytical laboratory, may exceed the project screening level for dissolved copper of 2.4 microgram per liter; thus, results will be reported down to the method detection limit if needed. Results between the reporting limit and method detection limit will be flagged as estimated values.

# 5.0 QUALITY ASSURANCE/QUALITY CONTROL

This section describes both field and laboratory QA/QC procedures and provides a description of the data quality review that will be performed on the analytical and biological test results. Implementation of these procedures in conjunction with the sample collection and handling procedures described in Section 3.0 will provide a reasonable degree of confidence in the project data. QA/QC procedures for leaching test methods are outlined in the SAP (Appendix A).

## 5.1 Quality Assurance and Quality Control for Bioassay Analyses

Bioassay toxicity testing will be completed in compliance with the QA/QC performance standards described in PSEP (Ecology 2015a) as revised by subsequent agency-approved updates and as described in SCUM II (Ecology 2015b). QC procedures will include negative controls, positive controls, reference sediment sample analysis, laboratory replicates, and water quality measurement before, during, and after testing. Specific QA/QC requirements for bioassay analysis are included in Appendix B.

## 5.2 Quality Assurance and Quality Control for Chemical Analyses

Field and laboratory control samples that will be used for QC purposes during chemical analysis will include blind field duplicates, laboratory matrix spikes, laboratory matrix spike duplicates, laboratory duplicates, laboratory method blanks, laboratory control samples, and surrogate spikes. One blind field duplicate of sediment will be collected and submitted for chemical analysis for this sampling event. Additionally, one blind field duplicate of marine surface water will be collected and submitted for chemical analysis for this sampling event. Specific requirements for each QA/QC sample are included in the SAP (Appendix A).

## 5.3 Data Quality Evaluation

Data quality evaluation will be performed on chemical analytical results, both sediment chemical analysis and leaching test results. Data quality evaluation will also be performed by the bioassay lab. Data quality procedures for chemical and bioassay testing are described in the following sections.

### 5.3.1 Chemical Analysis Data Quality

An internal data quality evaluation will be conducted on all chemical sample data to determine acceptability of analytical results. Data quality evaluation will be conducted with guidance from applicable portions of EPA's National Functional Guidelines for Organic Data Review (EPA 1999, 2008) and the National Functional Guidelines for Inorganic Data Review (EPA 2004, 2010). The verification and validation check for each laboratory data package includes the following:

- Package contains all necessary documentation:
    - Chain-of-custody records

   - Identification of samples received by the laboratory
   - Date and time of receipt of the samples at the laboratory
   - Sample conditions upon receipt at the laboratory
   - Date and time of sample analysis
   - If applicable, date of extraction, definition of laboratory data qualifiers, all sample-related QC data, and QC acceptance criteria
- Verification that all requested analyses, special cleanups, and special handling methods were performed
- Evaluation of sample holding times
- Evaluation of QC data compared to acceptance criteria, including method blanks, surrogate recoveries, matrix spike results, laboratory duplicate and/or replicate results, and laboratory control sample results (Appendix A).

Data validation qualifiers are added to the sample results, as appropriate, based on the verification and validation check. The absence of a data qualifier indicates that the reported result is acceptable without qualification. Data qualification arising from data validation activities will be described in the data validation report, rather than in individual corrective action reports. Care will be taken by the laboratory to, where possible, not use method detection limits and instead use practical quantitation limits (also called reporting limits) in accordance with SCUM II (Ecology 2015b).

#### 5.3.1.1 Laboratory Reports

A written report will be prepared by the analytical laboratory documenting all the activities associated with sample analyses. At a minimum, the following will be included in the report:

- Results of the laboratory analyses and QA/QC results
- Methods used during sample preparation, extraction, and analyses
- Chain-of-custody procedures, including explanation of any deviation from those identified herein
- Any protocol deviations from this SAP (Appendix A)
- Location and availability of the data
- Batch identification for each analysis method
- Digestion/extraction/analysis dates for each QA/QC parameter corresponding to each batch definition (i.e., all QA/QC data will be batch-specific)
- A case narrative.

As appropriate, this SAP (Appendix A) may be referenced in describing protocols.

### 5.3.2 Additional Bioassay Data Evaluation

Bioassay testing performance will be evaluated against bioassay data reporting requirements. This evaluation is further described in Appendix B.

## 5.4 Data Management Procedures

All laboratory analytical results, including QC data, will be submitted to LAI. Following validation of the data, any qualifiers will be added to the project database. All field data will be entered into an Excel spreadsheet and verified to determine all entered data is correct and without omissions and errors. Following data validation, analytical results will be formatted electronically and uploaded to Ecology's Environmental Information Management (EIM) system.

# 6.0 DATA EVALUATION AND REPORTING

Following receipt of final supplemental RI chemical and bioassay data, either an updated RI report or RI report addendum will be submitted to Ecology within 90 days. The report will describe field activities, presentation and evaluation of chemical and bioassay data, and conclusions regarding whether chemical concentrations detected in the sediment are toxic to benthic organisms and the leaching potential of the raw grit material in regards to its potential to impact groundwater.

If the bioassay results indicate that surface sediment does adversely impact benthic organisms, the bioassay results will override chemical analysis results and releases of grit material to sediment related to Tru-Grit operations will not be considered a hazard to the environment.

Results of leaching tests will be evaluated along with surface water results and groundwater sample results obtained during the previous phase of the RI. Data will be evaluated to determine the potential for grit material to adversely impact groundwater at the site and potential contribution of concentrations in marine water.

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Data Source: Esri 2012

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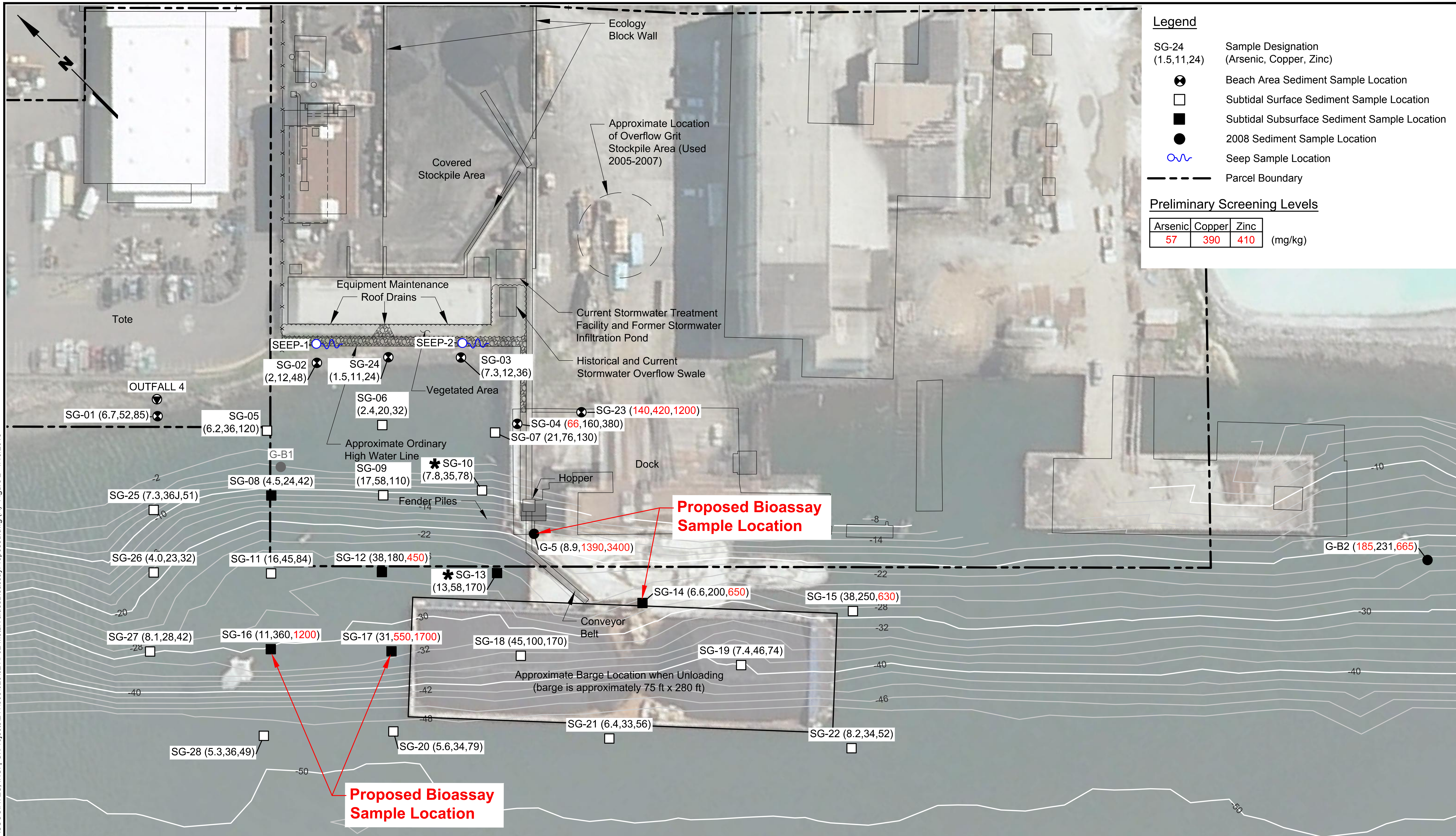
| Tru-Grit Site<br>Tacoma, Washington | **Vicinity Map** | Figure<br>1 |
|---|---|---|




LANDAU ASSOCIATES, INC. | G:\Projects\241\008\020\021\F02 MetalValuesBioassayProposed.dwg (A) "Figure 2" 1/19/2018

Source: Imagery ©Google Earth Pro 2010; Aerial dated 04-30-2009

**Notes**

1. * Full SMS list analyzed at these locations.
2. Preliminary screening level, equivalent to SMS sediment cleanup objective (SCO) protective of the benthic community.
3. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

0 60 120
Scale in Feet

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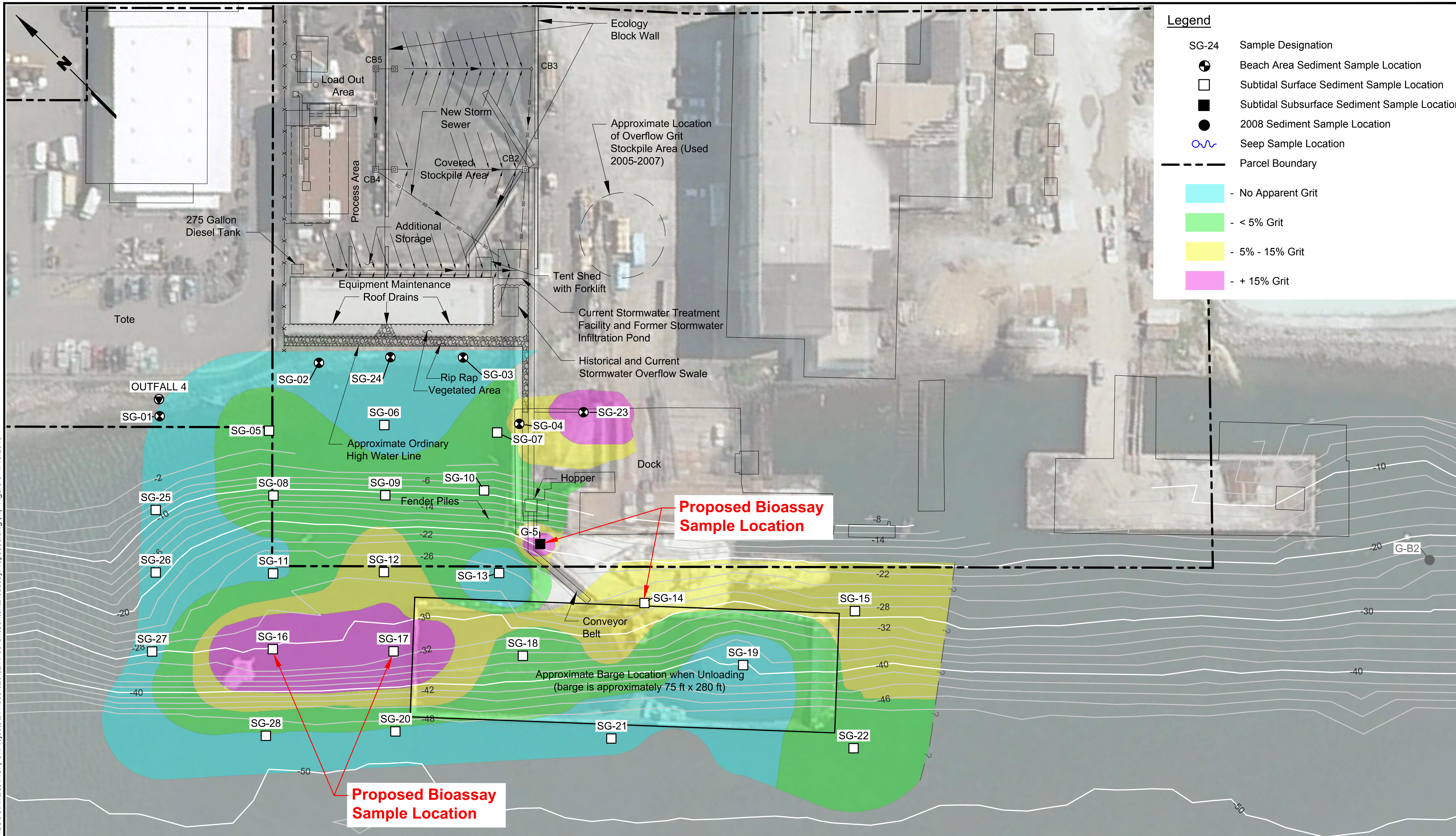
Tru-Grit Site
Tacoma, Washington

**Metals Concentrations in Surface Sediments and Proposed Bioassay Sample Locations**

Figure 2




Source: Imagery ©Google Earth Pro 2010; Aerial dated 04-30-2009

Notes
1. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

0 60 120
Scale in Feet

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Tru-Grit Site
Tacoma, Washington

**Grit Content in Surface Sediments and Proposed Bioassay Sample Locations**

Figure 3

**Table 1**
**Proposed Sampling Locations**
**Tru-Grit Site**
**Tacoma, Washington**

| Location | Easting (a) | Northing (a) |
|---|---|---|
| SG-14 | 1168635 | 712475 |
| SG-16 | 1168442 | 712620 |
| SG-17 | 1168497 | 712565 |
| G-5 | 1168613 | 712549 |

**Note:**
(a) Washington State Plane Cordinate System North American Datum of 1983

# Table 2
## Sample Size, Containers, and Analytical Methods
## Tru-Grit Site
## Tacoma, Washington

| Sample Type | | Method | Container | Preservation | Maximum Holding Time |
|---|---|---|---|---|---|
| Sediment | Total Metals (As, Cd, Cr, Cu, Pb, Ni, Ag, Zn) | 6020A LL | Two 4 oz-WMG with teflon-lined lid | Cool, 4$^o$C | 6 months |
| | SMS Mercury (CVAA) | 7471A | | Cool, 4$^o$C | 28 days for mercury |
| | SMS Semivolatiles- low level | 8270D LL | | Cool, 4$^o$C | 14 days (a), 1 year (b) |
| | SMS PCBs- low level | 8280A LL | | Cool, 4$^o$C | 14 days (a), 1 year (b) |
| | PSEP TOC | 9060 | | Cool, 4$^o$C | 28 days, 6 months (b) |
| | Grain Size | Plumb 1981 | 16 oz-WMG with teflon-lined lid | Cool, 4$^o$C | n/a |
| | Archive | n/a | Two 8 oz-WMG with teflon-lined lid | Freeze | 1 year |
| | Bioassay | n/a | Eight 32 oz-Bioassay sediment bags or equivalent | Cool, 4$^o$C | 56 days |
| Sediment Splits (Port of Tacoma) | n/a | n/a | Two 8 oz-WMG with teflon-lined lid | Cool, 4$^o$C | n/a |
| Marine Water | Dissolved metals (Cu)- low level [field filtered] | 6020A LL | 250 mL Plastic w/nitric acid | Cool, 4$^o$C | 6 months |
| Solids (Grit) | Dissolved Copper | 1312 SPLP extraction w/6020A LL-dissolved (lab field filter) | 8 oz-WMG with teflon-lined lid | Cool, 4$^o$C | 6 months |

**Notes:**
(a) Holding time shown is from sample collection to extraction; holding time from extraction to analysis is 40 days.
(b) Holding time shown is from sample collection to extraction if sample is frozen.

**Abbreviations/Acronyms:**
$^o$ C = degrees Celsius
EPA = U.S. Environmental Protection Agency
oz = ounces
PCBs = polychlorinated biphenyls
TOC= total organic carbon
WMG = wide mouth glass

APPENDIX A

# Tru-Grit Sampling and Analysis Plan

# Sampling and Analysis Plan
# Remedial Investigation / Feasibility Study
# Work Plan
# Tru-Grit Facility
# Tacoma, Washington

February 6, 2013

Prepared for

**CanAm Minerals, Inc.**
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# 1.0 INTRODUCTION

This sampling and analysis plan (SAP) describes the procedures for conducting field activities during the remedial investigation (RI) and feasibility study (FS) at the Tru-Grit Facility (facility), located at 1110 Alexander Avenue and bordering the Blair Waterway in Tacoma, Washington. This SAP is Appendix A to the main document: *Site Work Plan, Remedial Investigation/Feasibility Study, Tru-Grit Facility, 1110 Alexander Avenue Tacoma, Washington* (Work Plan; Landau Associates 2012). The primary objective of this SAP is to provide sampling, sample handling, and analytical testing methodologies consistent with accepted procedures such that the data collected will be adequate for using in characterizing sediment and upland conditions.

This SAP was prepared consistent with the requirements of the Washington Administrative Code (WAC) Chapter 173-340-820, the Sediment Management Standards (SMS; WAC 173-204; Ecology 1995), and the Sediment Sampling and Analysis Plan Appendix (Ecology 2008). This SAP provides field, sampling, and analytical procedures to be use during the RI.

# 2.0 OBJECTIVES AND DESIGN OF INVESTIGATION

This section establishes the objectives of the sediment and upland investigation, gives a brief overview of the design of the investigation, and provides an explanation of chemical analyses that will be performed as part of this investigation.

## 2.1 OBJECTIVE

As noted in Section 1.0, the goal of the RI is to determine the lateral and vertical extent of contamination at the facility and to determine if there are ongoing sources of sediment contamination in upland areas. This information will be used to develop, evaluate, and select appropriate cleanup actions in the FS.

## 2.2 SAMPLING STANDARDS

Sampling will be undertaken to address the analytical requirements set forth in the SMS (WAC 173-204). The current Washington State Department of Ecology (Ecology) guidance document *Sediment Sampling and Analysis Plan Appendix* (Ecology 2008) was used to develop this SAP and to ensure that SMS requirements are achieved. To address the SMS requirements, a subset of the sediment samples collected during this investigation will be analyzed for the full suite of 47 SMS chemicals and the additional conventional sediment variables. The remaining sediment and soil samples will be analyzed for copper, zinc, and arsenic to delineate the lateral and vertical extent of contamination.

Sediment sample results are compared against two sets of criteria. The marine sediment quality standard (SQS; WAC 173-204-320) and the sediment cleanup screening level criteria (CSL; WAC 173-204-520). The SQS is the concentration below which effects to biological resources and human health are unlikely, where as the CSL is the concentration above which more than minor adverse biological effects may be expected. Sediment criteria are presented in Table 7 of the Work Plan.

Upland soil samples will be compared against preliminary screening levels (PSLs) presented in Table 6 of the work plan. PSLs were developed in accordance with MTCA (WAC 173-340).

## 2.3 OVERALL SAMPLING DESIGN

As noted in the Work Plan, previous investigation at the facility determined that the area directly under and near the ship-to-shore conveyance system at the Tru-Grit facility contained raw grit material (Landau 2009). At the southwestern end of the ship-to-shore conveyance system, the grit material was present in a sufficient quantity to increase the copper and zinc concentrations in the sediment to levels above the CSL in one location (G-5). Results of the previous investigation were indicative of localized impacts at the site resulting from release of grit material to the waterway but also of area-wide impact to sediment from past industrial operations at multiple facilities. Although arsenic is not a contaminant of concern at the site, sampling for arsenic will be conducted to differentiate site contamination from area-wide impacts. In accordance with the results of the previous investigation, as well as the main goal of the current RI, the following three areas of concern have been identified:

- The upland area
- The intertidal (beach) area
- The subtidal waterway.

## 2.4 SAMPLE STATION LOCATIONS

Sampling locations are shown on Figure A-1 and A-2, and include 5 upland area soil samples, 4 beach sediment samples, and 20 sediment grab samples and cores in the subtidal waterway (including background samples). Sample locations were chosen based on information from the previous sediment investigation and possible contaminant sources and pathways.

# 3.0 FIELD INVESTIGATION PROCEDURES

Planned field investigation activities associated with the RI/FS include collecting surface samples from the upland and beach areas and collection of surface grab and sediment core samples from the subtidal waterway.

## 3.1 SITE RECONNAISSANCE AND UTILITY LOCATE

A site reconnaissance and utility locate will be conducted prior to investigation activities to identify underwater obstructions (e.g., underwater cables) to planned sediment coring, sediment grab samples, or anchoring required to accomplish these activities. If necessary, exploration locations will be relocated to accommodate obstructions.

## 3.2 UPLAND AND BEACH INVESTIGATION

Within the beach and the upland areas, discrete soil samples will be collected. Of these samples, six will come from within the beach area and six samples from within the upland area. Sample locations are identified in Figure A-1. Sample locations will be identified in the field using a hand-held global positioning system (GPS); GPS coordinates for each sample location are provided in Table A-1. Field coordinates will be recorded on the sample collection form or in the GPS unit. At each discrete sampling location, a soil punch, hand corer, hand auger or other hand implement will be used to excavate soil to the appropriate maximum sampling depth. The maximum sampling depth for upland soil is 6 inches. The maximum sampling depth for surface sediment grab samples is 10 centimeters (cm). Individual soil and sediment samples will be collected from large enough areas to ensure that adequate sample volume is collected. Using a decontaminated stainless steel trowel, any vegetation will first be removed from the sample area. All soil or sediment within the sample area, to a depth of 6 inches or 10 cm, respectively, will be removed and placed in a stainless steel bowl. Thorough homogenization in the bowl, with a stainless steel spoon, will ensure sample representativeness. The corer, spoons, and steel tape will be thoroughly decontaminated before each use by an Alconox® wash and scrub followed by a deionized water rinse. Wash and rinse water will be disposed of on the ground at least 10 ft from any sample location.

Two groundwater samples will be collected from seeps along the shoreline. Numerous seeps were noted along the shoreline at low tide during a September 17, 2012 site visit. Final determination of the location of the two samples will be made in the field during sample collection. Samples will be collected at a tide elevation of +2.5 ft or lower.

Groundwater samples will be collected using a peristaltic pump and will be field-filtered using a 0.45 micron filter and preserved with nitric acid in accordance with dissolved metals sampling protocols (EPA 2000). Dedicated tubing will be used for each sample, thus decontamination of equipment will not be necessary. Field parameters including pH, temperature, dissolved oxygen, oxygen reduction potential, and turbidity will be measured during sample collection. Seep water will also be monitored for conductivity, to evaluate if seep water represents upland groundwater discharge, or seawater that has infiltrated the upland area during higher tide cycles.

## 3.3 SEDIMENT INVESTIGATION

Sampling locations will be identified using a GPS or differentially corrected global positioning system (DGPS), if available. Actual sample coordinates will be recorded on the sample collection forms or in the GPS unit. Table A-1 presents a list of sample locations, their GPS coordinates, and approximate depth to the mudline.

### 3.3.1 SHALLOW SEDIMENT INVESTIGATION

Surface grab sediment samples will be collected with a stainless steel power-grab sediment sampler. The sampler will be decontaminated between grab samples. Once collected, overlying water will be removed with a siphon. Sediment samples will be collected and homogenized only if the following conditions are met:

- Water above the sediment has equal or less turbidity than ambient water conditions. Excess turbidity is indicative of sample disturbance during collection.
- The power-grab sampler is not overfilled, resulting in the loss of finer grained sediment. If the sampler is overfilled, weights will be removed, and the sample attempted from a new, nearby [within 10 feet (ft)], location.
- The sediment sampler did not close incompletely due to obstructions (e.g., woody debris), resulting in erosion or disturbance of the sediment surface.
- The sampler experiences complete penetration. If this does not occur, weights will be added to the sampler and the sample attempted from a new, nearby (within 10 ft), location. If three attempts fail to reach the penetration depth, the final sample will be collected and the conditions noted in the field log.

Only shallow sediment grabs that meet the above standards will be collected. Prior to collection and homogenization, a photograph will be taken of the full power-grab sampler that includes a scale and the sample name and date. Sediment will be characterized in the field sample collection form. This characterization will include color, odor, sheen, grain size, and a soil description consistent with the Unified Soil Classification System (USCS). To determine if grit is present, approximately 1 ounce of sediment will be rinsed through an ASTM International No. 200 sieve to remove fines. Because grit material is processed to remove significant fines prior to transport by barge to the Tru-Grit facility, further sieving is unlikely to remove significant grit material. Sediment remaining in the sieve after rinsing will be placed under a microscope or hand lens for identification of grit material within the sample.

Sediment will be collected from the benthic zone (top 10 cm of the sediment) from an area large enough to ensure adequate sample volume and excluding portions that are touching the power-grab sampler. This collected sediment will exclude large, unrepresentative material (e.g., shells, woody debris). Sediment will be homogenized in decontaminated stainless steel bowls, using a decontaminated stainless steel spoon. After sufficient homogenization, sediment will be placed into laboratory supplied containers and placed on ice. Unused sediment will be contained for later disposal.

### 3.3.2 DEEP SEDIMENT INVESTIGATION

Deeper sediments will be collected using vibrocore sediment coring methods, using the following steps:

- Actual tide level will be confirmed at a surveyed location on or near the site at least three times during the sampling day, and compared to listed tides. If these are in exact agreement throughout the first day, this step can be eliminated during later sampling.
- Water depth will be measured using a weighted line.
- The actual mudline at the sample location will be calculated (this may be slightly different than the expected mudline at the sample location based on mapped bathymetry).
- A minimum penetration depth of 100 cm (~3.3 ft) will be achieved.
- Collected cores will be allowed to settle until the overlying water is almost clear and the recovery length will be measured.
- Cores that have over 75 percent recovery length will be saved and numerically decompacted during processing.
- Cores that have under 75 percent recovery length will be returned to the collection site and a new core will be collected in the nearby (within 10 ft) vicinity. If refusal is encountered prior to full penetration, the vibrocore will be repositioned and a new core will be attempted. If refusal is met during the third such attempt, this core will be collected and noted in the field log.
- Cores will be drained of overlying water, capped, and labeled with the sample number, date, and surface direction. Cores will be stored on ice overnight in a secure location, or will be transported to the lab for storage each night.

#### 3.3.2.1 Core processing

Core processing will be accomplished at the laboratory, after sample collection is complete. Length intervals to be sampled are discussed in Section 6.5of the work plan. Core processing will be accomplished using the following steps:

- Care will be taken to avoid spills and plastic sheeting will cover all laboratory surfaces during processing.
- Cores will be cut open lengthwise using sheet metal sheers, leaving the caps on the core to maintain core-tube integrity.
- The outer sediment surface (that was touching the liner) will be removed with a decontaminated stainless steel spoon and disposed of.
- The core will be split in half lengthwise, using a decontaminated stainless steel spatula.
- All interval measurements will be adjusted according to the percent retention (length of sediment sample retrieved/penetration depth of core tube) of the sediment collected within each individual core tube. For example, if 6 ft of sediment were retrieved from a core with a penetration depth of 8 ft, the retention ratio was 0.75 or 75 percent. The resulting intervals to be sampled would then be adjusted for 75 percent sediment retention.
- A photograph will be taken of the individual sediment layers that include the sample name, the layer depth range, processing date and original surface direction.
- Sediment will be characterized in the sample collection form. This characterization will include color, odor, grain size, and a soil description consistent with the USCS. This description will also include any oil sheen, biological activity (e.g., shells, worms, etc.), debris, or other distinguishing features and the depth at which these occur.
- Sediment will be collected from each individual layer, excluding sediment portions that are touching core walls, and homogenized in decontaminated stainless steel bowls using a decontaminated stainless steel spoon.
- An aliquot of sample from each layer will be placed under a microscope and examined for the presence of grit. The approximate percentage of grit in the sample will be noted in the field log. As needed, magnified photographs of the sediment may be taken to document the presence or absence of grit material.
- After sufficient homogenization, sediment will be placed into laboratory supplied containers and placed on ice. Enough volume (0.5 inch headspace) will be left in archived sample containers to accommodate expansion of the sample during freezing.

### 3.3.3 DECONTAMINATION

All field sampling equipment, including vibrocore heads, stainless steel bowls and spoons, and sample core tubes will be decontaminated in the following manner:

- Rinsed with clean site water
- Scrubbed with Alconox
- Rinsed with clean site water.

Equipment used during core processing at the laboratory will be rinsed with clean tap water initially, and the final rinse will be with de-ionized water.

### 3.3.4 LABELING

Sample identification will adhere to the following format: Sampling Type and location (e.g., U-1 for upland samples, SG-1 for sediment grab or BSG-1 for background sediment grab, SC-1 for sediment core, respectively), followed by the depth range for core samples [e.g., SC-1(2-3) for core sample one from a depth of between 2 and 3 ft).

### 3.3.5 SAMPLE CUSTODY

The primary objective of sample custody is to create an accurate, written record that can be used to trace the possession and handling of samples so that their quality and integrity can be maintained from collection until completion of all required analyses. Adequate sample custody will be achieved by means of approved field and analytical documentation. Such documentation includes the chain-of-custody record that is initially completed by the sampler and is, thereafter, signed by those individuals who accept custody of the sample. A sample is in custody if at least one of the following is true:

- It is in someone's physical possession
- It is in someone's view
- It is secured in a locked container or otherwise sealed so that tampering will be evident
- It is kept in a secured area, restricted to authorized personnel only.

Sample control and chain-of-custody in the field and during transportation to the laboratory will be conducted in general conformance with the procedures described below:

- As few persons as possible will handle samples.
- Sample bottles will be obtained new or pre-cleaned from the laboratory performing the analyses.
- The sample collector will be personally responsible for the completion of the chain-of-custody record and the care and custody of samples collected until they are transferred to another person or dispatched properly under chain-of-custody rules.
- The coolers in which the samples are transported will be accompanied by the chain-of-custody record identifying their contents. The original record and laboratory copy will accompany the shipment. The other copy will be forwarded to Landau Associates along with sample collection forms.

When samples are transferred, the individuals relinquishing and receiving the samples will sign the chain-of-custody form in the appropriate space and record the date and time of transfer.

A designated sample custodian at the laboratory will accept custody of the samples and certify that the sample identification numbers match those on the chain-of-custody record. The custodian will then enter sample identification number data into a log that will be maintained by the laboratory.

All documentation and other project records will be safeguarded to prevent loss, damage, or alteration. If an error is made on a document, corrections will be made by drawing a single line through the error and entering the correct information. The erroneous information will not be obliterated. Corrections will be initialed and dated and, if necessary, a footnote explaining the correction will be included. Errors will be corrected by the person who made the entry, whenever possible.

### 3.3.6 RESIDUAL WASTE MANAGEMENT

Contaminated sediments that are not immediately returned to the site location will be disposed in 55-gallon drums at a designated site, awaiting analytical results. Each drum will be labeled identifying the contents and the approximate extent that the drum is filled, the accumulation start date, and the explorations from which the sediment was generated. The client will assume responsibility of disposing of site waste appropriately.

# 4.0 QUALITY ASSURANCE AND QUALITY CONTROL

This section describes the quality assurance/quality control (QA/QC) procedures in support of the RI at the Tru-Grit facility. This section will establish the project team organization and responsibilities, the quality assurance objectives, laboratory analytical methods, QA/QC requirements and corrective actions for this project.

## 4.1 PROJECT TEAM ORGANIZATION AND RESPONSIBILITIES

The project team organizational structure was developed based on the requirements of the field and laboratory activities. The key positions/contractors and associated responsibilities are described below:

- **Consultant Project Manager:** Responsible for implementation of all aspects of the project plans. Specific responsibilities include review and approval of revisions to RI documentation, overseeing that all technical procedures are followed, reporting of deviations from the Ecology-approved project plans to the Ecology project coordinator and overseeing that data collected will satisfy the quality assurance objectives.
- **Ecology Project Coordinator:** Responsible for overseeing implementation of all aspects of the project plans. Specific responsibilities include review and approval of revisions to RI documentation, overseeing that all technical procedures are followed, and overseeing that data collected will satisfy the quality assurance objectives.
- **Analytical Laboratory:** Responsible for providing sample bottles, performing chemical analyses per the SAP and reporting of data as required by the SAP.
- **Field Sampling Personnel:** Responsible for implementing sampling procedures as specified in the project plans, notifying the consultant project manager of any deviations from the project plans.

## 4.2 QUALITY ASSURANCE OBJECTIVES

The Quality Assurance (QA) objectives for this project are to develop and implement procedures that will ensure collection of representative data of known, acceptable, and defensible quality. The data quality parameters used to assess the acceptability of the data are representativeness, comparability, precision, accuracy, bias, and completeness.

- Representativeness expresses the degree to which data accurately and precisely represent an actual condition or characteristic of a population. Representativeness can be achieved by selecting appropriate sampling locations and by using appropriate sampling methods.
- Comparability expresses the confidence with which one data set can be evaluated in relation to another data set. For this work, comparability of data will be established through the use of standard analytical methodologies with analytical limits of quantitation (LOQs) that can meet screening level criteria to the extent practicable and by utilizing standard reporting formats.

- Precision measures the reproducibility of measurements under a given set of conditions. Specifically, it is a quantitative measure of the variability of a group of measurements compared to their average values. Analytical precision is measured through matrix spike/matrix spike duplicate (MS/MSD) and/or through laboratory control sample/laboratory control sample duplicate (LCS/LCSD) for organic analysis and through laboratory duplicate samples for inorganic analyses. The quantitative relative percent difference (RPD) for laboratory duplicates, MS/MSD and field duplicates will be used to assess sampling and analytical precision.
- Accuracy is an expression of the degree to which a measured or computed value represents the true value. Field accuracy is controlled by adherence to sample collection procedures as outlined in this SAP.
- Bias is the systematic or persistent distortion of a measured process that causes errors in one direction. Bias of the laboratory results will be evaluated based on analysis of method blanks and matrix spike samples.
- Completeness is a measure of the proportion of data obtained from a task sampling plan that is determined to be valid. It is calculated as the number of valid data points divided by the total number of data points requested.

## 4.3 LABORATORY ANALYTICAL METHODS

Soil samples from the upland and beach areas will be analyzed for copper and zinc using U.S. Environmental Protection Agency (EPA) Method 6010. If groundwater samples are collected during a second phase of investigation, they will be analyzed for copper and zinc by EPA Method 6010. Two sediment samples will be analyzed for the full suite of 47 SMS chemicals and the additional conventional sediment variables. The remaining sediment and soil samples will be analyzed for copper, zinc, and arsenic to delineate the lateral and vertical extent of contamination. Sample containers, preservation, and holding times are provided in Table A-2. Sample analytical methods and practical quantitation limits for sediments are presented in Table A-3. Analytical methods and quantitation limit goals for upland soil and groundwater samples are provided in Table A-4.

## 4.4 FIELD AND LABORATORY QUALITY CONTROL PROCEDURES

Field and analytical laboratory quality control samples will be collected to evaluate data precision, accuracy, representativeness, completeness, bias, and comparability of the analytical results for the RI. The quality control samples and the frequency at which they will be collected and/or analyzed are described below. Acceptance criteria for laboratory quality control samples are presented in Table A-5 for sediments and in Table A-6 for soil and water.

#### 4.4.1 BLIND FIELD DUPLICATES

A blind field duplicate will be collected at a frequency of at least 1 per 20 sediment samples per chemical analysis and not less than one field duplicate. The blind field duplicate will consist of a split sample collected at a single sample location, after homogenization of the sample. Blind field duplicate sample results will be used to evaluate data precision. Acceptance criteria for blind field duplicate samples are 20% relative percent difference (RPD) for water and 35% RPD for soil and sediment.

#### 4.4.2 LABORATORY MATRIX SPIKE/ MATRIX SPIKE DUPLICATE

A minimum of one laboratory MS/MSD will be analyzed per 20 samples, or one MS/MSD sample per batch of samples if fewer than 20 samples will be performed for all analyses. These analyses will be performed to provide information on accuracy and to verify that extraction and concentration levels are acceptable. The laboratory spikes will follow EPA guidance for MS and MSDs.

#### 4.4.3 LABORATORY DUPLICATES

A minimum of one laboratory duplicate per 20 samples, or one laboratory duplicate sample per batch of samples if fewer than 20 samples are contained in a batch, will be analyzed for metals. These analyses will be performed to provide information on the precision of chemical analyses. The laboratory duplicate will follow EPA guidance in the method.

#### 4.4.4 LABORATORY METHOD BLANKS

A minimum of one laboratory method blank per 20 samples, one every 12 hours, or one per batch of samples analyzed (if fewer than 20 samples are contained in a batch) will be analyzed for all parameters to assess possible laboratory contamination. Dilution water will be used whenever possible. Method blanks will contain all reagents used for analysis. The generation and analysis of additional method, reagent, and glassware blanks may be necessary to verify that laboratory procedures do not contaminate samples.

#### 4.4.5 LABORATORY CONTROL SAMPLE

A minimum of one LCS per 20 samples, or one LCS per sample batch if fewer than 20 samples are contained in a batch, will be analyzed for all parameters.

#### 4.4.6 SURROGATE SPIKES

All project samples analyzed for organic compounds will be spiked with appropriate surrogate compounds as defined by the analytical methods.

## 4.5 LABORATORY QUALITY ASSURANCE/QUALITY CONTROL

QA/QC for chemical testing includes laboratory instrument and analytical method QA/QC. Instrument QA/QC monitors the performance of the instrument and method QA/QC monitors the performance of sample preparation procedures. The analytical laboratory will be responsible for instrument and method QA/QC. QA/QC procedures to be performed by the laboratory will be in accordance with methods specified in Table A-3.

When an instrument or method control limit is exceeded, the laboratory will contact the project manager immediately. The laboratory will be responsible for correcting the problem and will reanalyze the samples within the sample holding time if sample reanalysis is appropriate.

## 4.6 CORRECTIVE ACTIONS

Corrective actions will be needed for two categories of nonconformance:

- Deviations from the methods or QA requirements established in this document
- Equipment or analytical malfunctions.

Corrective action procedures to be implemented based on detection of unacceptable data are developed on a case-by-case basis. Such actions may include one or more of the following:

- Altering procedures in the field or laboratory
- Using a different batch of sample containers
- Performing an audit of field or laboratory procedures
- Reanalyzing samples (if holding times allow)
- Resampling and analyzing
- Evaluating sampling and analytical procedures to determine possible causes of the discrepancies
- Accepting the data without action, acknowledging the level of uncertainty
- Rejecting the data as unusable.

During field operations and sampling procedures, the field personnel will be responsible for conducting and reporting required corrective actions. A description of any action taken will be entered in the daily field notebook. The project manager will be consulted immediately if field conditions are such that conformance with this SAP is not possible.

During laboratory analysis, the laboratory QA officer will be responsible for taking required corrective actions in response to equipment malfunctions. If an analysis does not meet data quality objectives outlined, corrective action will follow the guidelines in the noted EPA analytical methods and the EPA guidelines for data validation for organics and inorganics analyses (EPA 1999, 2004). At a minimum, the laboratory will be responsible for monitoring the following:

- Calibration check compounds must be within performance criteria specified in the EPA method or corrective action must be taken prior to initiation of sample analysis. No analyses may be performed until these criteria are met.
- Before processing any samples, the analyst should demonstrate, through analysis of a reagent blank that interferences from the analytical system, glassware, and reagents are within acceptable limits. Each time a set of samples is extracted or there is a change in reagents, a reagent blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement steps.
- Method blanks should, in general, be below instrument detection limits. If contaminants are present, then the source of contamination must be investigated, corrective action taken and documented, and all samples associated with a contaminated blank reanalyzed. If, upon reanalysis, blanks do not meet these requirements, Landau Associates will be notified immediately to discuss whether analyses may proceed.
- Surrogate spike analysis must be within the specified range for recovery limits for each analytical method utilized or corrective action must be taken and documented. Corrective action includes: 1) reviewing calculations, 2) checking surrogate solutions, 3) checking internal standards, and 4) checking instrument performance. Subsequent action could include recalculating the data and/or reanalyzing the sample if any of the above checks reveal a problem. If the problem is determined to be caused by matrix interference, reanalysis may be waived if so directed following consultation with Landau Associates. If the problem cannot be corrected through reanalysis, the laboratory will notify Landau Associates prior to data submittal so that additional corrective action can be taken, if appropriate.
- If the recovery of a surrogate compound in the method blank is outside the recovery limits, the blank will be reanalyzed along with all samples associated with that blank. If the surrogate recovery is still outside the limits, Landau Associates will be notified immediately to discuss whether analyses may proceed.
- If quantitation limits or matrix spike control limits cannot be met for a sample, Landau Associates will be notified immediately to discuss corrective action required.
- If holding times are exceeded, all positive and undetected results may need to be qualified as estimated concentrations. If holding times are grossly exceeded, Landau Associates may determine the data to be unusable.

If analytical conditions are such that nonconformance is indicated, Landau Associates will be notified as soon as possible so that any additional corrective actions can be taken. The laboratory project manager will then document the corrective action by a memorandum submitted to Landau Associates. A narrative describing the anomaly; the steps taken to identify and correct the anomaly; and any recalculation, reanalyses, or re-extractions will be submitted with the data package in the form of a cover letter.

## 4.7 DATA VERIFICATION AND VALIDATION

All RI data will be verified and validated to determine the results are acceptable and meet the quality objectives. Prior to submitting a laboratory report, the laboratory will verify that all the data are consistent, correct, and complete, with no errors or omissions.

Validation of the data will be performed by Landau Associates following the guidelines in the appropriate sections of the EPA Contract Laboratory Program *National Functional Guidelines for Organic and Inorganic Data Review* (EPA 1999, 2004) and will include evaluations of the following:

- Chain-of-custody records
- Holding times
- Laboratory method blanks
- Surrogate recoveries
- Laboratory MS/MSD
- Blank spikes/LCS
- Laboratory duplicates
- Corrective action records
- Completeness
- Overall assessment of data quality.

In the event that a portion of the data is outside the data quality objective limits or the EPA guidance (EPA 1999, 2004), or sample collection and/or documentation practices are deficient, corrective action(s) will be initiated. Corrective action will be determined by the field coordinator and Landau Associates' QA officer in consultation with the Landau Associates project/task manager and may include any of the following:

- Rejection of the data and resampling
- Qualification of the data
- Modified field and/or laboratory procedures.

Data qualification arising from data validation activities will be described in the data validation report, rather than in individual corrective action reports.

## 4.8 DATA MANAGEMENT PROCEDURES

All laboratory analytical results, including QC data, will be submitted electronically to Landau Associates. Electronic format will include a scanned PDF of the original laboratory data package and comma separated value (CSV) files that will be downloaded directly to an Excel spreadsheet and/or to the project database.  The laboratory data package should include a case narrative along with analytical and quality control results. Following validation of the data, any qualifiers will be added to the Excel spreadsheets and project database.  All survey data will be provided electronically in a format that can be downloaded into an Excel spreadsheet.  All field data will be entered into an Excel spreadsheet and verified to determine all entered data is correct and without omissions and errors.  Following receipt of all RI data and all survey data, water level measurements, field parameters, and analytical results will be formatted electronically and uploaded to Ecology's Environmental Information Management (EIM) system.

# 5.0 HEALTH AND SAFETY

A project health and safety plan for implementation of field activities described in the Work Plan and this SAP is provided in Appendix C of the main document. All Landau Associates employees will follow the procedures described in this plan. Landau Associates subcontractors will either adopt this plan or prepare their own plan that is at least as protective as this plan.

# 6.0 REFERENCES

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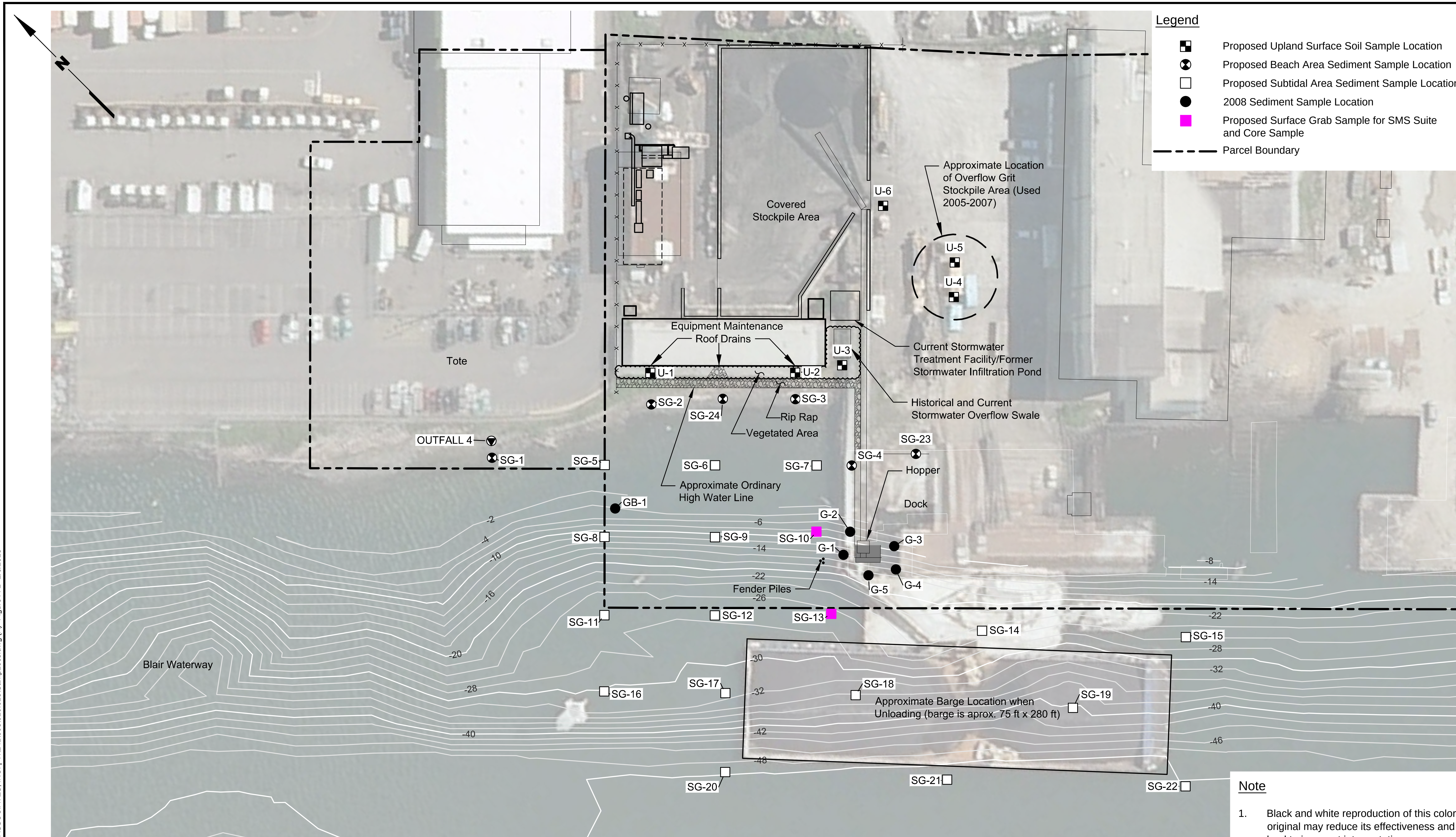


Source: Google Earth Pro 2010; Aerial dated 04-30-2009

0 60 120
Scale in Feet

LANDAU ASSOCIATES

Tru-Grit Facility
Tacoma, Washington

**Sampling Locations**

Figure
A-1

LANDAU ASSOCIATES, INC. | V:\241\008\010.013\SampLocs.dwg (A) "Figure A-1" 2/6/2013


Legend

Parcel Boundary Line

Background Grab Sample Location

Note

1. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

Source: Google Earth Pro 2010

0 300 600

Scale in Feet

LANDAU ASSOCIATES

Tru-Grit Facility
Tacoma, Washington

**Background Sample Locations**

Figure
A-2

# TABLE A-1
# SEDIMENT SAMPLE LOCATIONS AND ELEVATIONS
# TRU-GRIT FACILITY
# TACOMA, WASHINGTON

| Sample Location | Northing (a) | Easting (a) | Elevation (b) |
|---|---|---|---|
| SG-1 | 712778.7 | 1168493.7 | Beach |
| SG-2 | 712732.1 | 1168591.5 | Beach |
| SG-3 | 712670.1 | 1168660.7 | Beach |
| SG-4 | 712614.1 | 1168657.1 | Beach |
| SG-5 | 712724.9 | 1168542.8 | at or above MLLW |
| SG-6 | 712675.3 | 1168594.0 | at or above MLLW |
| SG-7 | 712629.7 | 1168641.0 | at or above MLLW |
| SG-8 | 712691.6 | 1168510.6 | at or above MLLW |
| SG-9 | 712642.0 | 1168561.7 | -11.1 |
| SG-10 | 712599.1 | 1168611.2 | -8.6 |
| SG-11 | 712655.4 | 1168475.5 | -27.0 |
| SG-12 | 712605.8 | 1168526.6 | -27.4 |
| SG-13 | 712554.5 | 1168581.3 | -26.5 |
| SG-14 | 712479.0 | 1168643.7 | -23.1 |
| SG-15 | 712384.8 | 1168735.3 | -25.7 |
| SG-16 | 712620.1 | 1168441.3 | -30.2 |
| SG-17 | 712565.1 | 1168496.7 | -31.6 |
| SG-18 | 712505.8 | 1168556.2 | -34.2 |
| SG-19 | 712402.6 | 1168651.2 | -40.2 |
| SG-20 | 712528.6 | 1168461.2 | -49.6 |
| SG-21 | 712425.6 | 1168560.7 | -51.0 |
| SG-22 | 712315.8 | 1168668.3 | -50.4 |
| SG-23 | 712590.7 | 1168692.1 | Beach |
| SG-24 | 712702.7 | 1168627.1 | Beach |
| BSG-1 | 712242.7 | 1168110.6 | -45.1 |
| BSG-2 | 713348.9 | 1167715.4 | -47.8 |
| G-5 | 712555.6 | 1168615.7 | -18.3 |
| U-1 | 712747.2 | 1168605.3 | aprox. 14 |
| U-2 | 712682.4 | 1168672.5 | aprox. 14 |
| U-3 | 712664.9 | 1168697.6 | aprox. 14 |
| U-4 | 712646.4 | 1168780.0 | aprox. 16 |
| U-5 | 712662.1 | 1168795.9 | aprox. 16 |
| U-6 | 712720.4 | 1168788.0 | aprox. 16 |

(a) Washington State Plane North Zone coordinate system [North America Datum (NAD) 83]
(b) Feet Mean Lower Low Water

# TABLE A-2
# SAMPLE SIZE, CONTAINERS, AND ANALYTICAL METHODS
# TRU-GRIT FACILITY
# TACOMA, WASHINGTON

| Sample Type | Container | Preservation | Maximum Holding Time |
|---|---|---|---|
| Metals | 8 oz - WMG with teflon-lined lid | Cool, 4° C | 6 months, 28 days for mercury |
| Volatiles | 2 oz - WMG with teflon-lined lid | Cool, 4° C | 14 days, 6 months (b) |
| Semivolatiles | 8 oz - WMG with teflon-lined lid | Cool, 4° C | 14 days (a), 1 year (b) |
| PCBs | 8 oz - WMG with teflon-lined lid | Cool, 4° C | 14 days (a), 1 year (b) |
| Pesticides | 8 oz - WMG with teflon-lined lid | Cool, 4° C | 14 days (a), 1 year (b) |
| TOC | 4 oz - WMG with teflon-lined lid | Cool, 4° C | 28 days, 6 months (b) |
| Total Solids | 4 oz - WMG with septa lid | Cool, 4° C | 14 days, 6 months (b) |

PCBs = Polychlorinated Biphenyls
TOC= Total Organic Carbon
WMG = Wide Mouth Glass
oz = ounces
° C = degrees Celsius
EPA = U.S. Environmental Protection Agency

(a) Holding time shown is from sample collection to extraction; holding time from extraction to analysis is 40 days.
(b) Holding time shown is from sample collection to extraction if sample is frozen.

# TABLE A-3
## RECOMMENDED SAMPLE PREPARATION METHODS, CLEANUP METHODS, ANALYTICAL METHODS, AND PRACTICAL QUANTITATION LIMITS FOR SEDIMENTS
## TRU-GRIT FACILITY
## TACOMA, WASHINGTON

| Chemical | Recommended Sample Preparation Methods (a) | Recommended Sample Cleanup Methods (b) | Recommended Analytical Methods (c) | Recommended PQLs (d,e) |
|---|---|---|---|---|
| **Metals** | | | | **(mg/kg dry weight)** |
| Arsenic | PSEP/3050B | -- | 6010B/6020/7061A | 19 |
| Cadmium | PSEP/3050B | -- | 6010B/6020/7131A | 1.7 |
| Chromium | PSEP/3050B | -- | 6010B/6020/7191 | 87 |
| Copper | PSEP/3050B | -- | 6010B/6020 | 130 |
| Lead | PSEP/3050B | -- | 6010B/6020 | 150 |
| Mercury | -- (f) | -- | 7471A/245.5 | 0.14 |
| Nickel | PSEP/3050B | | 6010B/6020 | 47 |
| Silver | PSEP/3050B | -- | 6010B/6020 | 2 |
| Zinc | PSEP/3050B | -- | 6010B/6020 | 137 |
| **Nonionizable Organic Compounds** | | | | **(µg/kg dry weight or as listed)** |
| **LPAH Compounds** | | | | |
| Naphthalene | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 700 |
| Acenaphthylene | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 433 |
| Acenaphthene | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 167 |
| Fluorene | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 180 |
| Phenanthrene | 3540C/3550B/3545 | 3640A/3660B | 8270/1625C | 500 |
| Anthracene | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 320 |
| 2-Methylnaphthalene | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 223 |
| **HPAH Compounds** | | | | |
| Fluoranthene | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 567 |
| Pyrene | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 867 |
| Benz[a]anthracene | 3540C/3550B/3545 | 3640A/3660B | 8270C (h) / 1625C | 433 |
| Chrysene | 3540C/3550B/3545 | 3640A/3660B | 8270C (h) / 1625C | 467 |
| Total benzofluoranthenes (g) | 3540C/3550B/3545 | 3640A/3660B | 8270C (h) / 1625C | 1067 |
| Benzo[a]pyrene | 3540C/3550B/3545 | 3640A/3660B | 8270C (h) / 1625C | 533 |
| Indeno[1,2,3-cd]pyrene | 3540C/3550B/3545 | 3640A/3660B | 8270C (h) / 1625C | 200 |
| Dibenz[a,h]anthracene | 3540C/3550B/3545 | 3640A/3660B | 8270C (h) / 1625C | 77 |
| Benzo[ghi]perylene | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 223 |
| **Chlorinated Benzenes** | | | | |
| 1,2-Dichlorobenzene | 3540C/3550B/3545 | 3640A/3660B | 8270C (h) / 1625C | 35 |
| 1,4-Dichlorobenzene | 3540C/3550B/3545 | 3640A/3660B | 8270C (h) / 1625C | 37 |
| 1,2,4-Trichlorobenzene | 3540C/3550B/3545 | 3640A/3660B | 8270C (h) / 1625C | 31 |
| Hexachlorobenzene | 3540C/3550B/3545 | 3640A/3660B | 8270C (h) / 1625C | 22 |
| **Phthalate Esters** | | | | |
| Dimethyl phthalate | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 24 |
| Diethyl phthalate | 3540C/3550B/3545 | 3640/A3660B | 8270C/1625C | 67 |
| Di-n-butyl phthalate | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 467 |
| Butyl benzyl phthalate | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 21 |
| Bis[2-ethylhexyl]phthalate | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 433 |
| Di-n-octyl phthalate | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 2067 |
| **Miscellaneous Extractable Compounds** | | | | **(µg/kg dry weight or as listed)** |
| Dibenzofuran | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 180 |
| Hexachlorobutadiene | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 11 |
| N-nitrosodiphenylamine | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 28 |
| PCBs | | | | |
| PCB Aroclors® | 3540/3550 | 3620B/3640A/3660B | 8082 | 6 |

# TABLE A-3
# RECOMMENDED SAMPLE PREPARATION METHODS, CLEANUP METHODS, ANALYTICAL METHODS, AND PRACTICAL QUANTITATION LIMITS FOR SEDIMENTS
# TRU-GRIT FACILITY
# TACOMA, WASHINGTON

| Chemical | Recommended Sample Preparation Methods (a) | Recommended Sample Cleanup Methods (b) | Recommended Analytical Methods (c) | Recommended PQLs (d,e) |
|---|---|---|---|---|
| **Volatile Organic Compounds** | | | | |
| Ionizable Organic Compounds | | | | |
| Phenol | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 140 |
| 2-Methylphenol | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 63 |
| 4-Methylphenol | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 223 |
| 2,4-Dimethylphenol | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 29 |
| Pentachlorophenol | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 120 |
| Benzyl alcohol | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 57 |
| Benzoic acid | 3540C/3550B/3545 | 3640A/3660B | 8270C/1625C | 217 |
| **Conventional Sediment Variables** | | | | |
| Grain size | -- (j) | -- | Plumb (1981) | 1% |
| TOC | -- (j) | -- | 9060 | 0.10% |

**Protocol, Appendix A (PSWQA, 1996) Krone 1998**

73 ug TBT/Kg Bulk
.05 ug TBT/L pour water
EPA - U.S. Environmental Protection Agency
GPC - gel permeation chromatography
HPAH - high molecular weight polycyclic aromatic hydrocarbon
LAET = Lowest Apparent Effects Threshold
LPAH - low molecular weight polycyclic aromatic hydrocarbon
NCASI = National Council for Air and Stream Improvement, Inc.
Ni = Nickel

PCB - polychlorinated biphenyl
PCDD = polychlorinated dibenzo-p-dioxins
PCDF = polychlorinated dibenzofurans
PQL = Practical Quantitation Limits
PSEP - Puget Sound Estuary Program
RA/FA =
Sb = Antimony
TOC - total organic carbon
VOC = Volatile Organic Compounds

mg/L = milligrams per liter
mL = milliliter
ng/kg = nanograms per kilogram
µg/kg - micrograms per kilogram

(a) Recommended sample preparation methods are:
- PSEP (1997a)
- Method 3050B and 3500 series - sample preparation methods from SW-846 (EPA 1996) and subjected to changes by EPA updates.

(b) Recommended sample cleanup methods are:
- Sample extracts subjected to GPC cleanup follow the procedures specified by EPA SW-846 Method 3640A. Special care should be used during GPC to minimize loss of analytes.
- If sulfur is present in the samples (as is common in most marine sediments), cleanup procedures specified by EPA SW-846 Method 3660B should be used.
- All PCB extracts should be subjected to sulfuric acid/permanganate cleanup as specified by EPA SW-846 Method 3665A.
- Additional cleanup procedures may be necessary on a sample-by-sample basis. Alternative cleanup procedures are described in PSEP (1997b) and EPA (1986).

(c) Recommended analytical methods are:
- Method 6000, 7000, 8000, and 9000 series - analytical methods from SW-846 (EPA 1986) and updates.
- The SW-846 and updates are available from the web site at: http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm.
- Method 1613 - analytical method from EPA-821/B-94-005 (EPA 1994c).
- Method 1624C/1625C - isotope dilution method.
- NCASI analytical methods.
- Plumb (1981) - EPA/U.S. Army Corps of Engineers Technical Report EPA/CE-81-1
- PSEP (1986).
- Acid volatile sulfide method for sediment (EPA 1991).

(d) To achieve the recommended PQLs for organic compounds, it may be necessary to use a larger sample size (approximately 100 g), a smaller final extract volume for gas chromatography/mass spectrometry analyses (0.5 mL), and one of the recommended sample cleanup methods, as necessary, to reduce interference, using different analytical methods with better sensitivity. Detection limits are on a dry-weight basis unless otherwise indicated. For sediment samples with low TOC, it may be necessary to achieve even lower detection limits for certain analytes in order to compare the TOC-normalized concentrations with applicable numerical criteria.

(e) The recommended PQLs are based on a value equal to one third of the 1988 dry weight LAET value (Barrick et al 1988) except for the following chemicals: 1,2-dichlorobenzene, 1,2,4-trichlorobenzene, hexachlorobenzene, hexachlorobutadiene, n-nitrosodiphenylamine, 2-methylphenol, 2,4-dimethylphenol, and benzyl alcohol, for which the recommended maximum detection limit is equal to the full value of the 1988 dry weight LAET.

(f) The sample digestion method for mercury is described in the analytical method (Method 7471A, September 1994).

(g) Total benzofluoranthenes represent the sum of the b, j, and k isomers.

(h) Selected ion monitoring may improve the sensitivity of method 8270C and is recommended in cases when detection limits must be lowered to human health criteria levels or when TOC levels elevate detection limits above ecological criteria levels. See PSEP organics chapter, appendix B–Guidance for Selected Ion Monitoring (PSEP 1997b).

(i) Sample preparation methods for VOCs analyses are described in the analytical methods.

(j) Sample preparation methods for sediment conventional analyses are described in the analytical methods.

# TABLE A-4
## QUANTITATION LIMIT GOALS FOR SOIL AND GROUNDWATER
## TRU-GRIT FACILITY
## TACOMA, WASHINGTON

| Analyte | Analytical Method (a) | SOIL Reporting Limits (b) | SOIL Units | WATER Reporting Limits (b) | WATER Units |
|---|---|---|---|---|---|
| **METALS** | | | | | |
| Copper | EPA-6010 | ND(<0.2) | mg/kg | ND(<2.0) | µg/L |
| Zinc | EPA-6010 | ND(<0.6) | mg/kg | ND(<10) | µg/L |

ND = Not Detected.

(a) Analytical methods are from SW-846 (EPA 1986) and updates, unless otherwise noted.
(b) Reporting limit goals are based on current laboratory data and may be modified during the investigation process as methodology is refined. Laboratory reporting will be based on the lowest standard on the calibration curve. Instances may arise where high sample concentrations, nonhomogeneity of samples, or matrix interferences preclude achieving the desired reporting limits.

# TABLE A-5
# LABORATORY QC SAMPLE ACCEPTANCE CRITERIA
# SEDIMENT

| Analyte Description | CAS Number/ Method | LCSREC - Recovery Low | LCSREC - Recovery High | LCSREC - Units | LCSRPD - Precision | LCSRPD - Units | MSREC - Recovery Low | MSREC - Recovery High | MSREC - Units | MSRPD - Precision | MSRPD - Units | SUREC - Recovery Low | SUREC - Recovery High | SUREC - Units |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Metals (ICP)** | **6010B** | | | | | | | | | | | | | |
| Arsenic | 7440-38-2 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % | -- | -- | -- |
| Cadmium | 7440-43-9 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % | -- | -- | -- |
| Chromium | 7440-47-3 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % | -- | -- | -- |
| Copper | 7440-50-8 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % | -- | -- | -- |
| Lead | 7439-92-1 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % | -- | -- | -- |
| Silver | 7440-22-4 | 75 | 120 | % | 20 | % | 75 | 120 | % | 20 | % | -- | -- | -- |
| Zinc | 7440-66-6 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % | -- | -- | -- |
| **Organochlorine Pesticides (GC)** | **8081A** | | | | | | | | | | | | | |
| DCB Decachlorobiphenyl | 2051-24-3 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | 40 | 158 | % |
| Tetrachloro-m-xylene | 877-09-8 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | 49 | 123 | % |
| Hexachlorobenzene | 118-74-1 | 10 | 188 | % | 37 | % | 10 | 188 | % | 30 | % | -- | -- | -- |
| Hexachlorobutadiene | 87-68-3 | 37 | 119 | % | 39 | % | 37 | 119 | % | 30 | % | -- | -- | -- |
| **TOC (Puget Sound)** | **9060_PSEP** | | | | | | | | | | | | | |
| Total Organic Carbon | 7440-44-0 | 27.8 | 170 | % | 35 | % | 50 | 140 | % | 35 | % | -- | -- | -- |
| **Percent Moisture** | **Moisture** | | | | | | | | | | | | | |
| Percent Solids | STL00234 | 80 | 120 | % | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| Percent Moisture | STL00177 | 80 | 120 | % | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| Total Solids | STL00291 | 80 | 120 | % | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| **Semivolatile Organic Compounds (GC/MS)** | **8270C** | | | | | | | | | | | | | |
| 2-Fluorophenol | 367-12-4 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | 36 | 145 | % |

# TABLE A-5
# LABORATORY QC SAMPLE ACCEPTANCE CRITERIA
# SEDIMENT

| | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Phenol-d5 | 4165-62-2 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | 38 | 149 | % |
| 2,4,6-Tribromophenol | 118-79-6 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | 28 | 143 | % |
| Nitrobenzene-d5 | 4165-60-0 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | 38 | 141 | % |
| 2-Fluorobiphenyl | 321-60-8 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | 42 | 140 | % |
| Terphenyl-d14 | 1718-51-0 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | 42 | 151 | % |
| Phenol | 108-95-2 | 66 | 126 | % | 26 | % | 70 | 140 | % | 26 | % | -- | -- | -- |
| 1,4-Dichlorobenzene | 106-46-7 | 62 | 132 | % | 32 | % | 75 | 125 | % | 32 | % | -- | -- | -- |
| Benzyl alcohol | 100-51-6 | 42 | 147 | % | 60 | % | 55 | 125 | % | 60 | % | -- | -- | -- |
| 1,2-Dichlorobenzene | 95-50-1 | 68 | 118 | % | 60 | % | 75 | 125 | % | 60 | % | -- | -- | -- |
| 2-Methylphenol | 95-48-7 | 56 | 121 | % | 25 | % | 75 | 130 | % | 25 | % | -- | -- | -- |
| 3 & 4 Methylphenol | 15831-10-4 | 61 | 126 | % | 27 | % | 75 | 130 | % | 27 | % | -- | -- | -- |
| 2,4-Dimethylphenol | 105-67-9 | 58 | 133 | % | 60 | % | 65 | 140 | % | 60 | % | -- | -- | -- |
| Benzoic acid | 65-85-0 | 10 | 130 | % | 60 | % | 20 | 175 | % | 60 | % | -- | -- | -- |
| 1,2,4-Trichlorobenzene | 120-82-1 | 63 | 128 | % | 28 | % | 70 | 125 | % | 28 | % | -- | -- | -- |
| Naphthalene | 91-20-3 | 64 | 129 | % | 26 | % | 75 | 125 | % | 26 | % | -- | -- | -- |
| Hexachlorobutadiene | 87-68-3 | 59 | 134 | % | 60 | % | 75 | 125 | % | 60 | % | -- | -- | -- |
| 2-Methylnaphthalene | 91-57-6 | 65 | 125 | % | 27 | % | 75 | 125 | % | 27 | % | -- | -- | -- |
| Dimethyl phthalate | 131-11-3 | 65 | 125 | % | 60 | % | 60 | 160 | % | 60 | % | -- | -- | -- |
| Acenaphthylene | 208-96-8 | 69 | 129 | % | 28 | % | 75 | 125 | % | 28 | % | -- | -- | -- |
| Acenaphthene | 83-32-9 | 65 | 130 | % | 27 | % | 75 | 125 | % | 27 | % | -- | -- | -- |
| Dibenzofuran | 132-64-9 | 70 | 125 | % | 60 | % | 75 | 125 | % | 60 | % | -- | -- | -- |
| Diethyl phthalate | 84-66-2 | 64 | 129 | % | 26 | % | 60 | 155 | % | 26 | % | -- | -- | -- |
| Fluorene | 86-73-7 | 68 | 128 | % | 31 | % | 75 | 125 | % | 31 | % | -- | -- | -- |
| N-Nitrosodiphenylamine | 86-30-6 | 88 | 153 | % | 60 | % | 75 | 125 | % | 60 | % | -- | -- | -- |
| Hexachlorobenzene | 118-74-1 | 61 | 136 | % | 60 | % | 75 | 125 | % | 60 | % | -- | -- | -- |
| Pentachlorophenol | 87-86-5 | 29 | 124 | % | 68 | % | 55 | 125 | % | 68 | % | -- | -- | -- |

# TABLE A-5
# LABORATORY QC SAMPLE ACCEPTANCE CRITERIA
# SEDIMENT

| | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Phenanthrene | 85-01-8 | 65 | 125 | % | 28 | % | 75 | 125 | % | 28 | % | -- | -- | -- |
| Anthracene | 120-12-7 | 73 | 123 | % | 27 | % | 75 | 125 | % | 27 | % | -- | -- | -- |
| Di-n-butyl phthalate | 84-74-2 | 69 | 124 | % | 60 | % | 55 | 145 | % | 60 | % | -- | -- | -- |
| Fluoranthene | 206-44-0 | 61 | 121 | % | 36 | % | 70 | 125 | % | 36 | % | -- | -- | -- |
| Pyrene | 129-00-0 | 54 | 134 | % | 31 | % | 75 | 125 | % | 31 | % | -- | -- | -- |
| Butyl benzyl phthalate | 85-68-7 | 65 | 140 | % | 60 | % | 55 | 145 | % | 60 | % | -- | -- | -- |
| Benzo[a]anthracene | 56-55-3 | 64 | 124 | % | 27 | % | 75 | 125 | % | 27 | % | -- | -- | -- |
| Chrysene | 218-01-9 | 71 | 126 | % | 26 | % | 75 | 125 | % | 26 | % | -- | -- | -- |
| Bis(2-ethylhexyl) phthalate | 117-81-7 | 64 | 144 | % | 60 | % | 55 | 145 | % | 60 | % | -- | -- | -- |
| Di-n-octyl phthalate | 117-84-0 | 58 | 148 | % | 31 | % | 55 | 145 | % | 31 | % | -- | -- | -- |
| Benzofluoranthene | 56832-73-6 | 57 | 137 | % | 31 | % | 75 | 125 | % | 31 | % | -- | -- | -- |
| Benzo[a]pyrene | 50-32-8 | 68 | 128 | % | 30 | % | 75 | 125 | % | 30 | % | -- | -- | -- |
| Indeno[1,2,3-cd]pyrene | 193-39-5 | 59 | 139 | % | 29 | % | 75 | 125 | % | 29 | % | -- | -- | -- |
| Dibenz(a,h)anthracene | 53-70-3 | 57 | 142 | % | 30 | % | 75 | 125 | % | 30 | % | -- | -- | -- |
| Benzo[g,h,i]perylene | 191-24-2 | 57 | 142 | % | 28 | % | 75 | 125 | % | 28 | % | -- | -- | -- |
| **Polychlorinated Biphenyls (PCBs) by Gas Chromatography** | **8082** | | | | | | | | | | | | | |
| Tetrachloro-m-xylene | 877-09-8 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | 45 | 135 | % |
| DCB Decachlorobiphenyl | 2051-24-3 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | 50 | 140 | % |
| PCB-1016 | 12674-11-2 | 40 | 140 | % | 20 | % | 40 | 140 | % | 20 | % | -- | -- | -- |
| PCB-1232 | 11141-16-5 | 50 | 150 | % | 30 | % | 50 | 150 | % | 30 | % | -- | -- | -- |
| PCB-1242 | 53469-21-9 | 57 | 128 | % | 20 | % | 57 | 128 | % | 20 | % | -- | -- | -- |
| PCB-1254 | 11097-69-1 | 65 | 132 | % | 20 | % | 65 | 132 | % | 20 | % | -- | -- | -- |
| PCB-1260 | 11096-82-5 | 60 | 130 | % | 20 | % | 60 | 130 | % | 20 | % | -- | -- | -- |

# TABLE A-5
# LABORATORY QC SAMPLE ACCEPTANCE CRITERIA
# SEDIMENT



| | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Mercury (CVAA)** | **7471A** | | | | | | | | | | | | | |
| Mercury | 7439-97-6 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % | -- | -- | -- |

# TABLE A-6
# LABORATORY QC SAMPLE ACCEPTANCE CRITERIA
# SOIL AND WATER

| Analyte Description | CAS Number/ Method | LCSREC - Recovery Low | LCSREC - Recovery High | LCSREC - Units | LCSRPD - Precision | LCSRPD - Units | MSREC - Recovery Low | MSREC - Recovery High | MSREC - Units | MSRPD - Precision | MSRPD - Units |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Soil - Metals (ICP)** | **6010B** | | | | | | | | | | |
| Arsenic | 7440-38-2 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % |
| Barium | 7440-39-3 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % |
| Cadmium | 7440-43-9 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % |
| Chromium | 7440-47-3 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % |
| Lead | 7439-92-1 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % |
| Selenium | 7782-49-2 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % |
| Silver | 7440-22-4 | 75 | 120 | % | 20 | % | 75 | 120 | % | 20 | % |
| **Soil - Mercury (CVAA)** | **7471A** | | | | | | | | | | |
| Mercury | 7439-97-6 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % |
| **Water - Metals (ICP/MS)** | **6020** | | | | | | | | | | |
| Arsenic | 7440-38-2 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % |
| Barium | 7440-39-3 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % |
| Cadmium | 7440-43-9 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % |
| Chromium | 7440-47-3 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % |
| Lead | 7439-92-1 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % |
| Selenium | 7782-49-2 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % |
| Silver | 7440-22-4 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % |
| **Water - Mercury (CVAA)** | **7470A** | | | | | | | | | | |
| Mercury | 7439-97-6 | 80 | 120 | % | 20 | % | 80 | 120 | % | 20 | % |

APPENDIX B

# Bioassay Analysis Plan

# BIOASSAY ANALYSIS PLAN

## TOXICICTY TESTING PERFORMED UNDER THE PUGET SOUND ESTUARY PROGRAM (PSEP) FOR THE PURPOSES OF SEDIMENT MANAGEMENT STANDARD (SMS) EVALUATION

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**December 2017**

# Contents



# Tables

## ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| AFDW | Ash-Free Dry Weight |
| Ccm | Centimeter |
| CSL | Cleanup Screening Level |
| DMMP | Dredged Material Management Office (United States Army Corps of Engineers, Seattle District (USACE); the United States Environmental Protection Agency, Region 10 (EPA); the Washington Department of Ecology (Ecology); and the Washington State Department of Natural Resources (DNR).) |
| Ecology | Washington Department of Ecology |
| $LC_{50}/EC_{50}$ | Lethal/Effect Concentration to 50% of Test Population |
| MIG | Mean Individual Growth per Day (mg/ind/day) |
| mg/L | Milligrams per Liter |
| mL | Milliliter |
| % | Percent |
| PSEP | Puget Sound Estuary Program/Protocols |
| SCO | Sediment Cleanup Objective |
| SCUM II | Sediment Cleanup Users Manual II (WDOE 2015) |
| SMARM | Sediment Management Annual Review Meeting |
| SMS | Sediment Management Standards (Washington State Administrative Code (WAC 173-204)) |
| USACE | United States Army Corps of Engineers |
| WA | Washington State |
| WDOE | Washington (State) Department of Ecology |

# 1. BIOLOGICAL TESTING

Biological testing procedures will be performed in accordance with the Puget Sound Estuary Program (PSEP) and Washington State Department of Ecology Sediment Cleanup User's Manual (SCUM II) (WDOE 2015) guidance. The laboratory analysis will be consistent with PSEP guidelines (PSEP 1995), and any recent modifications adopted through the Seattle District U. S. Army Corps of Engineers (USACE) Sediment Management and Review Meeting (SMARM) process.

A suite of three bioassays (amphipod mortality, larval development, and juvenile polychaete growth test) will be conducted on each sample submitted for biological testing. All biological testing will follow Recommended Guidelines for Conducting Laboratory Bioassays on Puget Sound Sediments (PSEP 1995), with appropriate modifications identified through the SMARM process.

## 1.1 Reference Sediment Selection

Bioassay testing requires that test sediments be matched and run with appropriate approved reference sediment(s) to factor out background conditions and sediment grain-size effects on bioassay organisms. SMS sediment evaluations may require the inclusion of one to four concurrent reference treatments depending on the sample size and range of grain sizes of the project areas. Reference sediment will be collected from Carr Inlet, Washington, or other approved reference location as directed by the DMMO Agencies. These reference sediments will be analyzed in the field for percent fines using the wet sieve method (PSEP 1986) to confirm correlation with the targeted grain size ranges. The target sediment will be within ±20% of the percent fines content of the sediment sample(s) being assessed. Reference sediments will be statistically compared to project samples that have similar percent fine material, unless otherwise approved.

## 1.2 Ammonia and Sulfide Monitoring

Prior to bioassay test initiation, a determination will be made regarding the likelihood of non-treatment effects from sulfides and ammonia in the amphipod, larval, and juvenile polychaete bioassays. Sediment evaluation conducted for the purposes of dredge material suitability includes an option to purge and/or conduct additional sensitive testing (positive control reference-toxicant tests) to account for these non-treatment effects. For the purposes of evaluating the potential toxicity of sediments under the Sediment Management Standards (SMS) program, purging is not required unless previously determined through coordination with Ecology. The threshold values listed below are for reference only and may aid in determining if ammonia and sulfides are a potential contributor to any observed toxicity. The purging trigger values are summarized in the document *DMMP Clarification Paper Modifications to Ammonia and Sulfide Triggers for Purging and Reference Toxicant Testing for Marine Bioassays* (Inouye 2015) and are summarize in Table 1-1.

**Table 1-1. Reference Toxicant and Purging Triggers for Marine Bioassays**

| Analyte/Action | Bedded Sediment Tests | | | Larval tests | |
|---|---|---|---|---|---|
| | ***Neanthes*** | ***Ampelisca*** | ***Eohaustorius*** | **Bivalve** | **Echinoderm** |
| Unionized Ammonia (mg/L) Action: Concurrent Reference-Toxicant Test | 0.23 | 0.118 | 0.4 | 0.02 | 0.007 |
| Unionized Ammonia (mg/L) Action: Pre-Test Purge | 0.46 | 0.236 | 0.8 | 0.04 | 0.014 |
| Hydrogen Sulfide (mg/L) Action: Pre-Test Purge | 3.4 | 0.0094 | 0.122 | 0.0025 | 0.01 |

## 1.3 Bioassays

The standard suite of marine bioassays will consist of: (1) sediment larval (bivalve or echinoderm) combined survival/normality test, (2) *Neanthes* arenaceodentata 20-day survival and growth test (survival; dry weight and ash-free dry-weight endpoints), (3) amphipod mortality test. General biological testing procedures, bioassay-specific procedures, test interpretation, and required inclusions in the laboratory report will be consistent with the procedures in the guidance.

## 1.4 Bioassay-Specific Procedures

This section describes specific procedures for the suite of bioassays used for sediment characterization.

## 1.5 Amphipod Mortality Bioassay

The amphipod test evaluates the survival of benthic-dwelling amphipods exposed to project sediment over a 10-day period. The PSEP bioassay test methodology allows for the use of different approved test species most appropriate for the grain size and salinity of the project area. The two primary amphipod species are *Eohaustorius estuarius* and *Ampelisca abdita*. The grain size criteria for species selection is summarized in the table below.

**Table 1-2. Amphipod Species Selection Based on Grain Size**

| Species | Sediment Grain Size |
|---|---|
| *Eohaustorius estuarius* | <60% fines (≥40% sand) |
| *Ampelisca abdita* | ≥ 60% fines (<40% sand) |

The amphipod bioassay is conducted as 10-day static exposure with five replicates for each test treatment. Two centimeters of sediment (approximately 175 mL) are placed into each 1-L glass chamber with 775 mL of overlying water. Trickle-flow aeration is provided through glass pipettes, and care is taken to avoid disturbing the sediment surface. Test chambers are placed into randomly assigned positions and allowed to equilibrate to test conditions overnight.

Prior to the test initiation, water quality measurements are taken in all replicates for each test treatment and included dissolved oxygen, temperature, salinity, and pH. Total ammonia and total sulfide concentrations are measured in both interstitial (pore water) and overlying water at initiation and termination. Evaluation of the interstitial water (pore water) will also include measurements of salinity, pH, and temperature to calculate unionized ammonia and hydrogen sulfide. These measurements are made from a sacrificial surrogate chamber for each test treatment. Sediment pore water is extracted via centrifugation. During the test, water quality is monitored daily in one surrogate replicate per treatment. A summary of the recommended amphipod test conditions are detailed in the table below.

To initiate the test, 20 organisms are randomly allocated to each of the test chambers. Amphipods that do not bury within approximately one hour are replaced with healthy amphipods. No food is provided during the 10-day exposure for the amphipod test.

At test termination, sediment from each test chamber is sieved through a 0.5-mm screen and the number of surviving and dead amphipods is enumerated.

**Table 1-3. Amphipod Test Conditions**

| | | |
|---|---|---|
| Test Procedures | PSEP 1995 with SMARM revisions, SCUM II (2015) | |
| Test Type/Duration | 10-Day/ Static | |
| Test Lighting | Continuous | |
| Test Chamber | 1000-mL Glass Chamber | |
| Replicates per Treatment | 5 test replicates and 2 surrogate chambers (minimum) | |
| Organisms per Replicate | 20 | |
| Exposure Volume | 175 mL sediment (2 cm) / 775 mL of overlying seawater | |
| Feeding | None | |
| Water Renewal | None | |
| Aeration | Trickle-flow Aeration | |
| Test Species | *Ampelisca abdita* | *Eohaustorius estuarius* |
| Organism Age | Immature | Mature amphipods 3-5 mm, mixed sexes |
| Test Dissolved Oxygen | > 60% Saturation<br>(4.6 mg/L @ 28ppt and 20°C) | > 60% Saturation<br>(5.1 mg/L @ 28ppt and 15°C) |
| Test Temperature | 20 ± 1°C | 20 ± 1°C |
| Test Salinity | 28 ± 1 ppt | 28 ± 1 ppt |
| Test pH | Measure; no limits | Measure; no limits |
| Control Performance Standard | ≤ 10% Mortality | ≤ 10% Mortality |

## 1.6 Larval Embryo Development Bioassay

This test monitors larval embryo development of an echinoderm or molluscan species (e.g., *Strongylocentrotus pupuratus*, *Dendraster excentricus*, *Mytilus galloprovincialis*, or *Crassostrea gigas*) in the presence of test sediment. The test is run until the appropriate stage of development is achieved in a sacrificial seawater control and can range from 48 hours (bivalves) to 96 hours (Echinoderms). At the end of the test, larvae from each test sediment exposure are examined to quantify normality and survival.

The larval development bioassay is conducted as a static exposure with five replicates for each test treatment. Approximately 18 g (±1 g) of sediment is placed into each 1-L glass chamber with 900 mL of overlying water. Test chambers are then shaken for 10 seconds and placed into randomly assigned positions. The larval test is performed without aeration unless dissolved oxygen levels fell below threshold levels for action.

Prior to the test initiation, water quality measurements are taken in the surrogate chamber for each test treatment and included dissolved oxygen, temperature, salinity, and pH. Ammonia and sulfide concentrations are measured in the overlying water at initiation and termination (Note: The thin depth of sediment used in the larval bioassay does not allow for the extraction of interstitial porewater for analysis). These measurements are made from a sacrificial surrogate chamber for each test treatment. During the test, water quality is monitored daily in one surrogate replicate per treatment. A summary of the recommended larval embryo test conditions are detailed in the table below.

To initiate the test, spawning is induced in adult organisms by injection of potassium chloride (echinoderms) or by heat shock (bivalves). Eggs and sperm are collected separately until a sufficient amount for testing has been obtained. A homogenized sperm solution (from several males) is then added to the eggs to create an embryo stock. Fertilization is checked after 30-mins. Only embryo stocks with >90% fertilization are used in test initiation. The density of the embryo stock s obtained by enumerating the number of embryos in a known subsample. This is used to determine the volume of embryo stock solution to deliver approximately 27,000 embryos to each test chamber.

For termination of the bivalve development test, the resuspension method will be employed (Kendall 2013) to liberate any larvae trapped in fine sediments or the flocculent layer at the sediment/water interface. Resuspension is conducted approximately 6 to 8 hours prior to test termination using a perforated plunger to gently homogenize the water, larvae, and settled sediment. This process is currently only recommended for the bivalve test to address a phenomenon of larvae that can become entrained in the sediment matrix. Tests conducted with the larvae of echinoderms will not be terminated with this procedure.

The larval developmental tests are terminated approximately 48 - 96 hours after initiation to ensure that approximately 90% of the control larvae had achieved the appropriate life-stage. The overlying water from each chamber is decanted and carefully mixed with a perforated plunger to suspend the larvae. Two 10-mL subsamples are transferred from each chamber to shell vials and preserved with a 5% buffered formalin and Rose Bengal solution. Normal and abnormal larvae are enumerated under an inverted compound microscope.

**Table 1-4. Larval Test Conditions**

| | | | | |
|---|---|---|---|---|
| Test Procedures | PSEP 1995 with SMARM revisions, SCUM II (2015) | | | |
| Test Type/Duration | 48 – 96 Hour/ Static | | | |
| Test Lighting | 14-hr light, 10-hr dark | | | |
| Test Chamber | 1000-mL Glass Chamber | | | |
| Replicates per Treatment | 5 test replicates and 1 surrogate chamber (minimum) | | | |
| Organisms per Replicate | 20 – 30 embryos/mL (Echinoderms)<br>20 – 40 embryos/mL (Bivalves) | | | |
| Exposure Volume | 18 g sediment/ 900 mL water | | | |
| Feeding | None | | | |
| Water Renewal | None | | | |
| Aeration | None, Unless Dissolved Oxygen is Fall Below 60% Saturation | | | |
| Test Species | *Dendraster excentricus* | *Strongylocentrotus purpuratus* | *Mytilus galloprovincialis* | *Crassostrea gigas* |
| Organism Age | <2-h old embryos | | | |
| Test Dissolved Oxygen | > 60% Saturation<br>(5.1 mg/L @ 28ppt and 20°C) | | > 60% Saturation<br>(5.0 mg/L @ 28ppt and 20°C) | > 60% Saturation<br>(4.6 mg/L @ 28ppt and 15°C) |
| Test Temperature | 15 ± 1°C | | 16 ± 1°C | 20 ± 1°C |
| Test Salinity | 28 ± 1 ppt | | | |
| Test pH | Measure; no limits | | | |

## 1.7 Juvenile Polychaete Survival and Growth Bioassay

The 20-day polychaete survival and growth test is conducted with juvenile polychaete worms (*Neanthes arenaceodentata*). The test endpoints are survival and mean individual growth rate (expressed as mg/individual/day).

The polychaete bioassay is conducted as a 20-day static-renewal test, with overlying water exchanges of 300 mL of water occurring every third day. Each test treatment consists of five replicates of 1-L glass chambers, which are filled with two centimeters of sediment (approximately 175 mL) and 775 mL of overlying water. Trickle-flow aeration is provided through glass pipettes, and care is taken to avoid disturbing the sediment surface. Test chambers are then randomly assigned positions and allowed to equilibrate to test conditions overnight.

Prior to the test initiation, water quality measurements are taken in a surrogate chamber for each test treatment and include dissolved oxygen, temperature, salinity, and pH. Total ammonia and total sulfide concentrations are measured in both interstitial (pore water) and overlying water at initiation and termination. Evaluation of the interstitial water (pore water) will also include measurements of salinity, pH, and temperature to calculate unionized ammonia and hydrogen sulfide. Porewater salinity in the polychaete should be confirmed to be >20 ppt prior to testing. These measurements are made from a sacrificial surrogate chamber for each test treatment. Sediment pore water is extracted via centrifugation. During the test, water quality is monitored daily in one surrogate replicate per treatment. A summary of the recommended juvenile polychaete test conditions are detailed in the table below.

To initiate the test, five organisms are randomly allocated to each of the test chambers. During the test, organisms are fed a diet of 40-mg of TetraMarin® slurry every other day (approximately 8-mg dry weight per worm). Pre-test initial biomass is determined by taking dry weight and ash-free dry weight (AFDW) measurements of three replicates of five worms each on Day 0.

At test termination, sediment from each test chamber are sieved through a 0.5-mm screen. All worms are recovered, enumerated, rinsed in deionized water (to remove salt), and transferred to pre-weighed aluminum foil weigh boats. After drying in an oven at 60°C for approximately 24 hours, each weigh-boat is removed, cooled in a desiccator and weighed to obtain dry weight measurements. They are then heated to 550°C for 2 hours to determine the ashed weight. Ash-free dry weights (AFDW) are calculated to correct for the influence of sediment grain size differences between treatments. The ash-free dry weight value is derived from by subtracting the final ashed weight from the final dry weight of each replicate. Both dry weight and AFDW are used to determine individual worm weight and growth rates.

**Table 1-5. Juvenile Polychaete Test Conditions**

| | |
|---|---|
| Test Procedures | PSEP 1995 with SMARM revisions, SCUM II (2015) |
| Test Type/Duration | 20-Day/ Static Renewal |
| Test Lighting | 16-hr light, 8-hr dark |
| Test Chamber | 1000-mL Glass Chamber |
| Replicates per Treatment | 5 test replicates and 2 surrogate chambers (minimum) |
| Organisms per Replicate | 5 |
| Exposure Volume | 175 mL sediment (2 cm) / 775 mL of overlying seawater |
| Feeding | 40 mg ground TetraMin® per chamber every other day |
| Water Renewal | Every third day (1/3 of overlying water) |
| Aeration | Trickle-flow Aeration |
| Test Species | *Neanthes arenaceodentata* |
| Organism Age | Juvenile; 2 – 3 weeks post-emergence<br>0.25 – 1.00 mg per individual (dry weight) |
| Test Dissolved Oxygen | > 60% Saturation<br>(4.6 mg/L @ 28ppt and 20°C) |
| Test Temperature | $20 \pm 1$°C |
| Test Salinity | $28 \pm 2$ ppt |
| Test pH | Measure; no limits |

### 1.8 Biological Laboratory Written Report

The biological laboratory will prepare a written report documenting all the activities associated with sample analyses and interpretation. At a minimum, the following will be included in the report:

- Results of the laboratory bioassay analyses and QA/QC results for test and reference sediments, reported both in hard copy and in the Ecology Environmental Information Management (EIM) data format (Raw data will be legible or typed. Illegible data may result in the need for a retest if the agencies cannot interpret the data);
- Results of positive and negative control, including reference toxicant specific laboratory control limits;
- Water quality monitoring results;
- All protocols used during analyses, including explanation of any deviation from the PSEP protocols and the approved sampling plan;
- Chain-of-custody procedures, including explanation of any deviation from the identified protocols;
- Source of test organisms; and
- Source of control sediment and control seawater
- Any deviations that require explanation to help validate the results
- Statistical analysis reports

## 2. BIOASSAY LABORATORY QUALITY ASSURANCE AND QUALITY CONTROL

Bioassay testing will be conducted in accordance with PSEP methodology and SMARM updates as detailed in SCUM II (WDOE 2015). The following sections describe the QA/QC components employed to ensure bioassays meet SMS performance requirements.

### 2.1 Sediment Holding Time

The bioassay testing will be initiated within 56 calendar days of sample collection. This includes a recognition of time to obtain test organisms and control and reference sediments in a timely manner. Every effort will be made to initiate the bioassays as soon as feasible to support the opportunity for any second-round (additional) biological testing within the allowable 56-day holding period if such need arises. The laboratory will maintain chain-of-custody procedures throughout biological testing.

### 2.2 Negative Controls

Negative control sediments will be run with each test batch and test species. Negative control sediments are clean sediments in which the test organism normally lives (native sediment for field collected organisms) or a known sediment matrix known to historically meet test acceptability criteria. Failure to the meet the established performance criteria may indicated the need to retest.

**Table 2-1. Negative Control Performance Standards**

| Test | Endpoint | Control Criterion |
|---|---|---|
| Amphipod | Mean Mortality | ≤ 10% |
| Embryo Larval Development | Mean Normal Survivorship (Mean Number Normal in the Control / Stocking Density) | >0.70 (70%) |
| Juvenile Polychaete | Mean Mortality | ≤ 10% |
| | Mean Growth (MIG) | >0.72 mg/ind/day (Optimal)<br>>0.38 mg/ind/day (Kendall 1996) |

## 2.3 Reference Sediment

Reference sediments will be run with each test batch and test species. Reference sediments are in-water natural marine sites within the Puget Sound region. As such, these samples are not free of anthropogenic inputs, but have generally met the established biological performance standards summarized below. The most common locations for field collected references to be utilized under PSEP biological testing programs are Carr Inlet and Sequim Bay. Reference sample results that do not meet the established performance standards may indicate that the material in question is not be suitable for comparison to the project sample. Note that the overall test acceptability is determined by a line-of-evidence approach evaluating the sum of the QA/QC criteria (including negative and positive control performance).

Within-test sample comparisons for SMS evaluation will be conducted between suitable reference sediment responses and the respective project samples that have similar grain size characteristics (±20% fines). If the reference sediment results do not meet the established performance standard criteria then comparisons may be performed against another concurrently tested reference, or in some cases, the negative control material. Any modifications that arise out of a reference sediment performance concern will be determined with Client and Agency coordination.

**Table 2-2. Reference Sediment Performance Standards**

| Test | Endpoint | Reference Criterion |
|---|---|---|
| Amphipod | Mean Mortality | ≤ 25% |
| Embryo Larval Development | Mean Control Normalized Normal Survivorship (Mean Number Normal in the Reference / Mean Number Normal in the Control) | >0.65 (65%) |
| Juvenile Polychaete | Mean Mortality | No Criterion |
| | Mean Reference Growth (MIG) / Mean Control Growth (MIG) | >0.80 (80%) |

## 2.4 Positive Controls

A positive control will be run for each bioassay. Positive controls are dose-response tests (reference-toxicant tests) using chemicals known to be toxic to the test organism and which provide an indication of the sensitivity of the organisms used in the bioassay. Ammonium chloride reference toxicant will be used for the amphipod mortality, larval development, and juvenile polychaete growth bioassays. Positive control tests are conducted as water-only exposures to remove the potential confounding factors of sediment matrix (grain size, TOC, etc.). The EcoAnalysts Port Gamble Laboratory has an extensive database for the ammonia test and currently uses it to validate organism health and predict potential ammonia toxicity associated with a sample.

The positive control endpoint results (usually point estimate values such as $LC_{50}$ or $EC_{50}$) are compared to historical values with control limit values defined as ±2 standard deviations from a running mean. Results that fall outside this range will prompt an evaluation of the results in the context of the related project test, but do not *de facto* invalidate the test results. For example, a positive control test that indicates that a population of test organism utilized in a study were potentially more sensitive than usual, but where no significant toxicity was observed among the respective project treatments (negative control, references, and project samples), may be qualified as reliable result.

## 2.5 Replication

Five laboratory replicates of test sediments, reference sediments, and negative controls will be run for each bioassay.

## 2.6 Bioassay Interpretation

Test interpretations consist of endpoint comparisons of the project treatments to suitable controls and reference on an absolute percentage basis as well as statistical comparison to reference. The solid phase bioassay test interpretation and performance standard guidelines are presented in Table 2-3. Biological test interpretation under the SMS program identifies two levels of response in test organisms: Sediment Cleanup Objective (SCO) and Cleanup Screening Level (SCL) failures. Specific guidelines for each of these response categories is presented in SCUM II (WDOE 2015). Under each category, a sample must exceed the two conditions defined (statistically significant difference and a numerical comparison) for a determination of an SCO or CSL exceedance.

Experiment-wide survival, growth, and development data are analyzed statistically using one-way analysis of variance (ANOVA). When ANOVA shows a significant difference, multiple comparison t-tests then compared survival in each of the control and test sediments against survival in the reference sediments. Prior to analyses, normality and homogeneity of variance are assessed. When necessary to satisfy these assumptions, proportional survival data are arcsine square-root transformed.

**Table 2-3. Marine Bioassay Performance Standards and Evaluation Guidelines (SCUM II; WDOE 2015)**

| Bioassay | Negative Control Performance Standard | Reference Sediment Performance Standard | Sediment Cleanup Objective (SCO) | Cleanup Screening Level (CSL) |
|---|---|---|---|---|
| Amphipod Mortality | $M_C \le 10\%$ | $M_R \le 25\%$ | $M_T$ vs. $M_R$ SD (p=0.05) **AND** | |
| | | | $M_T > 25\%$ | $M_T - M_R > 30\%$ |
| Larval Development | $N_C \div I \ge 0.70$ | $N_R \div N_C \ge 0.65$ | $N_T$ vs. $N_R$ SD (p=0.10) **AND** | |
| | | | $N_T \div N_R < 0.85$ | $N_T \div N_R < 0.70$ |
| *Neanthes* Growth | $M_C \le 10\%$ and $MIG_C \ge 0.38$ | $MIG_R \div MIG_C \ge 0.80$ | $MIG_T$ vs. $MIG_R$ SD (p=0.05) **AND** | |
| | | | $MIG_T \div MIG_R < 0.70$ | $MIG_T \div MIG_R < 0.50$ |

M = mortality
N = normal larvae
I = initial count (Stocking Density)
MIG = Mean Individual Growth Rate (mg/individual/day)
SD = Statistically Significant Difference

Subscripts:
R = Reference Sediment
C = Negative Control
T = Test Sediment

## 3. REFERENCES

Inouye 2015. Modifications to Ammonia and Sulfide Triggers for Purging and Reference Toxicant Testing for Marine Bioassays. DMMP Clarification Paper. Prepared by Laura Inouye (Ecology), Erika Hoffman (EPA) and David Fox (Corps) for the DMMP agencies. Final Paper August 14, 2015

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