



April 2018
Oakland Bay and Shelton Harbor Sediments Cleanup Site

Shelton Harbor Pre-Remedial Design Investigation Work Plan

Washington State Department of Ecology

April 2018
Oakland Bay and Shelton Harbor Sediments Cleanup Site

Shelton Harbor Pre-Remedial Design Investigation Work Plan

Prepared for
Washington State Department of Ecology
300 Desmond Drive SE
Lacey, Washington 98503

Prepared by
Anchor QEA, LLC
720 Olive Way #1900
Seattle, Washington 98101

TABLE OF CONTENTS

1	Introduction	1
1.1	Summary of Remedial Actions.....	1
2	Pre-Remedial Design Investigations.....	2
2.1	Base Map Development	2
2.2	Geotechnical Investigations	2
2.2.1	Push Coring	2
2.2.2	Vane Shear Testing	3
2.2.3	Subsurface Borings	3
2.2.4	Geotechnical Data Evaluation.....	4
2.3	SMA Footprint Delineations.....	4
2.3.1	SMA-1	5
2.3.2	SMA-2	5
2.3.3	SMA-3	6
2.3.4	Sediment Grab Collection and Processing Modifications	7
2.3.5	Analytical Method Additions and Modifications.....	7
2.4	SMA-3 Porewater Sulfide Characterization.....	7
3	Schedule.....	9
4	References	10

TABLES

Table 2-1	Shelton Harbor SCU Sediment Cleanup and Remedial Action Levels.....	5
-----------	---	---

FIGURES

Figure 1-1	Oakland Bay and Shelton Harbor Sediments Cleanup Site
Figure 1-2	Shelton Harbor Sediment Cleanup Unit and Bathymetry
Figure 2-1	PDI Sampling Locations

APPENDICES

Appendix A	Sampling and Quality Assurance Project Plan Addendum
------------	--

ABBREVIATIONS

ASTM	ASTM International
DGT	diffusive gradients in thin films
Ecology	Washington State Department of Ecology
IAP	<i>Shelton Harbor Interim Action Plan</i>
PDI	pre-remedial design investigation
RAL	remedial action level
RD	remedial design
SCL	sediment cleanup level
SCU	Sediment Cleanup Unit
Simpson	Simpson Timber Company
SMA	sediment management area
SMS	Sediment Management Standards
SQAPP	<i>Remedial Investigation/Feasibility Study Work Plan and Sampling and Quality Assurance Project Plan: Shelton Harbor Sediment Cleanup Unit, Oakland Bay and Shelton Harbor Sediments Cleanup Site</i>
TBT	tributyltin
TEQ	toxic equivalence quotient
TOC	total organic carbon
VST	vane shear testing

1 Introduction

In accordance with the *Shelton Harbor Interim Action Plan* (IAP; Anchor QEA 2018), this Pre-Remedial Design Investigation (PDI) Work Plan describes data collection efforts necessary to support engineering design of sediment cleanup actions in portions of the Shelton Harbor Sediment Cleanup Unit (SCU) within the Oakland Bay and Shelton Harbor Sediments Cleanup Site (Washington State Department of Ecology [Ecology] Cleanup Site ID 13007; Figure 1-1). The Shelton Harbor SCU (Figure 1-2) was delineated by Ecology in accordance with the Washington State Sediment Management Standards (SMS), as further described in Order DE 14091 between Ecology and the Simpson Timber Company (Simpson). Specifically, this PDI Work Plan describes investigations to define the extent and geotechnical stability of planned sediment interim actions within the SCU.

1.1 Summary of Remedial Actions

As described in the IAP, three sediment management areas (SMAs) have been identified within the SCU for remedial action (Figure 1-2). The selected remedy is engineered capping to isolate contaminated sediments exceeding sediment cleanup levels (SCLs) or remedial action levels (RALs) at each of the following SMAs:

- **SMA-1:** An estimated capping area of 4.4 acres in the Shelton Creek delta (within the footprint of the northern Shelton Harbor habitat restoration project) that exceeds the RAL for dioxin/furan.
- **SMA-2:** An estimated capping area of 0.6 acre in the former marine railway area (also within the footprint of the northern Shelton Harbor habitat restoration project) that exceeds the SCL for copper and/or tributyltin (TBT) and/or the RAL for dioxin/furan.
- **SMA-3:** An estimated capping area of 3.5 acres in the southwestern harbor (outside of the footprint of the northern Shelton Harbor habitat restoration project) that exceeds the RAL for dioxin/furan. Portions of SMA-3 include shoreline slopes with subsurface wood debris accumulations. Additional characterization of hydrogen sulfide levels (a wood debris breakdown product) will be conducted in this area to verify the suitability of the capping remedy as part of the remedial design (RD) process.

2 Pre-Remedial Design Investigations

This section summarizes PDI data collection efforts needed to support the RD of sediment cleanup actions in SMA-1 through SMA-3, including the following:

- Base map development
- Geotechnical investigations
- SMA footprint delineations
- SMA-3 porewater sulfide characterization

Each of these data collection efforts is outlined in the following sections.

2.1 Base Map Development

While existing bathymetric and topographic surveys are available for SMA-1 through SMA-3 from various sources, more detailed condition surveys will be needed to support RD plans and provide the level of recency and data quality needed for RD. Condition survey coverage of the interim action areas will be performed as part of the PDI.

2.2 Geotechnical Investigations

Geotechnical investigations will be performed to support engineering evaluations of the stability and constructability of sediment caps. The cap subgrade geotechnical characteristics and bearing capacity in SMA-1 through SMA-3 will be assessed by collecting field data using push cores and conducting in situ vane shear testing (VST). Cap slope stability will be evaluated in SMA-3 with push coring and VST (supplemented with subsurface borings using a drill rig to provide deeper subsurface information along the shoreline). PDI sampling locations are depicted in Figure 2-1. Exploration and test methods are summarized in the following sections.

2.2.1 *Push Coring*

For all SMAs, push cores will be advanced at low tide (in the dry) or from a boat, as access allows.

The following methods are to be used for the push coring investigation:

- Push cores will be advanced at selected locations within the cap footprints to the depth of refusal (typically 3 to 6 feet below mudline, depending on subsurface conditions).
- A geologic log of each core will be prepared in the field by a geologist or geotechnical engineer and will describe the observed soil type, color, moisture, and consistency.
- Subsamples of the major geologic units will be collected and submitted to a geotechnical laboratory for further classification. Tests will be assigned by the field engineer so that a representative number of tests are conducted for each major soil type encountered during the investigation.

- If a relatively undisturbed interval of fine-grained soil is encountered and can be preserved, this interval will be left intact in the core liner, capped, and submitted to the laboratory without processing in the field to facilitate consolidation testing.
- Laboratory testing will consist of the following:
 - Moisture content in accordance with ASTM International (ASTM) D2216
 - Atterberg limits (plasticity) in accordance with ASTM D4318
 - Grain size in accordance with ASTM D6913 and D7928
 - Bulk density in accordance with ASTM D2937 (if a relatively undisturbed sample can be collected)
 - One-dimensional consolidation in accordance with ASTM D2435 (if a relatively undisturbed sample can be collected)
 - Unconsolidated, undrained triaxial shear strength in accordance with ASTM D2850 (if a relatively undisturbed sample can be collected)

2.2.2 *Vane Shear Testing*

For all SMAs, in situ VST will be conducted at low tide (in the dry) or from a boat, as access allows. The following methods are to be used for the VST investigation:

- In situ VST will be conducted at selected locations within the SMA to measure the undrained shear strength of fine-grained soils.
- VST procedures will follow ASTM D2573 and the testing device manufacturer standard operating procedure.
- For a given VST location, the testing device will be advanced below the mudline at 1-foot intervals, and the peak and residual undrained shear strength will be measured.
- VSTs will be advanced up to 5 feet below the mudline or until refusal is met.
- Where coarse-grained soils are encountered, the VST will not be used.
- VST measurements will be corrected for plasticity using the laboratory Atterberg limits test results in accordance with the standard of practice.

2.2.3 *Subsurface Borings*

Subsurface borings will be advanced along the shoreline in SMA-3 to provide more information about soil conditions at depth, which can be important in assessing shoreline cap stability. Borings will be advanced from a truck or track-mounted drill rig that is compatible with the access limitations of the investigation area. Subsurface boring procedures will use the following methods:

- Borings will be advanced to a depth of 40 feet below ground surface using either hollow-stem auger or mud rotary drilling methods.

- Samples will be collected using split spoons and standard penetration testing (ASTM D1586) and thin-walled (Shelby tube) sampling (ASTM D1587) as determined in the field by the geologist/geotechnical engineer overseeing the investigation.
- Samples will be collected at 5-foot-depth intervals to the bottom of the exploration.
- A geologic log of each boring will be prepared in the field by a geologist or geotechnical engineer and will describe the observed soil type, color, moisture, and consistency.
- Laboratory testing will be assigned in the field for a representative subset of samples collected from the borings as follows:
 - Moisture content in accordance with ASTM D2216
 - Atterberg limits (plasticity) in accordance with ASTM D4318
 - Grain size in accordance with ASTM D6913 and D7928
 - Bulk density in accordance with ASTM D2937
 - One-dimensional consolidation in accordance with ASTM D2435
 - Unconsolidated, undrained triaxial shear strength in accordance with ASTM D2850
- Explorations will be abandoned in accordance with Washington State drilling regulations.

2.2.4 Geotechnical Data Evaluation

The field logs, laboratory test data, and in situ VST data will be used to evaluate the bearing capacity of cap subgrade materials to support the weight of the cap. This evaluation will determine whether the cap lift thickness will need to be controlled to limit the potential for bearing capacity failure of underlying sediment during construction and also to assist in the design of the planned habitat project, which will place additional materials on top of the cap(s). Potential consolidation of the subgrade beneath the cap will be assessed to inform the long-term monitoring program. The stability of caps in SMA-3 will be evaluated using limit equilibrium methods for key cross sections that represent typical post-cap conditions. Geotechnical engineering evaluation methods will follow the current standard of practice for projects of this nature.

2.3 SMA Footprint Delineations

The IAP established SCLs and RALs for the dioxin/furan toxic equivalence quotient (TEQ), as well as copper and TBT (organic carbon normalized) concentrations, as summarized in Table 2-1. As discussed in the IAP, based on the weight of evidence (i.e., sediment profile image results showing a healthy benthic community, oxygenated conditions, no visible wood, improved larval bioassay methods, and recent natural recovery), no benthic risks from wood debris degradation were identified in SMA-1 or SMA-2. Localized sediment toxicity was observed in the southern harbor, including within parts of SMA-3. SMA-specific delineation to refine interim action areas relative to these remediation targets are summarized in the following sections. Ultimately, the extent of capping will be determined by interpreting the PDI results in consultation with Ecology.

**Table 2-1
Shelton Harbor SCU Sediment Cleanup and Remedial Action Levels**

Site-Specific Sediment Action Levels	Toxicity from Wood Debris Degradation	Dioxin/Furan TEQ (ng/kg)	cPAH TEQ (µg/kg)	Copper (mg/kg)	TBT (mg/kg OC)
Sediment Cleanup Level (SCL)	Sediment Cleanup Objective Bioassay Criteria ¹	19 ¹	52 ¹	390 ²	7.5 ²
Remedial Action Level (RAL)	N/A	42	Not Required	N/A	N/A

Notes:

1. Surface-weighted average concentration-based point of compliance is the top 10 centimeters.
2. Sample-specific point of compliance is the top 10 centimeters.

µg/kg: micrograms per kilogram

cPAH: carcinogenic polycyclic aromatic hydrocarbon

mg/kg: milligrams per kilogram

N/A: not applicable

ng/kg: nanograms per kilogram

OC: organic carbon

2.3.1 SMA-1

The preliminary delineation of the SMA-1 footprint presented in the IAP was based largely on a single exceedance of the dioxin/furan TEQ RAL (Figure 2-1). A sampling grid originating from the historical sampling location exhibiting the exceedance (SH-03) was used for development of this PDI Work Plan. Based on balancing the cost of data acquisition with the incremental cost of remediation in this area, a 150-foot sampling grid was identified for this PDI. Due to design timing constraints, all 20 grid nodes emanating from SH-03 will be sampled and submitted for dioxin/furan testing during the initial phase of PDI analysis (Figure 2-1).

2.3.2 SMA-2

The preliminary delineation of the SMA-2 footprint presented in the IAP was based largely on historical (2000 to 2005 sampling) data collected in the former marine railway area that exceeded the copper and TBT SCLs, as well as a single 2008 sample that exceeded the dioxin/furan RAL (Figure 2-1). Because of the relatively small width and elongated shape of the prospective SMA-2 remediation area, balancing the cost of data acquisition with the incremental cost of remediation resulted in an 80-foot PDI sampling grid in this area. Three sample types will be collected from SMA-2 as follows:

1. A grid node is collocated with historical SCL/RAL exceedances for TBT (historical sampling locations 001 and 002) within the rail. This sample will be composed of three subsample locations in-between each rail pair and composited (six-point composite) for TBT, total organic

carbon (TOC), and copper testing. Mass will be archived at these locations for dioxin/furan testing, if necessary.

2. Two grab samples (non-composite) will be collected from either side of the rail alignment and submitted for TBT, TOC, and copper testing.
3. Five surface grab locations in the deeper intertidal area of SMA-2 will be collected and analyzed to recharacterize the historical exceedance locations at SCS-7 (copper) and SH-02 (dioxin/furan). These samples will be submitted for TBT, TOC, copper, and dioxin/furan testing.
4. An additional seven samples will be collected from the sampling grid nodes and archived for future testing as necessary to accurately delineate SMA-2.

2.3.3 SMA-3

As discussed in the IAP, based on the combined 2005 to 2017 data, approximately 10 acres in the southwestern harbor (outside of the footprint of the northern Shelton Harbor habitat restoration project) have recently exceeded the dioxin/furan TEQ RAL. However, given the potential for natural recovery over the past ten years, the IAP contemplated a smaller 3.5-acre remediation area in SMA-3 (Figure 2-1), to be refined by this PDI. A sampling grid originating along the projected boundary of SMA-3 based on interpolations of the existing dioxin/furan data (as depicted in the IAP) was used for development of this PDI Work Plan. Based on balancing the cost of data acquisition with the incremental cost of remediation in this area, a 150-foot sampling grid was identified for this PDI, similar to that of SMA-1. Six grid nodes along this projected boundary will be sampled and submitted for dioxin/furan testing during the initial phase of PDI analysis (Figure 2-1). An additional 14 samples (four inshore and 10 offshore of the projected boundary) will be collected from grid nodes offset from the initial stations and archived for future testing as necessary to accurately delineate SMA-3.

While dioxin/furan was the primary SMA-3 chemical of concern targeted in the IAP, localized sediment toxicity has also been observed within and adjacent to SMA-3 that exceeds sediment cleanup objective bioassay criteria (Table 2-1). As discussed in Section 1.1 and further described in Section 2.4, additional characterization of hydrogen sulfide levels will be conducted in SMA-3 to verify the suitability of the capping remedy during RD.

As discussed in the IAP, while interim actions in SMA-1 and SMA-2 would immediately achieve cleanup levels throughout the footprint of the northern Shelton Harbor habitat restoration project, interim actions in SMA-3 may only partially achieve cleanup levels throughout the rest of the Shelton Harbor SCU. Follow-up remedial actions that may be necessary in other areas of the Shelton Harbor SCU will be addressed in the forthcoming SCU-wide Cleanup Action Plan (targeted for 2019).

2.3.4 Sediment Grab Collection and Processing Modifications

Sediment sample collection and processing methodologies to be used in this PDI are described in the Ecology-approved *Remedial Investigation/Feasibility Study Work Plan and Sampling and Quality Assurance Project Plan: Shelton Harbor Sediment Cleanup Unit, Oakland Bay and Shelton Harbor Sediments Cleanup Site* (SQAPP; Anchor QEA 2017) and are incorporated into this PDI Work Plan by reference. However, several modifications to the sample processing methodology will be implemented as part of this PDI to improve the precision and repeatability of the data, particularly for dioxin/furan analyses. For example, relatively large volume surface sediment samples will be collected as described in the SQAPP, placed into an on-deck container, and subsequently transported to an upland processing station. Triplicate field replicates (three separate grabs from the same locations) will also be collected at two sampling locations included in the initial phase of dioxin/furan testing discussed previously (one in SMA-1 and one in SMA-3). All samples will then be thoroughly homogenized electromechanically (using a drill with a stainless-steel paddle) prior to filling the laboratory-provided sampling jars. Additional details of the sample processing methodology to be utilized in this PDI are included in the SQAPP Addendum (Appendix A).

2.3.5 Analytical Method Additions and Modifications

Dioxin/furan analysis methodologies to be used in this PDI are described in the Ecology-approved SQAPP and are incorporated into this PDI Work Plan by reference. However, analysis for bulk sediment TOC, copper, and TBT were not included in the SQAPP. Accordingly, analytical methods, handling, and quality assurance/quality control requirements for these additional analytes are included in the SQAPP Addendum (Appendix A).

2.4 SMA-3 Porewater Sulfide Characterization

Porewater sulfide sampling methodologies to be used in this PDI are described in the Ecology-approved SQAPP and are incorporated into this PDI Work Plan by reference. Porewater sulfide concentrations in SMA-3 will be characterized using diffusive gradients in thin films (DGT) passive sampling methods. For this PDI, however, the DGTs will be deployed *ex situ* to improve the precision and repeatability of the data and to characterize short-term peak hydrogen sulfide concentrations. Along three SMA-3 transects (as depicted in Figure 2-1), surface sediment porewater concentrations will be characterized at intertidal and shallow subtidal elevations of approximately +5, 0, -5, -10, and -15 feet (if encountered) below mean lower low water (i.e., at a total of up to 15 stations and co-located with SMA-3 footprint delineation samples as practicable). Triplicate field replicates (obtained from separate grabs and DGT deployments at the same sampling location) will also be collected at two sampling locations. All samples will be collected within 2 hours (\pm) of a projected low tide of 0 mean lower low water or lower to characterize short-term peak hydrogen sulfide concentrations that occur at the end of the ebb tide groundwater discharge cycle. Intertidal samples

will be collected by hand, while subtidal samples will be collected using a sediment grab and processed as detailed in the SQAPP Addendum (Appendix A).

Porewater sulfide DGT analysis methodologies to be used in this PDI are described in the Ecology-approved SQAPP and are incorporated into this PDI Work Plan by reference. However, optical densitometry has been determined to provide a more accurate and precise method for analysis of sulfide concentrations in the DGT gels. The optical densitometry methodology is detailed in the SQAPP Addendum (Appendix A).

3 Schedule

Following Ecology approval of this PDI Work Plan and SQAPP Addendum, field sampling is currently targeted to commence on or about April 16, 2018, to be completed by approximately April 27, 2018. The initial round of preliminary (unvalidated) PDI analysis results is anticipated by the end of May 2018. Following collaborative evaluation of the preliminary data with Ecology, selected archived samples will be submitted for analysis, with results anticipated by early July 2018.

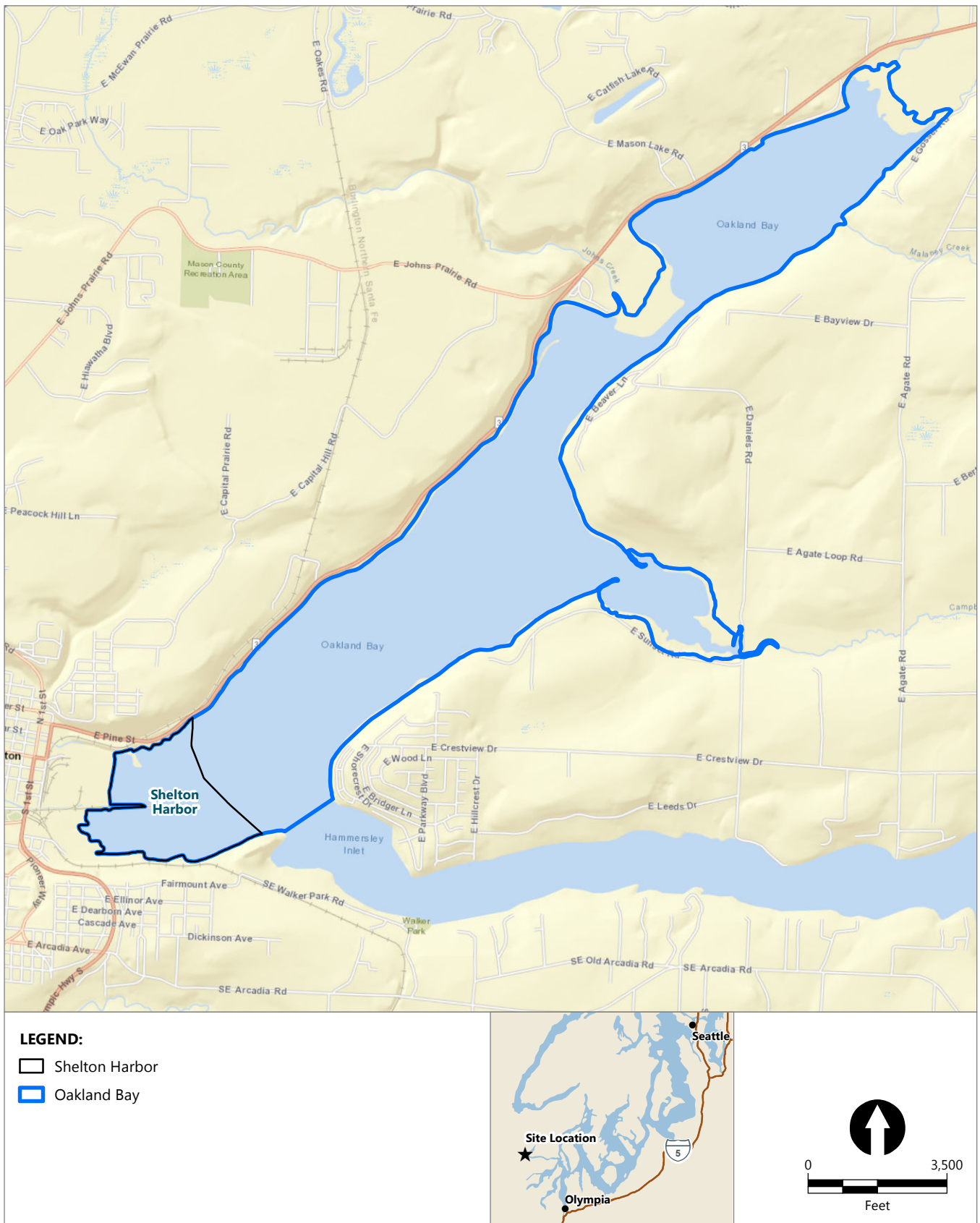
Anchor QEA, Simpson, and Ecology will collaboratively interpret the PDI data to delineate SMA footprints for RD. Final validated PDI data are anticipated by early August 2018, at which point the RD deliverables will be assembled. Depending largely on the time required to delineate Ecology-approved SMA footprints, the draft RD for SMA-1 through SMA-3 is currently targeted to be submitted for Ecology review in late August or early September 2018.

4 References

Anchor QEA (Anchor QEA, LLC), 2017. *Remedial Investigation/Feasibility Study Work Plan and Sampling and Quality Assurance Project Plan: Shelton Harbor Sediment Cleanup Unit, Oakland Bay and Shelton Harbor Sediments Cleanup Site*. Prepared for Simpson Timber Company and the Washington State Department of Ecology. July 2017.

Anchor QEA, 2018. *Interim Action Plan: Shelton Harbor Sediment Cleanup Unit, Oakland Bay and Shelton Harbor Sediments Cleanup Site*. Prepared for Simpson Timber Company and the Washington State Department of Ecology. January 2018.

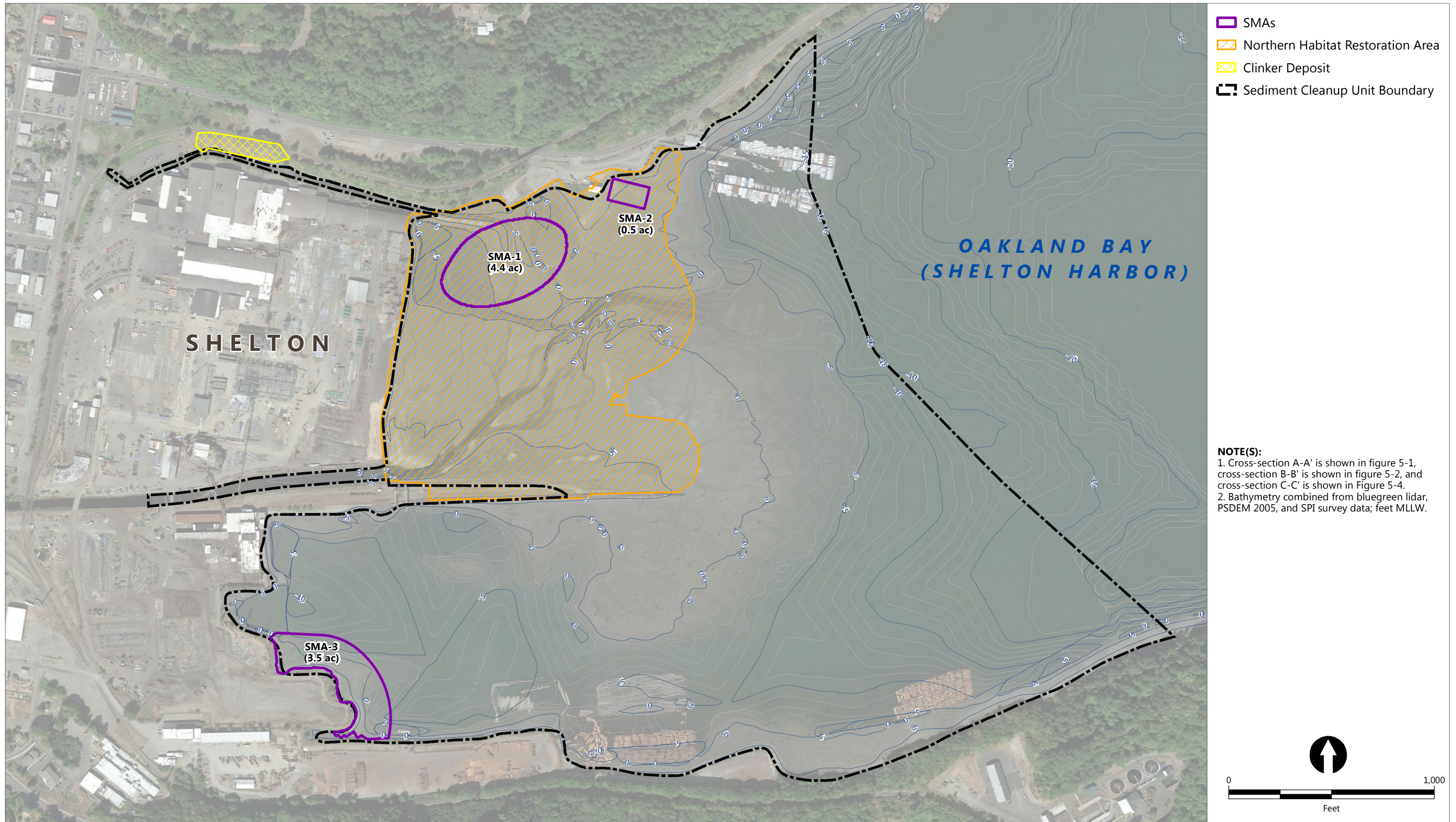
Figures



Publish Date: 2018/03/19, 11:40 AM | User: ckiblinger
 Filepath: \\corcas\gis\Jobs\Simpson_Timber_0008\SheltonHarbor\Maps\PDIWP\Shelton_Vicinity_Map_PDIWP.mxd



Figure 1-1
Oakland Bay and Shelton Harbor Sediments Cleanup Site
 Shelton Harbor Pre-Design Investigation Work Plan
 Oakland Bay and Shelton Harbor Sediments Cleanup Site



Publish Date: 2018/04/03, 12:28 PM | User: ckiblinger
 Filepath: \\orcas\gis\Jobs\Simpson_Timber_0008\SheltonHarbor\Maps\PDIWP\Shelton_SMA_PDIWP.mxd

Figure 1-2
Shelton Harbor Sediment Cleanup Unit and Bathymetry
 Shelton Harbor Pre-Design Investigation Work Plan
 Oakland Bay and Shelton Harbor Sediments Cleanup Site



Publish Date: 2018/04/06, 5:32 PM | User: ckiblinger
 Filepath: \\orcas\gis\Jobs\Simpson_Timber_0008\SheltonHarbor\Maps\PDIWP\Shelton_Proposed_Sample_Locs_PDIWP.mxd

Figure 2-1
PDI Sampling Locations
 Shelton Harbor Pre-Design Investigation Work Plan
 Oakland Bay and Shelton Harbor Sediments Cleanup Site

Appendix A

Sampling and Quality Assurance Project
Plan Addendum



April 2018
Shelton Harbor Sediment Cleanup Unit
Oakland Bay and Shelton Harbor Sediments Cleanup Site (Cleanup Site ID: 13007)



Sampling and Quality Assurance Project Plan Addendum

Prepared for Simpson Timber Company and the Washington State Department of Ecology

April 2018
Shelton Harbor Sediment Cleanup Unit
Oakland Bay and Shelton Harbor Sediments Cleanup Site (Cleanup Site ID: 13007)

Sampling and Quality Assurance Project Plan Addendum

Prepared for

Simpson Timber Company
1305 5th Avenue, Suite 2700
Seattle, Washington 98101

Prepared by

Anchor QEA, LLC
720 Olive Way, Suite 1900
Seattle, Washington 98101

Washington State Department of Ecology
Toxics Cleanup Program, Southwest Region
PO Box 47775
Olympia, Washington 98504-7775

TABLE OF CONTENTS

1	Introduction	1
2	Data Generation and Acquisition	2
2.1	Bathymetric Survey.....	2
2.2	Field Sampling Methods.....	2
2.2.1	Sampling Vessels and Field Equipment.....	2
2.2.2	Surface Sediment Collection and Processing.....	2
2.2.3	Surface Sediment Sample Collection Procedures.....	2
2.2.4	Subsurface Sediment Collection.....	5
2.2.5	Vane Shear Testing Procedures.....	7
2.2.6	Upland Geotechnical Boring Collection Procedures.....	7
2.2.7	Horizontal Positioning and Vertical Control.....	7
2.2.8	Sample Station Locations and Sample Identification.....	7
2.2.9	Equipment Decontamination Procedures.....	8
2.2.10	Sample Containers for Analysis	8
2.2.11	Sample Identification	8
3	Field and Laboratory Quality Assurance and Quality Control.....	10
3.1	Field Quality Assurance and Quality Control.....	10
3.1.1	Field Duplicates and Triplicates	10
3.1.2	Equipment Blanks.....	10
3.2	Analytical Laboratory Quality Assurance and Quality Control.....	10
4	Documentation, Recordkeeping, and Reporting Requirements	11
5	Data Validation and Usability	12
6	References	13

TABLES

Table 1	Target PDI Locations
Table 2	Parameters for Analysis, Methods, and Target Quantitation Limits
Table 3	Guidelines for Sample Handling and Storage
Table 4	Laboratory Quality Assurance/Quality Control Analysis Summary
Table 5	Data Quality Objectives

FIGURES

- Figure 1 SMA-1 PDI Target Locations
- Figure 2 SMA-2 PDI Target Locations
- Figure 3 SMA-3 PDI Target Locations

ATTACHMENTS

- Attachment 1 DGT Method Summary
- Attachment 2 Vane Shear Log Form

ABBREVIATIONS

DGT	diffusive gradient thin film
Ecology	Washington State Department of Ecology
PDI	pre-design investigation
SMA	sediment management area
SQAPP	Sampling and Quality Assurance Project Plan

1 Introduction

This document has been produced as an Addendum to the *Sampling Quality Assurance and Project Plan* (SQAPP) approved by the Washington State Department of Ecology (Ecology) as part of the *Remedial Investigation/Feasibility Study Work Plan* (Anchor QEA 2017). The Addendum is limited to updating sampling and analytical methods that were not included in or have changed since the SQAPP was developed. Refer to the Ecology-approved SQAPP for matters not directly addressed in this Addendum.

2 Data Generation and Acquisition

This section describes data to be acquired as documented in the *Pre-Remedial Design Investigation Work Plan* (Anchor QEA in preparation). Discrete target investigation locations are depicted for each sediment management area (SMA) in Figures 1 through 3. Target coordinates for the locations and required analysis are included in Table 1. Analytical methods for the testing required under this program are included in Table 2.

2.1 Bathymetric Survey

While rough bathymetric data for Shelton Harbor exist, they do not exist at the quality necessary for effective pre-design investigation (PDI) sample planning and remedial design. A bathymetric survey will be conducted for the interim action areas. The survey will be performed by a qualified subcontractor under the direction of Anchor QEA, LLC, and the survey data will be supplied following the completion of the survey.

2.2 Field Sampling Methods

This section describes the methodology for positioning, sample collection, processing, identification, documentation, equipment decontamination, and handling of investigation-derived waste for the field investigation.

2.2.1 *Sampling Vessels and Field Equipment*

An appropriately outfitted research vessel will be used to collect surface sediment samples. Sediment will be collected from the research vessel using a modified powered Van Veen device.

2.2.2 *Surface Sediment Collection and Processing*

Surface sediment will be collected and processed as described in the following sections. Bulk surface sediment will be collected and submitted for testing in accordance with the total organic carbon, dioxin/furan, copper, and tributyltin methods included in Table 2. Porewater sulfide analysis will be conducted by diffusive gradient thin film (DGT) in accordance with the optical densitometry methods detailed in Attachment 1.

2.2.3 *Surface Sediment Sample Collection Procedures*

This section describes surface sediment sampling required by the PDI. Sampling under this program may be conducted by Van Veen sampler deployed from a vessel or by hand if accessible during low tide. A subset of locations will undergo testing for porewater sulfide using a DGT apparatus, which is also described in this section.

2.2.3.1 Diffusive Gradient Thin Film Sampling Location Procedure

At stations where porewater sulfide characterization is required, use the following procedure:

- Immediately after accepting or hand-collecting the grab, collect approximately 0.5 gallon of sediment from the 0- to 10-centimeter interval and place directly into a plastic bag.
- Immediately place the DGT piston into the bag containing the sediment.
- Squeeze all head space from the bag, seal it, and place it into a Mylar bag with oxygen-scavenging packets and ensure the DGT piston is completely covered.
- Take the temperature of the sediment, pH, and salinity and note data on the field form.
- Agitate the bag every couple of hours to refresh the DGT surface during the 24-hour exposure duration.
- After the exposure duration is complete, remove the DGT from the bag, rinse with distilled water, and place the DGT in foil and in a labeled plastic bag.
- Transport the bags to Anchor QEA's Portland, Oregon, laboratory for analysis.

Triplicate grabs will be collected at two locations (Table 1). At these locations, three separate grabs will be taken from the target location. If the grab appears to have re-entered the previous grab location, it will be rejected and the sampler will be moved several feet from the original location.

2.2.3.2 Subtidal Grab Collection

Surface sediment will be collected by Van Veen sampler according to the procedures included in the SQAPP. The 0- to 10-centimeter mass will be retained on deck using the following procedure:

- Remove the remaining material from the desired grab depth (0 to 10 centimeters) into a clean stainless-steel pot. To avoid cross-contamination, take care to remove only sediment that has not come into contact with the sides or bottom of the grab.
- When all material has been removed from the desired interval, place sediment in a sealed holding container, label it, and place it on ice on the deck of the collection vessel for upland processing.

Triplicate grabs will be collected at select locations (Table 1). At these locations, three separate power grabs will be taken from the target location. If the grab appears to have re-entered the previous grab location, it will be rejected and the sampler will be moved several feet from the original location.

2.2.3.3 Intertidal Grab Collection

At stations that are accessible at low tide, sample collection will be conducted by hand as follows:

- Navigate to the target location.
- Characterize the sediment as follows:
 - Physical soil description of the grab in accordance with the Unified Soil Classification System (includes soil type, density/consistency of soil, moisture, and color)

- Odor (e.g., hydrogen sulfide and petroleum)
- Note any vegetation
- Debris
- Biological activity (e.g., detritus, shells, tubes, bioturbation, or live or dead organisms)
- Presence of oil sheen
- Any other distinguishing characteristics or features
- Using a decontaminated stainless-steel spoon or trowel, excavate material to depth of 10 centimeters and place the material in a decontaminated stainless-steel pot.
- At location SMA-2-SG04, the surface sediment sample will be composed of a six-point composite, comprised of three subsamples collected from approximately 5 feet apart in-between each rail pair alignment.
- Label the holding container, store it on ice, and transport it to the upland processing location.

2.2.3.4 Upland Surface Sediment Sample Processing Procedures

Sediment grab processing will be conducted at an upland processing area. All working surfaces and instruments will be thoroughly cleaned, decontaminated, and prepared to minimize cross-contamination between sampling stations. Disposable gloves will be discarded after processing each station and replaced prior to handling decontaminated instruments or work surfaces. The steps for processing the samples are as follows:

- Open the one sealed container and record time of processing.
- Photograph the unhomogenized sample.
- Homogenize with a decontaminated stainless-steel paddle and drill.
- Photograph the homogenized sample.
- Using a clean stainless-steel spoon, completely fill pre-labeled sample containers as specified in Table 3.
- Immediately after filling the sample container with sediment, place the screw cap on the sample container and tighten.
- Thoroughly check all sample containers for proper identification, analysis type, and lid tightness.
- Pack each container carefully to prevent breakage and place inside a cooler with ice for storage at the proper temperature ($4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for all samples).
- In accordance with the SQAPP, a homogenization duplicate (from a single homogenate) will be collected once for every 20 samples.

Samples will be submitted for testing as presented in Table 1.

2.2.4 *Subsurface Sediment Collection*

Subsurface sediment locations will either be accessed by vessel during high tide or by land during low tide. Access to each location will be determined in the field to maximize sampling efficiency. Sampling from the vessel during high tide will be done using a polycarbonate tube and by advancing the tube using a slide hammer. If sample locations are accessed by land, they will be sampled using a similar polycarbonate tube and advanced using a mini-vibracore head.

2.2.4.1 **Geotechnical Core Collection Procedures**

The target coordinates for each subsurface station are included in Table 1 but will be adjusted in the field based on accessibility. The target depth will be 3 to 6 feet below the mudline. The core will be driven to its maximum length or to refusal. Acceptance criteria for a sediment core sample are as follows:

- The core penetrated and retained material to project depth.
- Cored material did not extend out of the top of the core tube or contact any part of the sampling apparatus at the top of the core tube.
- There were no obstructions in the cored material that might have blocked the subsequent entry of sediment into the core tube and resulted in incomplete core collection.
- If multiple core rejections (three attempts) require the core station to be relocated, the proposed station relocation will be documented and the actual coordinates will be recorded after sample collection is complete. Recovered cores will be cut, if necessary, placed on ice, and processed at the analytical laboratory (or equivalent).

Logs and field notes of all core samples will be maintained as samples are collected and correlated to the sampling location map. The following information will be included in the logs:

- Elevation of each station sampled as measured from mean lower low water
- Location of each station as determined by differential global positioning system
- Date and time of collection of each sediment core sample
- Names of the field coordinator and person(s) collecting and handling the sample
- Observations made during sample collection, including complications and other details associated with the sampling effort
- The sample station identification
- Length and depth intervals of each core section and estimated recovery for each sediment sample as measured from mean lower low water
- Qualitative notation of apparent resistance of sediment column to coring
- Any deviation from the approved SQAPP

2.2.4.2 Geotechnical Core Processing Procedures

Sediment core processing will be conducted in the same upland location as the surface sediment samples. Transported cores will be handled consistent with ASTM International procedures (ASTM D4220) and stored upright in the analytical laboratory refrigerators or on site until processed. Filled sample containers will be stored in coolers containing ice to maintain the samples at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ until delivery or shipping to the analytical laboratory.

The steps for processing the samples are listed as follows:

1. Cut the core longitudinally using a circular saw or power shears, taking care not to penetrate the sediment while cutting.
2. Use appropriate utensils to split the core to expose the center of the two halves for sampling.
3. Photograph the entire length of the core.
4. Record the description of the core sample on the core log form for the following parameters as appropriate and present:
 - a. Sample recovery (depth in feet of penetration and sample compaction)
 - b. Physical soil description in accordance with the Unified Soil Classification System (includes soil major and minor constituents, density/consistency of soil, moisture content, and color) in accordance with ASTM D2488
 - c. Odor (e.g., hydrogen sulfide or petroleum)
 - d. Vegetation
 - e. Debris
 - f. Biological activity (e.g., detritus, shells, tubes, bioturbation, or live or dead organisms)
 - g. Visual stratification, structure, and texture
 - h. Presence of oil sheen
 - i. Any other distinguishing characteristics or features
5. The field engineer will determine sampling intervals so that a representative number of tests are conducted for each major soil type encountered during the investigation.
6. Once sampling intervals are determined, a representative sample will be removed from the core and filled into the appropriate sample container.
7. Immediately after filling the sample container with sediment, place the screw cap on the sample container and tighten. Label the sample container appropriately (Table 2).
8. Thoroughly check all sample containers for proper identification, analysis type, and lid tightness.
9. Pack each container carefully to prevent breakage and place inside of a cooler with ice for storage at the proper temperature ($4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for all samples).

2.2.5 Vane Shear Testing Procedures

Access to the vane shear testing locations will be conducted in the same manner as the subsurface sediment locations (Section 2.2.4). Once on location, the steps for testing are as follows:

1. If testing is conducted from a vessel, measure and record the depth of water.
2. Assemble the vane shear testing apparatus. If working from a vessel, be sure to add enough length to the apparatus to extend to the first testing depth. Additional rods can be added between tests.
3. The field engineer will select the appropriate vane for the sediment composition.
4. Insert the vane until it reaches the appropriate testing depth. Avoid pounding or vibrating the vane.
5. Zero the slip collar on the apparatus.
6. Rotate the apparatus clockwise at a rate of 1 degree per 6 minutes.
7. Continue rotating the apparatus until the sediment fails and the vane begins to rotate with the apparatus.
8. Observe and record the maximum torque applied to the vane.
9. Rapidly rotate the apparatus 5 full revolutions.
10. Re-zero the apparatus. Repeat steps 6 through 8 to determine the remolded shear strength.
11. Advance approximately 1 foot further and repeat steps 4 through 10.
12. Continue to advance the vane and test until the vane has been advanced 6 feet below mudline or refusal is encountered.
13. Record all measurements taken on the field form included as Attachment 2.

2.2.6 Upland Geotechnical Boring Collection Procedures

Following utility locates, geotechnical borings will be advanced under the direction of a geotechnical engineer, processed in the vicinity boring operation, and submitted for testing, as necessary, for physical tests (Table 2).

2.2.7 Horizontal Positioning and Vertical Control

Refer to the SQAPP for horizontal positioning of the sample vessel and vertical control of the sample. Target coordinates for each sampling location are included in Table 1. As-collected coordinates will be recorded at each station.

2.2.8 Sample Station Locations and Sample Identification

Figures 1 through 3 depict the target locations of the proposed grab, core, vane shear test, and DGT samples. Table 1 includes listings of all station locations, sample identifiers, and analysis or testing required for each location. The sample identification schemes are described in Section 2.2.11.

2.2.9 Equipment Decontamination Procedures

Refer to the SQAPP for equipment decontamination procedures.

2.2.10 Sample Containers for Analysis

The contract laboratory will provide certified, pre-cleaned, U.S. Environmental Protection Agency-approved containers for all chemistry and geotechnical samples. Sediment for ex situ DGT testing will be placed in commercially available food-grade polyethylene bags and sealed airtight. Table 3 lists container size, holding times, and preservation for the categories of analytes. At a minimum, each sample container will be labelled with the following information:

- Project name and number
- Sample identifier
- Date and time of sample collection
- Initials of field personnel responsible for sample collection
- Analyses required
- Preservative type (if applicable)

2.2.11 Sample Identification

Each sample will be assigned a unique alphanumeric identifier using the following format:

- The first three characters identify the type of investigation (i.e., PDI for pre-design investigation).
- The next three characters identify the sediment management location (i.e., SMA1, SMA2, or SMA3).
- The next characters identify the collection method and location:
 - SG for surface sediment grab
 - PC for sediment push core
 - VST for vane sheet test
 - DGT for diffusive gradient thin film test locations
 - A two-digit location ID will follow the method ID (i.e., 01, 02, 03, and so on)
- For sediment cores, a field will be included to identify the depth interval in feet. The top 4 feet of the core would be identified as 0004.
- The last characters will identify the sampling date (YYMMDD).

Sample “PDI-SMA1-SG01-180418” represents a pre-design investigation sediment grab collected inside of sediment management area 1 at location 01 on April 18, 2018.

Sample “PDI-SMA3-PC07-0002-180420” represents a pre-design investigation sediment core collected inside sediment management area three at location seven and sampled from 0 to 2 feet on April 18, 2018.

Field duplicate or triplicate samples will be identified by adding 100 or 1000 to the location ID, respectively. Duplicate sample "PDI-SMA1-SG103-180419" is the field duplicate of sample "PDI-SMA1-SG03-180419." Triplicate sample "PDI-SMA1-SG1003-180419" is the field triplicate of sample "PDI-SMA1-SG03-180419."

3 Field and Laboratory Quality Assurance and Quality Control

This section describes the quality assurance/quality control procedures specific to the PDI. For quality assurance/quality control procedures not listed, refer to the SQAPP.

3.1 Field Quality Assurance and Quality Control

3.1.1 Field Duplicates and Triplicates

In consultation with Ecology, two predetermined locations have been selected to undergo triplicate testing (three separate grabs, processing and analytical samples) to better understand small-scale variability in dioxin/furan results (Table 1). As described in Section 2.2.3.4, electrotechnical homogenization will be conducted to reduce variability in the sample matrix.

Field homogenization duplicates will be collected and tested in accordance with the SQAPP. No data will be qualified based solely on field duplicate precision.

3.1.2 Equipment Blanks

A rinse blank will be collected from the sediment processing equipment used to prepare the sample in accordance with the procedures in the SQAPP.

3.2 Analytical Laboratory Quality Assurance and Quality Control

An updated laboratory quality assurance/quality control analysis summary is provided in Table 4, and laboratory data quality objectives are detailed in Table 5.

4 Documentation, Recordkeeping, and Reporting Requirements

Refer to the SQAPP for all documentation, recordkeeping, and reporting requirements.

5 Data Validation and Usability

Refer to the SQAPP for data validation and usability procedures.

6 References

Anchor QEA (Anchor QEA, LLC), in preparation. *Pre-Remedial Design Investigation Work Plan*.

Prepared for Simpson Timber Company and the Washington State Department of Ecology.

In preparation.

Anchor QEA, 2017. *Sampling and Quality Assurance Project Plan – Remedial Investigation/Feasibility Study Work Plan*. Prepared for Simpson Timber Company and the Washington State Department of Ecology. June 2017.

Tables

Table 1
Target PDI Locations

Location ID	Latitude	Longitude	Triplicate Station	Dioxin/Furan	Copper	Tributyltin	Total Organic Carbon	Porewater Sulfide	Geotechnical	Archive
SMA-1										
PDI-SMA1-SG01	-123.0909041	47.21270905	--	X	--	--	--	--	--	--
PDI-SMA1-SG02	-123.0903689	47.21289926	--	X	--	--	--	--	--	--
PDI-SMA1-SG03	-123.0898338	47.21308946	--	X	--	--	--	--	--	--
PDI-SMA1-SG04	-123.0895547	47.21272482	--	X	--	--	--	--	--	--
PDI-SMA1-SG05	-123.0892755	47.21236019	--	X	--	--	--	--	--	--
PDI-SMA1-SG06	-123.0898107	47.21216999	--	X	--	--	--	--	--	--
PDI-SMA1-SG07	-123.0903458	47.21197979	X	X	--	--	--	--	--	--
PDI-SMA1-SG08	-123.0906249	47.21234442	--	X	--	--	--	--	--	--
PDI-SMA1-SG09	-123.0906481	47.21326389	--	X	--	--	--	--	--	--
PDI-SMA1-SG10	-123.0892987	47.21327965	--	X	--	--	--	--	--	--
PDI-SMA1-SG11	-123.0890195	47.21291502	--	X	--	--	--	--	--	--
PDI-SMA1-SG12	-123.0887404	47.21255038	--	X	--	--	--	--	--	--
PDI-SMA1-SG13	-123.0889964	47.21199556	--	X	--	--	--	--	--	--
PDI-SMA1-SG14	-123.0895315	47.21180536	--	X	--	--	--	--	--	--
PDI-SMA1-SG15	-123.0900667	47.21161516	--	X	--	--	--	--	--	--
PDI-SMA1-SG16	-123.0908809	47.21178959	--	X	--	--	--	--	--	--
PDI-SMA1-SG17	-123.09116	47.21215422	--	X	--	--	--	--	--	--
PDI-SMA1-SG18	-123.0914392	47.21251885	--	X	--	--	--	--	--	--
PDI-SMA1-SG19	-123.0911832	47.21307368	--	X	--	--	--	--	--	--
PDI-SMA1-SG20	-123.091718	47.212883	--	X	--	--	--	--	--	--
PDI-SMA1-PC01	-123.0898087	47.2131155	--	--	--	--	--	--	X	--
PDI-SMA1-PC02	-123.0893311	47.21236256	--	--	--	--	--	--	X	--
PDI-SMA1-PC03	-123.0906159	47.21238083	--	--	--	--	--	--	X	--
PDI-SMA1-VST01	-123.0897858	47.21309294	--	--	--	--	--	--	X	--
PDI-SMA1-VST02	-123.0892661	47.21238783	--	--	--	--	--	--	X	--
PDI-SMA1-VST03	-123.0906578	47.21232831	--	--	--	--	--	--	X	--
SMA-2										
PDI-SMA2-SG01	-123.0880113	47.21376898	--	--	X	X	X	--	--	--
PDI-SMA2-SG02	-123.087689	47.213746	--	X	X	X	X	--	--	--
PDI-SMA2-SG03	-123.087375	47.2137	--	X	X	X	X	--	--	--
PDI-SMA2-SG04	-123.0880377	47.21368464	--	--	X	X	X	--	--	--
PDI-SMA2-SG05	-123.0880594	47.21361559	--	--	X	X	X	--	--	--
PDI-SMA2-SG06	-123.087442	47.213486	--	X	X	X	X	--	--	--
PDI-SMA2-SG07	-123.087757	47.213532	--	X	X	X	X	--	--	--
PDI-SMA2-SG08	-123.0876557	47.21385336	--	--	--	--	--	--	--	X
PDI-SMA2-SG09	-123.0873409	47.21380758	--	--	--	--	--	--	--	X
PDI-SMA2-SG10	-123.0870261	47.2137618	--	--	--	--	--	--	--	X
PDI-SMA2-SG11	-123.0870933	47.21354729	--	--	--	--	--	--	--	X
PDI-SMA2-SG12	-123.0871605	47.21333279	--	--	--	--	--	--	--	X
PDI-SMA2-SG13	-123.0874753	47.21337857	--	X	X	X	X	--	--	--

Table 1
Target PDI Locations

Location ID	Latitude	Longitude	Triplicate Station	Dioxin/Furan	Copper	Tributyltin	Total Organic Carbon	Porewater Sulfide	Geotechnical	Archive
PDI-SMA2-SG14	-123.0877901	47.21342436	--	--	--	--	--	--	--	X
PDI-SMA2-SG15	-123.0881049	47.21347014	--	--	--	--	--	--	--	X
PDI-SMA2-PC01	-123.0880071	47.21369373	--	--	--	--	--	--	X	--
PDI-SMA2-PC02	-123.0874553	47.21353657	--	--	--	--	--	--	X	--
PDI-SMA2-VST01	-123.0880052	47.21366951	--	--	--	--	--	--	X	--
PDI-SMA2-VST02	-123.087463	47.21351371	--	--	--	--	--	--	X	--
SMA-3										
PDI-SMA3-DGT01	-123.0941336	47.20714822	--	--	--	--	--	X	--	--
PDI-SMA3-DGT02	-123.0941119	47.20722711	X	--	--	--	--	X	--	--
PDI-SMA3-DGT03	-123.0934393	47.20715217	--	--	--	--	--	X	--	--
PDI-SMA3-DGT04	-123.0933316	47.20721557	--	--	--	--	--	X	--	--
PDI-SMA3-DGT05	-123.0927635	47.20650238	X	--	--	--	--	X	--	--
PDI-SMA3-DGT06	-123.0926787	47.20653616	--	--	--	--	--	X	--	--
PDI-SMA3-SG01	-123.0943419	47.20776733	--	X	--	--	--	--	--	--
PDI-SMA3-SG02	-123.093514	47.20762609	--	X	--	--	--	--	--	--
PDI-SMA3-SG03	-123.0926861	47.20748485	--	X	--	--	--	X	--	--
PDI-SMA3-SG04	-123.0923758	47.20713213	X	X	--	--	--	--	--	--
PDI-SMA3-SG05	-123.0920655	47.20677941	--	X	--	--	--	X	--	--
PDI-SMA3-SG06	-123.0917552	47.20642668	--	X	--	--	--	--	--	--
PDI-SMA3-SG07	-123.0940316	47.20741462	--	--	--	--	--	X	--	X
PDI-SMA3-SG08	-123.0932037	47.20727338	--	--	--	--	--	X	--	X
PDI-SMA3-SG09	-123.0928934	47.20692066	--	--	--	--	--	--	--	X
PDI-SMA3-SG10	-123.0925831	47.20656794	--	--	--	--	--	X	--	X
PDI-SMA3-SG11	-123.0912376	47.20663815	--	--	--	--	--	--	--	X
PDI-SMA3-SG12	-123.0915479	47.20699087	--	--	--	--	--	X	--	X
PDI-SMA3-SG13	-123.0918582	47.20734359	--	--	--	--	--	--	--	X
PDI-SMA3-SG14	-123.0921685	47.20769632	--	--	--	--	--	X	--	X
PDI-SMA3-SG15	-123.0924788	47.20804904	--	--	--	--	--	--	--	X
PDI-SMA3-SG16	-123.0929964	47.20783757	--	--	--	--	--	--	--	X
PDI-SMA3-SG17	-123.0933068	47.20819028	--	--	--	--	--	--	--	X
PDI-SMA3-SG18	-123.0938244	47.20797881	--	--	--	--	--	X	--	X
PDI-SMA3-SG19	-123.0936171	47.208543	--	--	--	--	--	X	--	X
PDI-SMA3-SG20	-123.0946523	47.20812005	--	--	--	--	--	--	--	X
PDI-SMA3-PC01	-123.0938707	47.2075206	--	--	--	--	--	--	X	--
PDI-SMA3-PC02	-123.0939979	47.20726951	--	--	--	--	--	--	X	--
PDI-SMA3-PC03	-123.0933154	47.20747687	--	--	--	--	--	--	X	--
PDI-SMA3-PC04	-123.0932353	47.20722087	--	--	--	--	--	--	X	--
PDI-SMA3-PC05	-123.0925023	47.20714404	--	--	--	--	--	--	X	--
PDI-SMA3-PC06	-123.092648	47.20675831	--	--	--	--	--	--	X	--
PDI-SMA3-VST01	-123.0939074	47.20751746	--	--	--	--	--	--	X	--
PDI-SMA3-VST02	-123.094032	47.20726885	--	--	--	--	--	--	X	--

Table 1
Target PDI Locations

Location ID	Latitude	Longitude	Triplicate Station	Dioxin/Furan	Copper	Tributyltin	Total Organic Carbon	Porewater Sulfide	Geotechnical	Archive
PDI-SMA3-VST03	-123.093355	47.20748346	--	--	--	--	--	--	X	--
PDI-SMA3-VST04	-123.0932611	47.20723048	--	--	--	--	--	--	X	--
PDI-SMA3-VST05	-123.0925229	47.20716354	--	--	--	--	--	--	X	--
PDI-SMA3-VST06	-123.0926753	47.20677332	--	--	--	--	--	--	X	--
PDI-SMA3-VST07	-123.0926766	47.20647136	--	--	--	--	--	--	X	--
PDI-SMA3-VST08	-123.0922907	47.20644291	--	--	--	--	--	--	X	--
PDI-SMA3-SPT01	-123.0935816	47.20710271	--	--	--	--	--	--	X	--
PDI-SMA3-SPT02	-123.092914	47.20666548	--	--	--	--	--	--	X	--

Notes:

--: not applicable for testing

PDI: pre-design investigation

X: location to be submitted for testing

Table 2
Parameters for Analysis, Methods, and Target Quantitation Limits

Parameter	Method	Target Reporting Limit
Conventionals (%)		
Total organic carbon	EPA 9060M	0.10
Total solids	EPA 160.3	0.10
Geotechnical Parameters (%)		
Moisture content	ASTM D2216	0.10
Atterberg limits	ASTM D4318	0.10
Bulk density	ASTM D2937	0.10
One-dimensional consolidation	ASTM D2435	N/A
Unconsolidated, undrained triaxial shear strength	ASTM D2850	N/A
Vane shear testing	ASTM D2573	N/A
Grain size	ASTM D422	0.10
Organotins (µg/kg)		
Di-n-butyltin	GC/MS	1.0
n-Butyltin	GC/MS	1.0
Tetra-n-butyltin	GC/MS	1.0
Tri-n-butyltin	GC/MS	1.0
Metals (mg/kg)		
Copper	EPA 6020A	1.0
Dioxin/Furans (ng/kg)		
Dioxins		
2,3,7,8-TCDD	EPA 1613B	0.5
1,2,3,7,8-PeCDD	EPA 1613B	2.5
1,2,3,4,7,8-HxCDD	EPA 1613B	2.5
1,2,3,6,7,8-HxCDD	EPA 1613B	2.5
1,2,3,7,8,9-HxCDD	EPA 1613B	2.5
1,2,3,4,6,7,8-HpCDD	EPA 1613B	2.5
OCDD	EPA 1613B	5.00
Furans		
2,3,7,8-TCDF	EPA 1613B	0.5
1,2,3,7,8-PeCDF	EPA 1613B	2.5
2,3,4,7,8,-PeCDF	EPA 1613B	2.5
1,2,3,4,7,8-HxCDF	EPA 1613B	2.5
1,2,3,6,7,8-HxCDF	EPA 1613B	2.5
1,2,3,7,8,9-HxCDF	EPA 1613B	2.5
2,3,4,6,7,8-HxCDF	EPA 1613B	2.5
1,2,3,4,6,7,8-HpCDF	EPA 1613B	2.5
1,2,3,4,7,8,9-HpCDF	EPA 1613B	2.5
OCDF	EPA 1613B	5
Porewater Conventionals (mg/L)		
DGT Sulfide	Densitometry	0.01

Notes:

GC/MS per Dredged Material Management Office by Grignard derivatization and analysis by GC/MS (e.g., Krone et al. 1989).

‰: percent

µg/kg: micrograms per kilogram

ASTM: ASTM International

Table 2
Parameters for Analysis, Methods, and Target Quantitation Limits

DGT: diffusive gradient thin film

EPA: U.S. Environmental Protection Agency

GC/MS: gas-chromatography and mass spectrometry

mg/kg: milligrams per kilogram

mg/L: milligrams per liter

N/A: not applicable

ng/kg: nanograms per kilogram

Table 3
Guidelines for Sample Handling and Storage

Parameter	Sample Size	Container Size and Type ¹	Holding Time	Preservative
Moisture content	150 g	16-oz jar or 1-quart zip-top bag	None	None
Atterberg limits			None	None
Bulk density	3 to 5 feet	Shelby tube	None	None
One-dimensional consolidation			None	None
Unconsolidated, undrained triaxial shear strength			None	None
Vane shear testing	In-situ	N/A	None	None
Grain size	150 g	16-oz HDPE	None	None
Total solids	50 g	8-oz glass	None	Cool/4°C
Total organic carbon	50 g	From TS container	28 days	Cool/4°C
			6 months	Freeze -18°C
Organotins	150 g	8-oz glass	14 days until extraction	Cool/4°C
			1 year until extraction	Freeze -18°C
			40 days after extraction	Cool/4°C
Dioxin/furans	150 g	8-oz amber glass	None	Cool/4°C
DGT sediment	0.5 gallon	Large plastic bag	7 days	Cool/4°C

Notes:

1. All sample containers will have lids with Teflon inserts.

DGT: diffusive gradient thin film

g: gram

HDPE: high-density polyethylene

oz: ounce

TS: total solids

Table 4
Laboratory Quality Assurance/Quality Control Analysis Summary

Analysis Type	Initial Calibration	Ongoing Calibration	Replicates	Matrix Spikes	LCS/Blank Spike	Matrix Spike Duplicates	Method Blanks	Surrogate Spikes
Total solids	Daily or each batch ¹	N/A	1 per 20 samples	N/A	N/A	N/A	N/A	N/A
Total organic carbon	Daily or each batch	1 per 10 samples	1 per 20 samples	1 per 20 samples	1 per 20 samples	N/A	Each batch	N/A
Organotins	As needed ³	Every 12 hours	N/A	1 per 20 samples	1 per 20 samples	1 per 20 samples	Each batch	Each sample
Dioxin/furans	As needed ³	Every 12 hours	1 per 20 samples	N/A ⁴	1 per 20 samples	N/A ⁴	Each batch	Each sample
DGT sulfide	Daily or each batch	1 per 10 samples	N/A	N/A	N/A	N/A	Each batch	N/A

Notes:

1. Calibration and certification of drying ovens and weighing scales are conducted biannually.
2. A matrix spike duplicate may be analyzed in lieu of a sample replicate.
3. Initial calibrations are considered valid until the ongoing continuing calibration no longer meets method specifications. At that point, a new initial calibration is performed.
4. Isotope dilution required by the method.

DGT: diffusive gradient thin film

LCS: laboratory control sample

N/A: not applicable

Table 5
Data Quality Objectives

Parameter	Precision (Percentage)	Accuracy¹ (Percentage)	Completeness (Percentage)
Total organic carbon	± 30 RPD	75 to 125 R	95
Total solids	± 20 RPD	N/A	95
Organotins	± 35 RPD	50 to 150 R	95
Dioxin/furans	± 35 RPD	50 to 150 R	95

Notes:

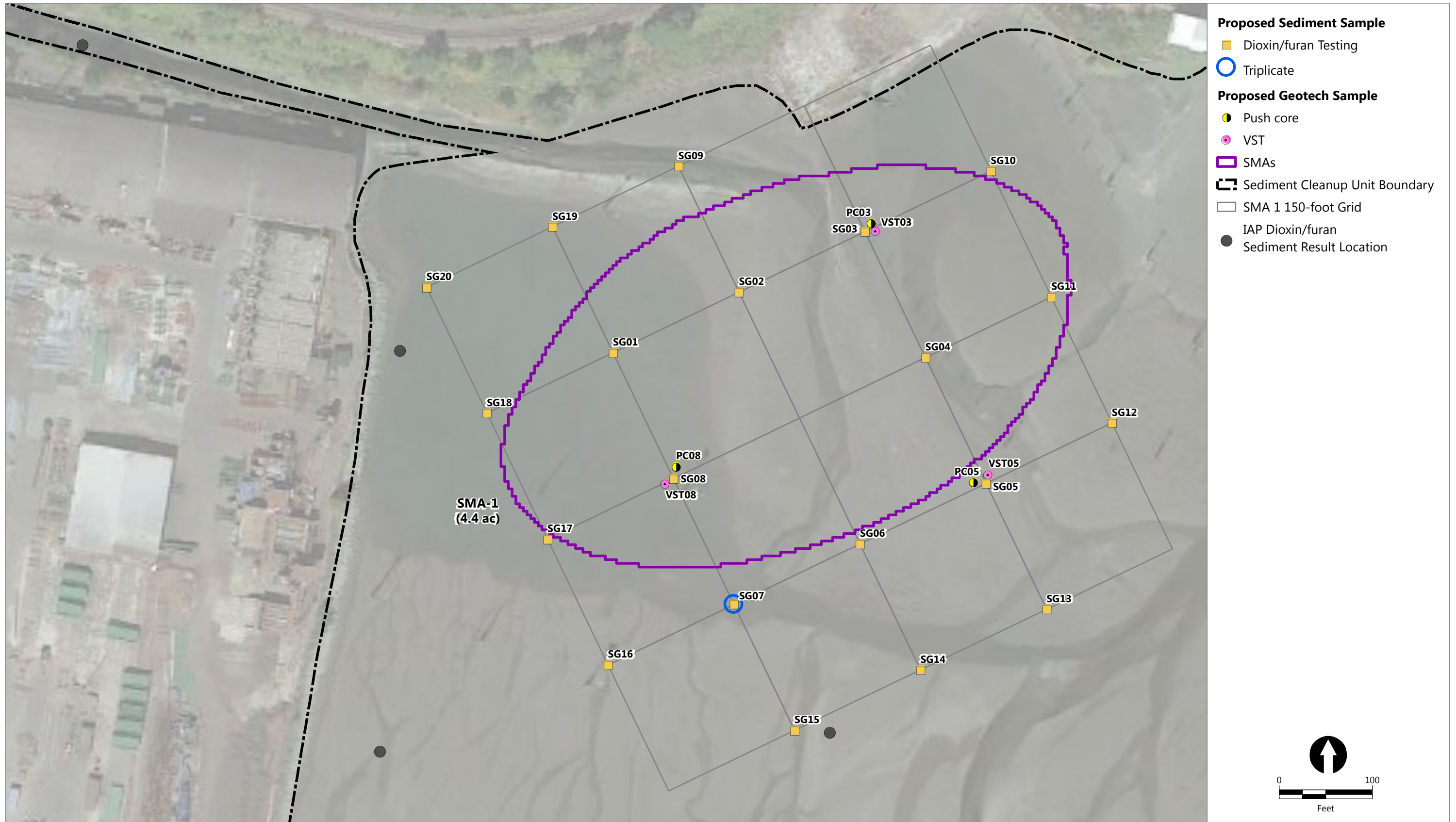
1. Laboratory control sample and matrix spike/matrix spike duplicate percent recovery.

N/A: not applicable

R: recovery

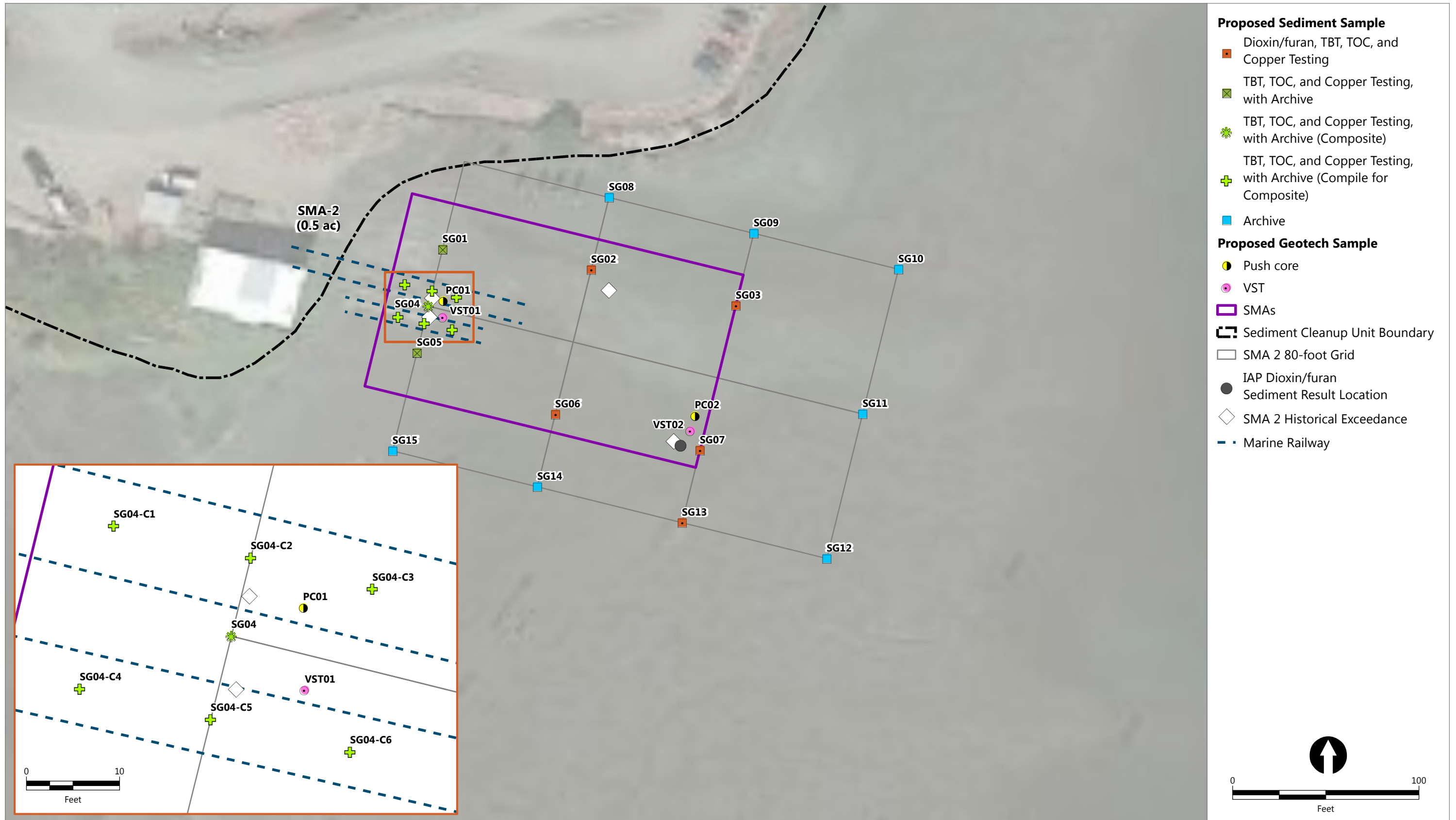
RPD: relative percent difference

Figures



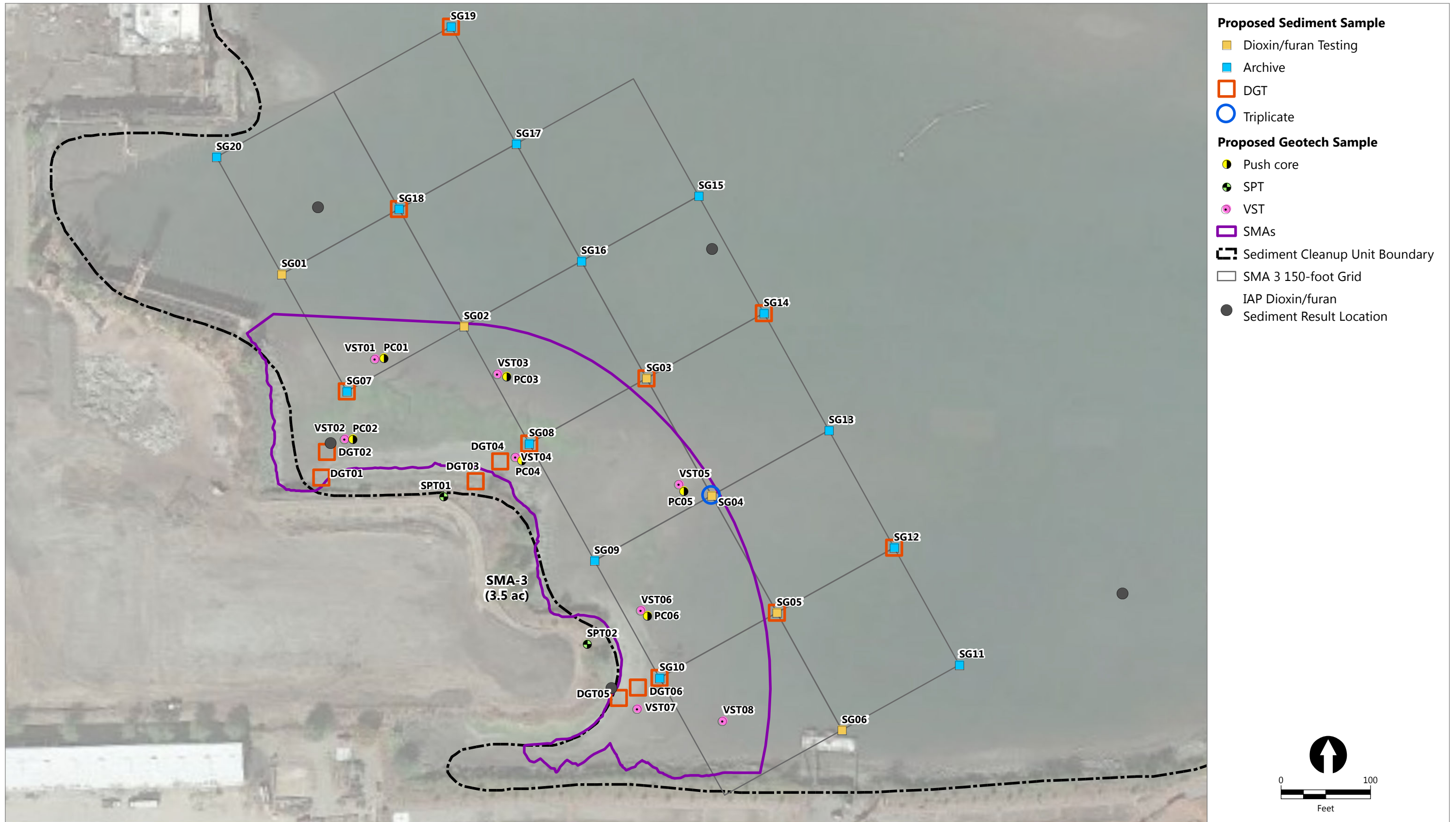
Publish Date: 2018/04/05, 3:38 PM | User: ckiblinger
 Filepath: \\orcas\gis\Jobs\Simpson_Timber_0008\SheltonHarbor\Maps\PDIWP\SQAPP_Attchmt\Shelton_SMA_SProposed_Sample_Locs_PDIWP_SQAPP_DDP.mxd

Figure 1
SMA-1
 Shelton Harbor Pre-Design Investigation Work Plan
 Oakland Bay and Shelton Harbor Sediments Cleanup Site



Publish Date: 2018/04/11, 11:12 AM | User: ckiblinger
 Filepath: \\orcas\gis\Jobs\Simpson_Timber_0008\SheltonHarbor\Maps\PDIWP\SQAPP_Attchmt\Shelton_SMA2_Proposed_Sample_Locs_PDIWP_SQAPP_SMA2_DDP.mxd

Figure 2
SMA-2
 Shelton Harbor Pre-Design Investigation Work Plan
 Oakland Bay and Shelton Harbor Sediments Cleanup Site



Publish Date: 2018/04/05, 3:39 PM | User: ckiblinger
 Filepath: \\orcas\gis\Jobs\Simpson_Timber_0008\SheltonHarbor\Maps\PDIWP\SQAPP_Attchmt\Shelton_SMA3_Proposed_Sample_Locs_PDIWP_SQAPP_DDP.mxd

Figure 3
SMA-3
 Shelton Harbor Pre-Design Investigation Work Plan
 Oakland Bay and Shelton Harbor Sediments Cleanup Site

Attachment 1

DGT Method Summary

Memorandum

November 10, 2017

To: Clay Patmont

From: Masa Kanematsu, PhD; Dimitri Vlassopoulos, PhD

Re: Method Development and Verification Study for Sulfide Measurement in Porewater Using Diffusive-Gradients-in-Thin-Films (DGT)

This document presents the results of a laboratory verification study of a passive porewater sampling technique for dissolved sulfide using Diffusive Gradients in Thin-films (DGT). The method, developed over the last two decades (Teasdale et al. 1999; Rearick et al. 2005), is finding increasing use as a reliable in situ technique for quantifying sulfide levels in sediment porewater. The method is based on the reaction of sulfide with silver iodide (AgI), a white powder impregnated in a gel to produce silver sulfide (Ag₂S), a black solid. The intensity of the color developed is proportional to the amount of sulfide accumulated in the gel.

For this study, we developed a calibration curve using optical densitometry (OD) to quantify the amount of sulfide accumulated in the DGT device over a dissolved sulfide concentration range of 0 to 6 mg/L sulfide. To verify the DGT technique, sulfide was extracted from exposed DGTs and quantified using two different extraction and analysis methods:

- Extraction of chromium reducible sulfur (CRS) from the DGT gel and analysis by sulfide ion-specific electrode
- Extraction of the DGT gel by nitric acid/hydrogen peroxide (commonly used in selective sequential extraction schemes to quantify metals associated with sulfides) and analysis of total sulfur released by ICP-MS

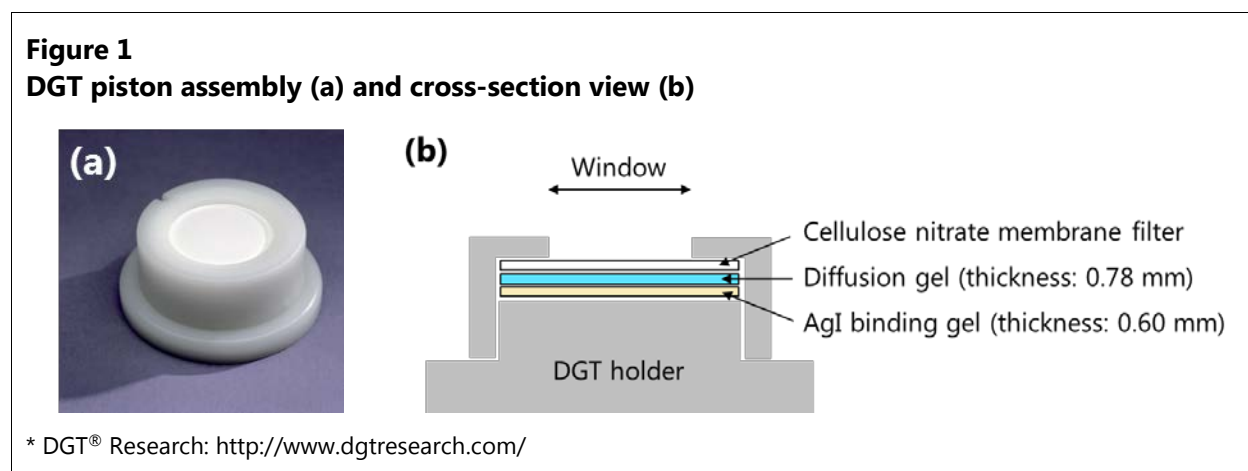
Results from both methods were in excellent agreement and quantitative recovery of sulfide from the DGT gels was confirmed by comparison with the theoretical mass of accumulated sulfide calculated using a mass-transfer model. Sulfide recoveries from the DGT gels averaged 101.0% and 96.9% for the CRS and nitric acid/peroxide extraction methods, respectively.

Materials and Methods

DGT Samplers

DGT piston devices for deployment in soil were obtained from DGT[®] Research (<http://www.dgtresearch.com>). The DGT samplers were preloaded for sulfide measurement and

consist of a standard DGT holder containing a 0.6 mm thick silver iodide (AgI) impregnated binding gel layer, overlain by a 0.78 mm thick polyacrylamide diffusive gel, and held in place by a 0.45 μm cellulose nitrate membrane filter (Figure 1). The window size of the DGT sampler is 2.54 cm^2 . Prior to use, the DGT assemblies were deoxygenated by immersion in 0.01 M sodium nitrate purged with high-purity nitrogen gas for at least 2 days to remove any residual oxygen.



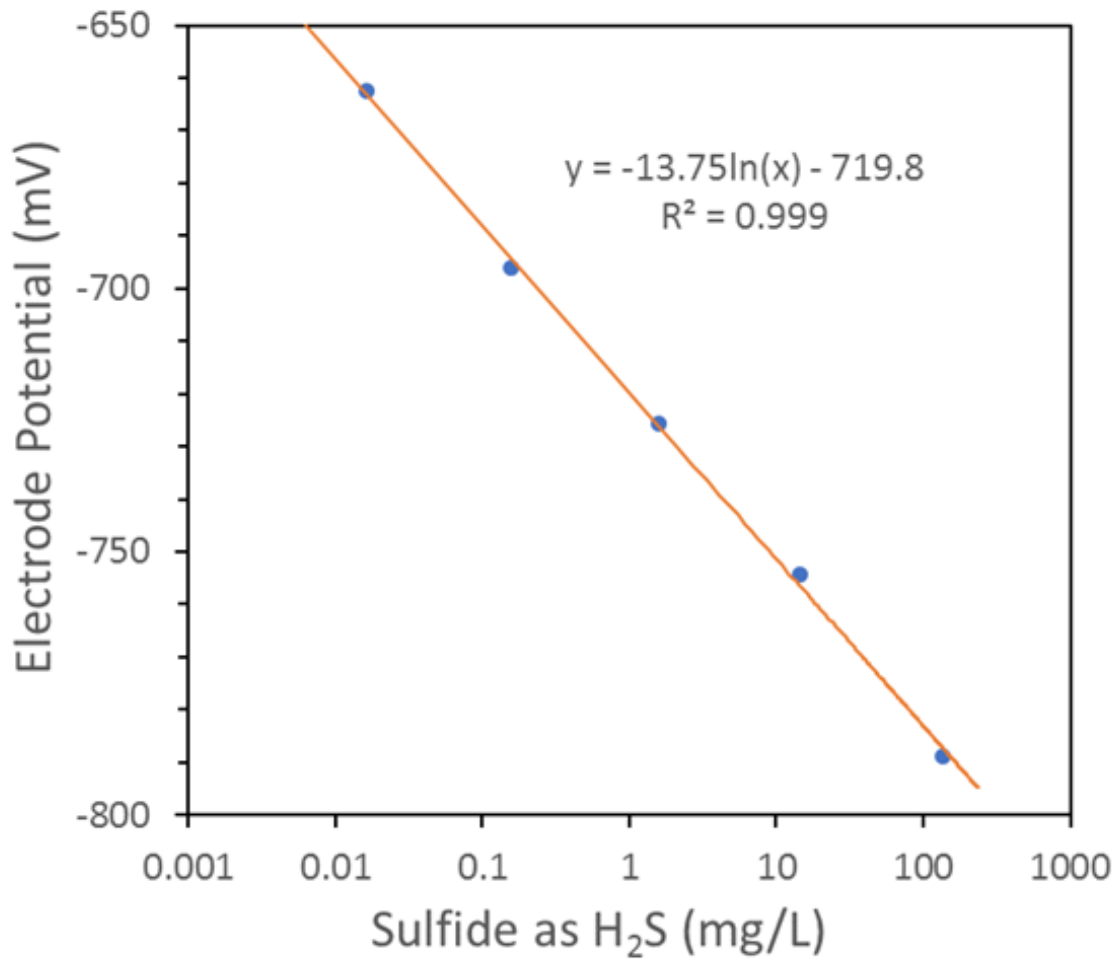
Preparation of Stock Sulfide Solutions

All sulfide solutions were prepared from a stock solution of approximately 1,000 mg/L as S (1,063 mg/L as H_2S). The stock solution was prepared by first washing a crystal of sodium sulfide nonahydrate ($\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$) with deionized (DI) water to remove oxidation products, blotting it dry on tissue paper, accurately weighing it, and dissolving it in deoxygenated DI water. Between uses, the stock solution was stored under nitrogen sealed in a Mylar bag with oxygen absorber packets to minimize exposure to oxygen. The sulfide stock solution was standardized daily by iodometric titration (Standard Method 4500-S2-F; APHA 2005).

Sulfide Measurement by Ion Selective Electrode

Dissolved sulfide concentrations were measured by Standard Method 4500-S2-G (APHA 2005) using a Thermo Scientific silver sulfide solid-state ion selective electrode (ISE) connected to a Thermo Scientific Orion Star A211 potentiometer. Alkaline anti-oxidant buffer (Thermo Scientific Orion 941609) was added to samples and standards to inhibit oxidation of sulfide by oxygen and to provide a constant ionic strength and pH during analysis. A five-point calibration curve was developed daily (Figure 2).

Figure 2
Example calibration curve for sulfide ISE



Calibration Curve for the Densitometric Determination of Sulfide

DGT sampler assemblies were exposed in duplicate to test solutions prepared at 8 different sulfide concentrations (0 to 6 mg/L as H₂S) for 24 hours. The solutions were prepared in 0.7 M sodium chloride to simulate seawater ionic strength. To minimize sulfide loss, tests were prepared in glass jars with cone seal caps, all solutions were purged with N₂ gas before addition of the sulfide stock solution, and headspace in the glass containers was minimized. Sulfide concentrations in the standard solutions were monitored by ISE before and after deploying the DGT assemblies.

Following the 24-hour exposure period, the DGT assemblies were retrieved and rinsed with DI water. The binding gel layers were removed and placed on blotting paper. The binding gels were then laid on a thin cellophane sheet (Bio-Rad), and covered with a second cellophane sheet. The sheet assembly was then placed in a vacuum gel dryer (Bio-Rad, Model 583) and dried for 2 hours at 60°C. The dried sheet was digitally scanned (Konica Minolta BizHub-C364) and saved as a gray-scale image. Gel analysis software (UN-SCAN-IT Gel Version 7.1) was used to measure and record the gray-scale intensity of each binding gel on the scanned image.

Extraction and Quantification of Sulfide in DGT Binding Gels

Sulfide was extracted from the exposed DGT binding gels by a modified CRS method (Burton et al., 2008) as well as a nitric acid/peroxide extraction method. Six replicate sulfide-exposed DGT samplers were prepared by exposing the devices to a 3.00 mg/L H₂S solution for 24 hours. Three DGTs were extracted by each method to quantitatively assess sulfide recovery and method reproducibility.

The modified CRS method developed by Burton et al. (2008) was adopted to this study. Briefly, an acidic chromium(II) solution was added to a gas-tight reaction vessel containing the sulfide-reacted binding gel to convert sulfide to hydrogen sulfide gas, which was trapped in an alkaline zinc acetate solution contained in a small centrifuge tube placed inside the reaction vessel. On completion of the extraction, dissolved sulfide concentrations in the trapping solutions were immediately measured by ISE.

A nitric acid/peroxide extraction is one of the steps employed in some selective sequential extraction procedures to quantify metals associated with organic matter and/or sulfides in soil and sediment (Tessier et al. 1979, Zimmerman and Weindorf 2010) and was used to extract sulfur from the DGT. In brief, the sulfide-reacted binding gel is extracted in acidic hydrogen peroxide followed by nitric acid. A detailed description of the procedure is provided in Appendix A. Sulfide released from the gel to the extraction fluid is oxidized to sulfate which is analyzed as total sulfur by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). ICP-OES analysis of the extracts was performed in the analytical geochemistry laboratory of the Geology Department at Portland State University. A procedural blank was also prepared and analyzed and sample results were blank corrected.

Results

Determination of Sulfide in DGT Samplers by Optical Densitometry

Dissolved sulfide concentrations in test solutions were measured before and after deployment of the DGT assemblies (Table 1). Sulfide losses from the solutions over the 24-hour exposure period ranged from 13 to 26 % and averaged 20 %. This is comparable to losses reported by Rearick et al. (2005)

due to oxidation, volatilization, or adsorption. The digital images of the sulfide-accumulated binding gels are shown in Figure 3 and the measured gray-scale intensities of the gels are given in Table 1.

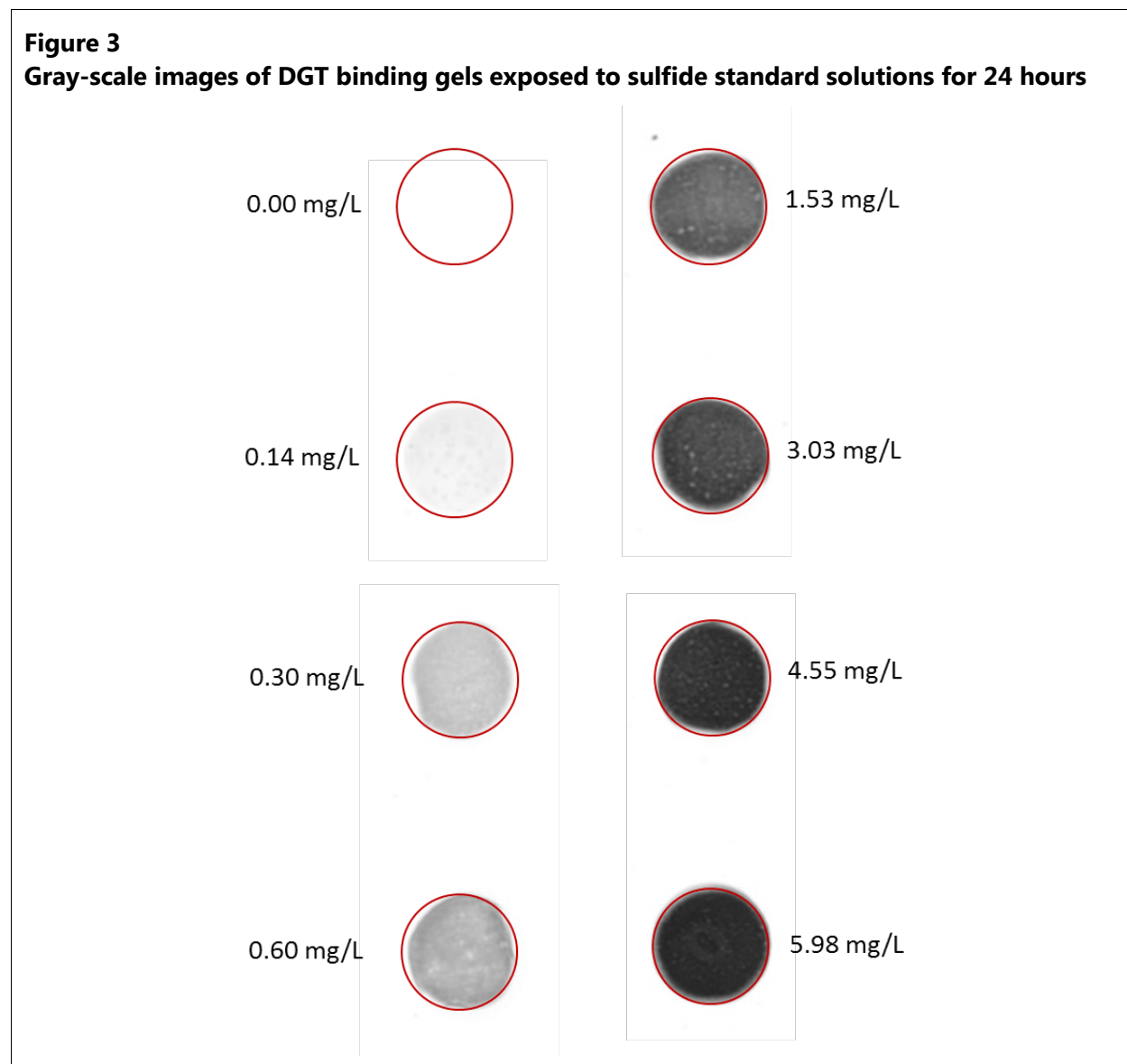


Table 1
Sulfide Mass Balance and Optical Densitometry Results

Solution	Target Concentration (mg/L as H ₂ S)	Measured Sulfide Concentration (mg/L as H ₂ S) ¹			Sulfide Loss ² (%)	Gray-Scale Intensity ³		
		Initial	Final	Average		1	2	Average
1	0.00	0.00	0.00	0.00	-	0	0	0 (0)
2	0.17	0.16	0.12	0.14	26%	6	14	10 (4)
3	0.34	0.33	0.26	0.30	23%	33	52	43 (10)
4	0.68	0.67	0.52	0.60	23%	100	77	89 (12)
5	1.70	1.70	1.35	1.53	21%	147	138	142 (5)
6	3.40	3.28	2.77	3.03	15%	171	155	163 (8)
7	5.10	4.86	4.24	4.55	13%	183	195	189 (6)
8	6.80	6.64	5.31	5.98	20%	194	193	194 (1)

Notes:

1. Measured by ISE.
2. Sulfide loss (%) during the experiments may be attributed to oxidation, volatilization, or adsorption.
3. Two DGTs were exposed at each sulfide concentration.

A calibration curve was developed by plotting the gray-scale intensity dissolved sulfide exposure concentrations and fitting the data to a function relating intensity to exposure sulfide concentration. As shown in Figure 4, the data were well fit by an exponential function (Equation 1) over the entire range of sulfide concentrations tested (0 – 6 mg/L as H₂S).

Equation 1 is useful for gray-scale intensities greater than about 10 (corresponding to sulfide concentrations greater than 0.15 mg/L). For lower concentrations, a three-point calibration curve was developed using the lowest concentration gel data. For the low range calibration, a power function was found to fit the data best (Equation 2).

Equation 1 is recommended for estimating porewater sulfide concentrations from DGT gels for gray-scale intensity greater than 10 and Equation 2 is appropriate for gray-scale intensity less than or equal to 10.

Equation 1. DGT calibration curve for sulfide concentration using optical densitometry (24-hour exposure)

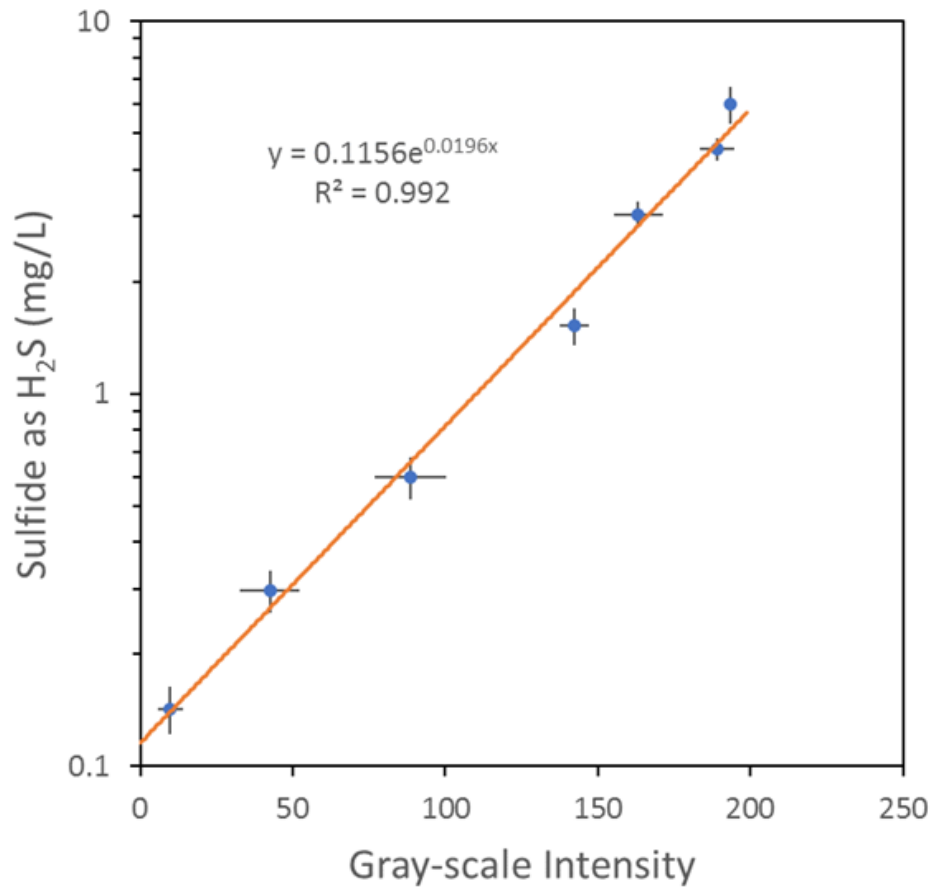
$$S = 0.1156 \times \exp\{0.0196 \times I\}$$

$$R^2 = 0.992$$

where:

- S = sulfide concentration (mg/L as H₂S)
- I = gray-scale intensity of binding gel image (10-256)

Figure 4
DGT calibration curve for a 24-hour exposure period



Equation 2. Low-range DGT calibration curve for sulfide concentration using optical densitometry (24-hour exposure)

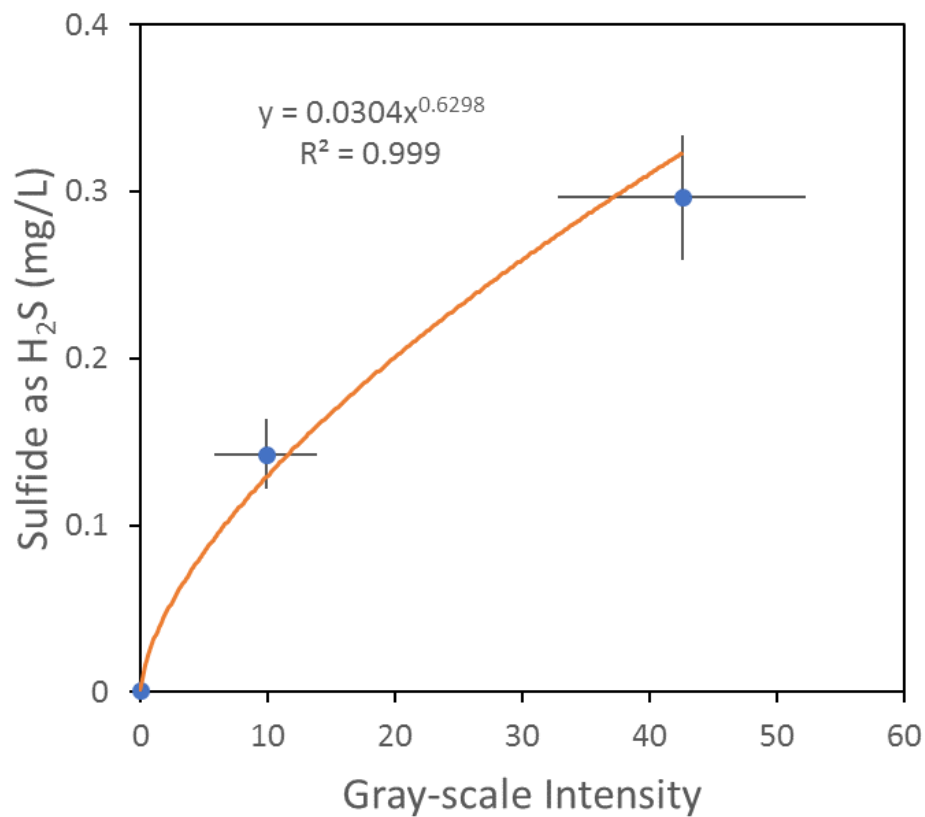
$$S = 0.0304 \times I^{0.6298}$$

$$R^2 = 0.999$$

where:

- S = sulfide concentration (mg/L as H_2S)
- I = gray-scale intensity of binding gel image (0-10)

Figure 5
Low-range DGT calibration curve for a 24-hour exposure period



Verification of Sulfide Accumulation in DGT Samplers

Sulfide recovered from DGT binding gels after exposure to 3.00 mg/L H₂S solution for 24 hours by the CRS and nitric acid/peroxide extraction methods are summarized in Table 2. The sulfide mass extracted by the two methods is in very good agreement and excellent sulfide mass recoveries were obtained.

Table 2
Verification of Sulfide Accumulation in DGT

Extraction Method	Measured Sulfide Concentration (mg/L as H ₂ S) ¹			Mass of Sulfide Extracted from DGT (µg as H ₂ S)		Theoretical Mass of Sulfide Accumulated in DGT (µg as H ₂ S) ²	Recovery (%) ³
	Initial	Final	Average				
Chromium Reducible Sulfur	3.65 ±0.11	2.34 ±0.04	2.99	1	13.5	13.9	97
				2	13.6		98
				3	15.2		109
				Average	14.1		101
Nitric acid /Peroxide	3.65 ±0.11	2.36 ±0.01	3.00	1	12.9	13.9	92
				2	14.5		104
				3	13.4		96
				Average	13.6		98

Notes:

1. Measured by iodometric titration.
2. Calculated with Equation 2 using the average sulfide concentration, $D=1.65 \times 10^{-6} \text{ cm}^2/\text{s}$ (derived from data in Teasdale et al. 1999), $t=86,400 \text{ s}$, $A=2.54 \text{ cm}^2$, and $\Delta g=0.078 \text{ cm}$.
3. $\text{Recovery}=100 \times (\text{mass of sulfide extracted})/(\text{theoretical mass of sulfide accumulated})$

The extracted sulfide masses were compared to the theoretical amount accumulated calculated using the mass-transfer equation for sulfide accumulation in the DGT assembly (Equation 3) and used to calculate percent recoveries (Table 2).

Equation 3. Model equation for calculating sulfide mass accumulation in DGT binding gel (Teasdale et al. 1999)

$$M = \frac{CDtA}{\Delta g}$$

where:

- M = mass of sulfide accumulated in binding gel (mg as H₂S)
- C = sulfide concentration in solution (mg/cm³ as H₂S)
- D = effective diffusion coefficient of sulfide in DGT (cm²/s)
- t = exposure time (s)
- A = cross-sectional area of DGT window (cm²)
- Δg = thickness of diffusion layer (cm)

The sulfide mass recoveries were in excellent agreement with the theoretical sulfide mass accumulated calculated by equation 3 and averaged 101 % of theoretical for the CRS method and 98 % for the nitric acid/peroxide extraction (Table 2).

Summary

The present study verified the DGT method for passive sampling of dissolved sulfide by establishing a quantitative relationship between dissolved sulfide concentrations in the range of 0 to 6 mg/L and the gray-scale intensity developed by the DGT binding gel after a 24-hour exposure period, and confirmed that the sulfide accumulated in the DGT gel agreed with the amount expected from theory. The results of this study show that the DGT method with quantification by optical densitometry gives accurate and reproducible results for dissolved sulfide concentrations and calibration curves were developed for field application of the DGT devices.

References

- American Public Health Association (APHA), 2005. Standard Methods for the Examination of Water and Wastewater, 21st ed. <http://standardmethods.org>
- Burton, E.D., Sullivan, L.A., Bush, R.T., Johnston, S.G., and A.F. Keene, 2008. A simple and inexpensive chromium-reducible sulfur method for acid-sulfate soils. *Applied Geochemistry* 23:2759-2766.
- Rearick, M.S., Gilmour, C.C., Heyes, A., R.P. Mason, 2005. Measuring sulfide accumulation in diffusive gradients in thin films by means of purge and trap followed by ion-selective electrode. *Environmental Chemistry & Toxicology* 24:3043-3047.

- Teasdale, P.R., Hayward, S., and W. Davison, 1999. In situ, high-resolution measurement of dissolved sulfide using diffusive gradients in thin films with computer-imaging densitometry. *Analytical Chemistry* 71:2186-2191.
- Tessier, A, PGC Campbell and M. Bisson, 1979. Sequential extraction procedure for the speciation of particulate trace metals. *Analytical Chemistry* 51:844-851.
- Zimmerman, A.J., and D.C. Weindorf, 2010. Heavy metal and trace metal analysis in soil by sequential extraction: a review of procedures. *International Journal of Analytical Chemistry* (dx.doi.org/10.1155/2010/387803).

Appendix A

Nitric acid/peroxide extraction procedure

Fluid 1: 0.02 M nitric acid

Fluid 2: 30% hydrogen peroxide adjusted to pH 2 with nitric acid

Fluid 3: 3.2 M ammonium acetate in 20% v/v nitric acid

1. Place a sulfide exposed DGT binding gel in a 50 mL centrifuge tube
2. Add 3 mL of fluid 1 and 5 mL of fluid 2 and heat in shaker-water bath at 85 ± 2 °C for 2 hours
3. Add 3 mL of fluid 2 and heat for an additional 3 hours
4. After cooling, add 5 mL of fluid 3, dilute to 20 mL and agitate for 30 minutes at room temperature
5. Centrifuge at 10,000 rpm for 25 minutes (if solid is present)
6. Decant fluid
7. Repeat steps 3-6 with DI water (if solid is present)
8. Combine all decanted fluid, acidify with nitric acid to pH <2
9. Dilute with DI water to a final volume of 50 mL and submit for analysis

Attachment 2
Vane Shear Log Form
