EPA Comments and Responses on Draft (dated August 28, 2020), Revised Draft (dated December 10, 2020), Draft Final (dated May 3, 2021, and Final (dated June 10, 2021) Pre-Design Investigation Work Plan
Willbridge Cove Project Area

This is the U.S. Environmental Protection Agency's (EPA's) conditional approval of the Final Pre-Design Investigation Work Plan (PDI WP) for the Willbridge Cove Project Area (WCPA). The PDI WP was prepared by Jacobs on behalf of Willbridge Cove Group and dated June 10, 2021. EPA finds the revisions to the Final PDI WP to be generally responsive to EPA's comments to date and conditionally approves the document. The Willbridge Cove Group should consider the text below as it implements the PDI WP and in future phases of the project.

The conditional approval excludes PDI WP Appendix D Health and Safety Plan (HASP). EPA does not approve HASPs but reviews for completeness.

EPA Comments on the PDI WP

Unless otherwise noted, Willbridge Cove Group's responses to EPA's comments on the Draft PDI WP, the Revised Draft PDI WP, the Draft Final PDI WP, and the Final PDI WP are acceptable. EPA submits the following clarifications, supplemental information, and/or correction requests for General Comments 1 and 3; Dive Operations Plan Comments, and November 20, 2020 email comments.

EPA General Comment 1 (October 26, 2020)

Contaminant Delineation: The proposed sampling plan is insufficient to refine sediment management areas (SMAs) and the riverbanks pending characterization according to EPA's *Remedial Design Guidelines and Considerations* (RDGC). EPA requests the following modifications.

a. Additional sampling locations should be positioned in and around the preliminary SMAs to horizontally delineate contamination. Per Section 5.1.2 of EPA's RDGC, contaminated sediment shall be delineated on a nominal 150- by 150-foot (ft) grid with step-out samples in a 150-ft buffer zone to confirm the extent of contamination (EPA 2020). The PDI WP argues that there are cases of older data which are no longer representative of site conditions for various reasons. Quantitative rationale must be provided to substantiate that claim after SMA refinement sampling conducted as part of the PDI.

c. Include at least two additional samples on the north portion of the riverbank beyond S01 and S02 to horizontally delineate contamination on the bank adjacent to preliminary SMA 4 as well as at least two additional riverbank samples near SB-N6 (DDT removal area). Additional step-out samples should be located at both ends of the ordinary high water and mean low water transects as well as riverward of the samples near SB-N6 to confirm the extent of contamination. Furthermore, the Willbridge Cove Group should use a sampling technology, such as limited access direct push or angled borings, which can achieve greater

than 5-ft depth. If DOC is not fully delineated within the 5-ft depth, EPA will require additional sampling to delineate DOC in the riverbank.

d. The quantity and quality of sediment beneath dock structures within SMA footprints is not discussed. Because these areas have not been dredged, as evidenced by the residual "sand bars" in cross-section D-D', they could conceivably reflect higher historical sediment concentration. The sediment quality beneath the docks also is a factor in the recontamination assessment. The lack of sediment quality data beneath the docks is a data gap that should be considered in the PDIWP.

Willbridge Cove Group's Response (December 10, 2020)

a. Sampling designs have been revised to collect additional data needed to confirm and delineate SMAs. The location and types of samples are based on preliminary SMA characteristics (e.g., bathymetry and authorized dredge depths), anticipated remedial technology assignments based on the ROD Technology Decision Tree (e.g., dredging or capping), and the location, depths, and chemical results for existing FS and Pre-RD samples within and adjacent to the preliminary SMAs. Where appropriate, the sampling designs use the recommended 150-ft grid spacing. Sections 2.2 and 2.3 of the revised PDI Work Plan describes the locations, intervals, and analytical data for historical samples that were dredged in 2011 and are no longer representative of site conditions. This information was considered in development of the PDI sampling designs and will be reiterated as applicable in the PDI Evaluation Report.

c. The river bank sampling design has been updated, but does not include the additional samples requested in the comment. The objective of river bank sampling is to determine whether the adjacent preliminary SMA is contiguous with river bank soil contamination. The DDT removal area in the vicinity of SB-N6 is not adjacent to Preliminary SMA 4 and elevated PCBs (the focused COC with RAL exceedances at Preliminary SMA 4) are not the driver for the DDT removal action under the KMLT dock. The under-dock removal action will include collection of post-removal confirmation samples. These post-removal results will be considered in the remedial design for the river bank if one is necessary.

   With regard to sample depths along the river bank adjacent to Preliminary SMA 4, sampling deeper than 5 feet to determine depth of contamination is not necessary for remedial design purposes in this area based on the expected technology assignment for the area and depth of feasible dredging or excavation.

d. The sampling designs for preliminary SMAs that extend beneath dock structures have been modified to include surface sediment sample locations beneath the docks.

EPA Response (January 21, 2021)

a. The Revised Draft PDI WP does include additional samples, but it still fails to delineate SMAs based on the 150- by 150-foot grid specified in the RDGC. Samples at the following locations should be added to the PDI WP.

- **SMA 1**: To achieve the required spatial resolution for SMA delineation, add one sample each in the downstream and upstream 150- by 150-foot grid squares (downstream of PD01 and upstream of PD07).

- **SMA 2**: To achieve the required spatial resolution for SMA delineation, add one sample in the grid square north of PD24 along the boundary of the preliminary SMA and adjacent to the upstream side of the Kinder Morgan dock. Note that if PDI step-out samples PD08, PD09, and PD10 exceed RALs and/or PTW thresholds, the lateral and vertical extent of navigation channel RAL and/or PTW threshold exceedances will need to be delineated to no more than half the distance across the navigation channel, as per the Willbridge Cove Group's Statement of Work Section 5.6(c).

- **SMA 4**: Locations PD40, PD44, and PD50 should be primary sampling locations to achieve the required spatial resolution for SMA delineation. Location PD43 should be a primary sample because of nearby subsurface PCB exceedances.

c. EPA notes that the requested additional riverbank samples adjacent to preliminary SMA 4 and riverward of SB-N6 were not included in the PDI WP. Additionally, the requested riverbank soil sampling depth increase was not incorporated into the PDI WP. If the lateral extent of riverbank soil contamination exceeding RALs and/or PTW thresholds is not delineated adjacent to and potentially extending beyond preliminary SMA 4, or if adequate subsurface soil data are not generated for design of a riverbank remedial action in the WCPA, EPA will consider this a data gap requiring additional data collection in a supplemental PDI.

d. The planned approach to include surface sediment sample locations beneath the docks is acceptable; however, if RAL or PTW threshold exceedances are present in surface sediment, additional sampling to delineate subsurface sediment exceedances will be required. Diver-assisted sediment cores up to 4 feet in length have been collected under structures in other RD project areas at Portland Harbor.

Willbridge Cove Group's Response (March 26, 2020)

a. Request for additional delineation cores and grabs:

   i. SMA 1: Additional cores were not added to the upstream and downstream ends of this preliminary SMA because there are already step-out sample/core locations upstream and downstream of the primary sample/core location at preliminary SMA 1. A step-out core and grab sample has been added between the shoreline and primary locations PD02, PD03, and PD04 based on comment Specific Comment 13.

   ii. SMA 2: Additional step-out samples were not added around SMA 2. This area has been routinely dredged and the additional suggested samples are unlikely to substantively change the interpretation or application of the ROD decision tree. The Willbridge Cove Group acknowledges that additional sampling may be needed in the navigation channel pending the results from the row of step-out samples on the eastern edge of the project boundary.

iii. SMA 4: The suggested sampling locations were changed to primary sampling locations and two additional step-out sampling locations have been added in this area.

c. River bank sampling

i. Two additional riverbank sampling locations have been added on the northern end of this area. The need for additional samples to characterize the river bank adjacent to SMA 4 will be determined after the PDI data are evaluated.

d. Under dock sample cores

i. Comment is acknowledged, the need for additional samples to characterize the under-dock areas will be determined after the PDI data are evaluated.

EPA Response, via email (May 19, 2021)

EPA notes that not all requested revisions to the planned sampling locations were made. EPA expects that data gaps will be reevaluated in a future RD deliverable after PDI data is collected and SMA refinement is finalized.

EPA General Comment 3 (October 26, 2020)

There are numerous RAL and/or PTW threshold exceedances in subsurface sediments outside of the SMA footprint for total PCBs; DDx; 2,3,7,8-TCDD; 1,2,3,7,8-PeCDD; and 1,2,3,4,7,8-HxCDF. These locations are shown on PDI WP Figures 2-12 through 2-17 for the focused COCs and tabulated for all ROD Table 21 contaminants in PDI WP Tables A-1 and A-2. Refinement of SMAs for RD needs to consider RAL and PTW threshold exceedances in subsurface sediment as well as at the surface as stated in RD principle #1 in EPA's RDGC. Whether or not areas with subsurface RAL exceedances will be included in an SMA is dependent on the chemical and physical stability of the buried material and reasonably anticipated future site uses (see RD principle #2 in EPA's RDGC). Additional subsurface sediment sampling should be performed to delineate these subsurface RAL and/or PTW threshold exceedances and EPA expects that a buried contamination evaluation will be performed as part of a future RD deliverable.

Willbridge Cove Group's Response (December 10, 2020)

The PDI Work Plan has been revised to include a physical/chemistry stability DQO (DQO 2) and describes the data requirements and evaluation process in Section 1.4.2 and the results of the related data gaps evaluations in Section 2.4. The data gap evaluations conclude that the existing data and planned PDI sampling data will be sufficient to characterize the chemical and physical stability of at-depth contamination in areas within and outside of the preliminary SMAs. No additional sampling specific to stability evaluations is needed.

EPA Response (January 21, 2021)

EPA does not agree that the planned PDI sampling data will provide adequate spatial coverage of the WCPA (see General Comment 1) or that no additional data is needed to evaluate the physical and chemical stability of buried contamination in the WCPA.

EPA has the following comment on DQO 1 – SMA Refinement, and the PDI WP should be revised accordingly:

a. The lateral extent of an SMA is dependent on both surface and subsurface contamination, and the extent of RAL and/or PTW threshold exceedances must be characterized to delineate SMAs. Additionally, the buried contamination evaluation must demonstrate physical and chemical stability of subsurface RAL exceedances before they are considered to be excluded from an SMA. Subsurface sediment exceeding PTW thresholds must be actively remediated in accordance with the ROD. Step 5 of Table 1-2 should be revised accordingly.

EPA has the following comments on DQO 2 – Physical and Chemical Sediment Stability and the PDI WP should be revised accordingly:

a. EPA disagrees with Willbridge Cove Group that no additional sampling specific to the stability evaluations is needed. There are multiple subsurface RAL and/or PTW threshold exceedances outside of the preliminary SMAs near which additional PDI samples should be collected. If Willbridge Cove Group elects to not collect additional samples outside of the preliminary SMAs, there may be insufficient data for SMA refinement (DQO 1) and/or the physical and chemical stability evaluations (DQO 2) that would require additional data collection as part of a supplemental PDI.

b. Complete chemical stability modeling requires additional data which are not currently available or planned to be collected. It is acceptable to use porewater upwelling information from Appendix C of the RI; however, EPA recommends collecting current site-specific porewater contaminant concentrations for inputs to chemical fate and transport modeling. If the Willbridge Cove Group chooses to use literature based partition coefficients to determine porewater concentrations for modeling, EPA should be consulted in the selection of appropriately conservative partition coefficients and resulting porewater concentrations should be compared to the site-specific porewater concentrations provided in Appendix C of the RI.

c. Depth profiles of Cesium-137 and ROD Table 21 contaminants should be considered as an additional line of evidence.

d. Hydrodynamic modeling and shear stress evaluations should use the 100-year flood event and include an analysis of the effects of climate change and seismic impacts, consistent with ROD requirements for caps.

e. EPA expects a buried contamination long-term monitoring plan to be developed in future RD deliverables.

f. Temporal trends in surface sediment contaminant concentrations, potential in-water construction, and reasonably anticipated future use should all be considered as additional lines of evidence to inform the stability of buried deposits.

Willbridge Cove Group's Response (March 26, 2020)
DQO 1 Comment Response "a":

The PDI Work Plan includes samples intended to characterize and delineate the lateral and vertical extent of RAL/PTW threshold exceedances in SMAs such that remedies appropriate to future use or location can be designed.

The Willbridge Cove Group is developing a weight of evidence approach to evaluate the physical and chemical stability of buried contamination in the WCPA using existing data and data to be gathered during the PDI. The approach and associated lines of evidence for the stability evaluations will be developed prior to the presentation of the PDI results.

The last sentence of the comment response regarding the need to actively remediate PTW threshold exceedances in subsurface sediment is inconsistent with the description of the selected remedy in Sections 11 and 14.2 of the ROD and EPA's guidance documents as summarized below:

Section 6.5.1 of the ROD states "The PTW evaluation only included surface sediment, which poses the greatest risk of exposure given Site-specific conditions." Figure 8 of the ROD identifies PTW in surface sediments that are characterized as Source Material/NAPL, Not Reliably Contained and Highly Toxic. Section 11 of the ROD then goes on to state that "If NAPL or PTW that is not reliably contained is identified in any of the remediation areas under the Selected Remedy, it will be addressed as specified in Section 14.2." This sentence does not address Highly Toxic PTW nor areas with no surface RAL or PTW threshold exceedances.

We agree that the ROD states that surface sediment exceeding PTW thresholds must be remediated, but the ROD does not address subsurface sediments in areas without a surface sediment exceedance of RALs or PTW thresholds. The Statutory Determination (page iv) states that where there are surface sediment

exceedances of RALs or PTW thresholds, "[t]he Selected Remedy will address all principal threat waste (PTW) by excavation and off-site disposal, or, if left in place, with augmented reactive caps to provide in-situ treatment." Sections 14.2.2 through 14.2.5 of the ROD state that "NAPL or PTW that cannot be

reliably contained will be dredged [or excavated]…". Subsurface PTW in areas with no surface RAL or PTW threshold exceedances is not addressed. Hence, ROD statements apply to PTW in surface sediments and subsurface PTW in areas with surface RAL exceedances.

Further, the decision tree (ROD Figure 28) and Section 14.2 of the ROD differentiate between different types of PTW. It treats PTW characterized as Highly Toxic (based on PTW threshold exceedances) in surface sediments by following the same approach as surface sediments with RAL exceedances, i.e., "N" path for Highly Toxic PTW, while NAPL and PTW not reliably contained are treated differently by following the "Y" path. By applying the same rationale, subsurface sediments where PTW is characterized as Highly Toxic based on PTW threshold exceedances, but not as NAPL or not reliably contained must be treated following the same approach as subsurface sediments with RAL exceedances.

This approach is consistent with the decision presented in Appendix B – Remedial Design Topics and Discussion (December 23, 2019), Topic 4 – Defining Depth of Contamination during SMA Delineation. This appendix is part of the Remedial Design Guidelines and Considerations – Portland Harbor

Superfund Site (dated February 28, 2020). The decision presented at the conclusion of the Topic 4 discussion states: "For sites where NAPL does not exist and/or the sampling location is not within a dredging area, sediments with concentrations exceeding RALs or PTW thresholds at depth may be left

in place if it can be demonstrated that the subsurface sediments will remain stable. In such cases, the physical (i.e., erosive conditions, slope stability) and chemical (i.e., advective and diffusive flux due to seepage) stability of the buried contamination will need to be demonstrated by conducting appropriate

evaluations to assess whether the material needs to be capped or to support cap design" (emphasis added). Thus, where there are no surface RAL or PTW threshold exceedances and no NAPL, subsurface exceedances of PTW thresholds will be treated identically to subsurface RAL exceedances and the physical and chemical stability of the buried contamination will be evaluated to determine whether remedial action is required.

Thus, step 5 of Table 1-2 has been revised to reflect the above discussion.

DQO 2:

a/b. See response to DQO 1 Comment Response "a" regarding SMA delineation and weight of evidence approach to be used for stability evaluations. Both empirical data and modeled results will be considered as lines of evidence in the evaluations. This will be developed prior to presentation of the PDI results.

c. Depth profiles of the Table 21 contaminants will be used as a line of evidence in evaluating the physical and chemical stability. Radiometric dating is not proposed because given development of the cove and the associated maintenance dredging history these types of data are not expected to produce adequate resolution or be useful for remedial design.

d. Comment acknowledged. Hydrodynamic modeling, where needed, will be performed in accordance with ROD requirements and the RD guidance, as applicable (and will be incorporated as part of physical stability protocol as appropriate).

e. Comment acknowledged. A project area monitoring plan is a required element of the SOW and will be prepared as part of the remedial design.

f. Comment acknowledged. These lines of evidence will be incorporated into the evaluation of chemical and physical stability, as appropriate.

EPA Response via email (May 19, 2021)

Areas where subsurface sediment has exceedances of ROD Table 21 RALs and/or PTW thresholds that are covered by at least 30 centimeters of sediment without Table 21 RAL or PTW threshold exceedances may not require active remediation if it can be demonstrated that the subsurface contamination is physically and chemically stable (see RD Principle #2).

Willbridge Cove Group's response to EPA General Comment #3 contains inaccurate statements and misrepresentations of the ROD. For example:

i. The Willbridge Cove Group response states that: "The Statutory Determination (page iv) states that ***where there are surface sediment exceedances of RALs or PTW thresholds***, '[t]he Selected Remedy will address all principal threat waste (PTW) by excavation and off-site disposal, or, if left in place, with augmented reactive caps to provide in-situ treatment.'" (***emphasis added).*** The ROD Statutory Determinations section does not state or imply that PTW needs to be addressed only in areas with surface sediment exceedances of RALs or PTW thresholds. There is no mention of surface sediment in the ROD Statutory Determinations section.

ii. EPA disagrees with the statement that, "ROD statements apply to PTW in surface sediments and subsurface PTW in areas with surface RAL exceedances." The selected remedy includes active remediation of Table 21 exceedances in surface and subsurface sediment per ROD Figure 28 Footnote #1 which states that: "Contamination is defined in three dimensions."

EPA Comments on the Appendix D HASP Dive Operations Plan

Because the Dive Operations Plan was submitted two weeks prior to dive operations, many of EPA's comments on the plan were addressed during the pre-dive safety briefing. Dive operations have been completed, so a revision to this Dive Operations Plan is unnecessary at this time. However, EPA comments, dated June 24, 2021, on the Dive Operations Plan are included here for the Willbridge Cove Group's information. If supplemental diving is necessary, the following deficiencies should be corrected in future dive plans prior to commencing dive operations.

Note that EPA does not approve health and safety plans or dive safety plans but reviews for completeness.

General Comments on Dive Plan:

1. **Section Headings.** Section headings should be sequential.

2. **Personnel.** When using commercial divers rather than divers trained as underwater scientists, include scientific sampling procedures in the Dive Plan and review at daily safety briefings to ensure nonscientific divers collect samples that meet the pre-design investigation work plan's data quality objectives.

3. **Signatures.** Before initiating diving, include and provide a signature page to EPA oversight staff that all divers have read and understand the Dive Plan. (EPA 2016; Appendix H)

Specific Comments on Dive Plan:

1. **Section 1.1 Dive Team Assignments and Responsibilities, page 3:** Minimum crew size for surface supplied diving operations for EPA is four, which may include the boat captain while the vessel is at anchor.

2. **Section 1.3 Safety Policy, page 5:** Ensure that work will comply with the latest Association of Diving Contractors International, Inc. (ADCI) standards for commercial diving as well as EPA's dive safety manual: https://www.epa.gov/diving/epas-diving-safety-program.

3. **Section 1.1 Ambient Conditions, page 6:** EPA has the following comments on this section:

a. A description of the river stage fluctuation (tidally influenced) and weather conditions should be added to this section as per OSHA 29 CFR 1910 Subpart T and EPA (2016) Appendix J.

b. Water visibility is listed at 10 to 25 feet, which is likely overestimated. Revise and plan for 1 to 5 feet of underwater visibility.

c. Water temperature is listed as 45 to 50 degrees Fahrenheit (F) which is underestimated. Revise to actual river conditions (60 to 70 degrees F) and plan for any thermal stress considerations.

4. **Section 1.3. Equipment, page 9.** EPA has the following comments on this section:

a. Oxygen kit must be capable of ventilation of two nonbreathing divers simultaneously to comply with the EPA standard, e.g., with ambu bags or manual trigger ventilation valves.

b. A backboard must be onsite.

c. The plan should specify that alpha and recreational dive flags will be flown while divers are in the water.

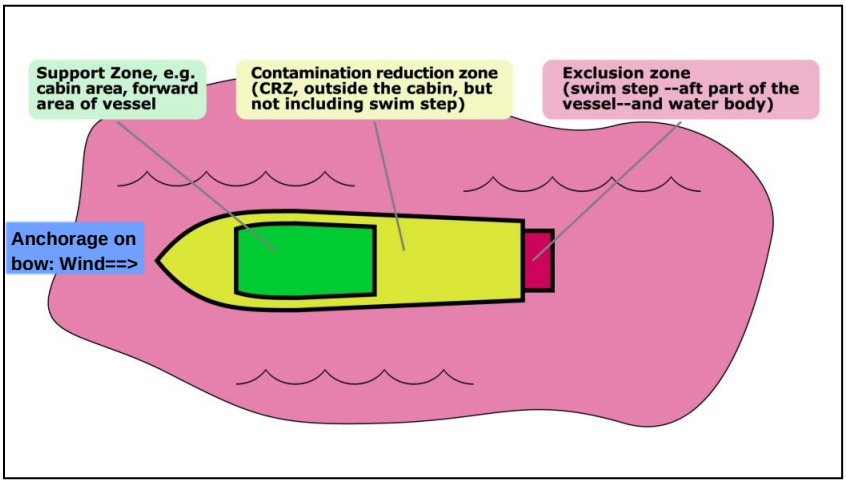
d. Tender personal protective equipment (PPE) should be specified.

e. Diver harness should be noted to meet the latest ADCI requirements.

f. The plan should specify that the divers will be using drygloves and at a minimum a latex neck dam if not mating the helmet to the suit directly. An activity hazard analysis (AHA) should include what will be done in the event of a suit leak.

g. Personal flotation devices should be noted as required when outside the cabin and not fully zipped into a drysuit. Hydrostatic inflation vests should be checked daily for operational status as per OSHA 29 CFR 1926.106.

h. While hazard zone figures are included, the wind direction and anchorage are not always included. Revised figures with wind direction and anchorage noted should be included in the Dive Plan. Wind direction and anchorage (which affects wind direction across the vessel) must be specified and configuration adjusted if the wind changes to avoid splashes outside of the exclusion zone. Specify how each zone is delineated, e.g., tape labels, physical barriers, etc. to prevent cross-contamination (EPA 2016, Appendix Q). The figure below is an example of zone delineation with additional depiction of wind direction and anchorage location.



i. Breathing gas used must meet or exceed the Compressed Gas Association Grade E standard (EPA 2016, Appendices A and J) and be tested within the last 6 months.

5. **Section 1.4 Lock Out / Tag Out Procedures, page 9:** This section should include procedures to ensure lock out / tag out for any intakes or discharges for near-vessel diving, as the dive area is near or potentially near berthed or berthing vessels. The Dive Plan should include a ship safe checklist as appropriate.

6. **Section 1.5 Equipment and Training Certifications, pages 9 through 10.** EPA has the following comments on this section:

    a. Divers should be 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) certified, with latest annual 8-hour refreshers as applicable. Certification for the 40-hour HAZWOPER should be provided for all assigned divers in Attachment 1.

    b. Divers that spend 30 days or more per year on HAZWOPER sites must be in a medical surveillance program.

    c. Some personnel have automated external defibrillator (AED) and oxygen administration certifications provided while others do not. Please provide AED and oxygen administration certifications for all assigned divers (EPA 2016, Appendices A and J).

7. **Section 2.0 Emergency Management Plan, pages 10 through 15.** EPA has the following comments on this section:

    a. Ensure an AED is onboard, and personnel are trained in first aid, CPR and the use of an AED.

    b. Emergency egress procedure must be specified in terms of how exactly an unconscious diver will be removed from the water, e.g., via davit, winch, cutting equipment off, etc., and this should be included in the AHA (EPA 2016, Appendix J).

    c. The DAN emergency number listed is incorrect. The correct number is (919) 684-9111.

d. The nearest hyperbaric chamber type (single vs. multi-lock) and capabilities (e.g., can reach a depth capable of Table 4, Table 6 treatment) should be specified in this section along with the address and phone number and hours of operation as per the ADCI standards and EPA (2016, Appendices A and J).

8. **Section 3.0 Job Safety Analysis, pages 17 through 22:** Beyond those hazards already identified in the AHA, the following should be added to address contaminated water diving procedures including at a minimum:

    a. Glove leak

    b. Suit leak

    c. Mask leak

    d. Heat stress (e.g., use of decontamination potable water to spray down the diver). Heat stress can be an issue on a sunny day even in the fall when using drysuits.

    e. Channel operations and Vessel traffic. The configuration of the anchored vessel and hose will avoid "cross channel" configurations especially where line of sight is limited (e.g., samples required at or near a river bend), which could hazard the diver's umbilical if a small craft approaches at high speed not seeing the dive flags. If line of sight is limited, language should be added to the AHA on how this will be dealt with, e.g., posting dive flags up and downstream on shore where approaching small craft can see them from a distance.

9. **Attachment 2 Decontaminated Diving and Decontamination Procedure:** While this generic standard operating procedure might be used in a variety of contaminated water settings, the Dive Plan must include a stepwise process for diver decontamination and containment/disposal of any solutions used for decontamination if not potable water applicable to the site in question. The Dive Plan must discuss the water column (e.g., bacteria from stormwater runoff) and sediment contamination (e.g., PCBs, metals, phthalates, pesticides, dioxins) likely to be encountered relative to the dive tasks to be accomplished as per OSHA 1910.120. The decontamination procedure needs to be detailed including when ambient water versus potable water may be used, various decontamination solutions, and PPE for tender(s). Potable water decontamination shall be used at a minimum after each dive for dives with low to moderate contamination. A dive ladder or other means of egress must be employed to allow the diver to exit the water with PPE intact for decontamination on the dive platform. Use appropriate portions of the ADCI and EPA procedures (EPA 2016, Appendix Q).

References

EPA. 2016. *Diving Safety Manual (Revision 1.3)*. Washington, D.C.: EPA Office of Administration and Resources Management, Safety and Sustainability Division.

EPA Comments on November 20, 2020 Comments to Willbridge Cove Group

After the November 18, 2020 teleconference meeting with the Willbridge Cove Group, EPA sent two comments via email to the Willbridge Cove Group on November 20, 2020 pertaining to buried contamination and data replacement. EPA submits the following clarifications and supplemental information for the Willbridge Cove Group's response to EPA's comments provided on November 20, 2020.

EPA Comment (November 20, 2020)

Buried Contamination: As stated in the Remedial Design Guidelines, whether or not buried contamination areas will be included in an SMA is dependent on the chemical and physical stability of the buried material and reasonably anticipated future uses. Stability determinations will be based on, but not limited to the following factors: erosion/scour potential; chemical concentration compared to RAL thresholds; depth of contamination; and advective transport. The Guidelines also state that fate and transport modeling will be required to support these evaluations. EPA is concerned that there is currently not enough information to make a buried contamination stability conclusion for the RAL exceedances proposed in our call. Because the primary data quality objective of the PDI is SMA delineation, it is premature to exclude known RAL or PTW exceedances from PDI sampling based on an anticipated buried contamination conclusion that is expected to be evaluated and determined as part of the BODR. Therefore, we recommend the inclusion of additional sample points near the known subsurface RAL and PTW exceedances.

Willbridge Cove Group's Response (December 10, 2020)

The PDI Work Plan has been revised to include a physical/chemistry stability DQO (DQO 2) and describes the data requirements and evaluation process in Section 1.4.2 and the results of the related data gaps evaluations in Section 2.4. The data gaps evaluations conclude that the existing and planned PDI sampling data will be sufficient to characterize the chemical and physical stability of at-depth contamination in areas within and outside of the preliminary SMAs. No additional sampling specific to stability evaluations is needed. The combination of exiting data and data proposed to address DQO 1 will be sufficient to address DQO 2.

EPA Response (January 21, 2021)

EPA's conditional approval of the framework for evaluating physical and chemical stability as presented in DQO 2 in the Revised Draft PDI WP and modified by the above comments should not be considered an approval of the results of the evaluation in future RD deliverables.

Willbridge Cove Group's Response (March 26, 2020)

Comment acknowledged.

An addendum to the revised draft PDI Work Plan will be prepared and submitted to document changes to the PDI sampling and analysis approach noted in previous responses. This addendum will include revised figures and the revised supporting tables with sampling, archiving, and analytical schedules.

The framework for the physical and chemical stability evaluation will be more fully developed prior to the presentation of the PDI results.

EPA Comment (November 20, 2020)

Data Replacement. We understand that the Willbridge Cove Group intends to use their forthcoming PDI data as well as the Pre-RD data as part of their surface sediment data replacement strategy. This is an acceptable approach but will require additional PDI surface sediment data than what was presented to EPA yesterday (11/18/2020) to achieve an adequate sample density to facilitate data replacement. The 150- by 150-foot sediment sampling grid is applicable for SMA delineation and is intended to augment RI/FS data to proceed towards the BODR with anticipation of higher resolution data to be collected for remedial design and data replacement purposes. As mentioned in the EPA comments, RI/FS data should only be replaced when concentrations have changed substantively over time and when spatial resolution is at least maintained or improved. EPA recommends the collection of additional samples to provide greater spatial density for the Willbridge Cove Group's future data replacement strategy.

Willbridge Cove Group's Response (December 10, 2020)

Section 1.4.1 added to describe the data replacement review process and includes references to EPA RD and data replacement guidance. Sampling designs in Section 3 have been revised to include additional grab surface sediment samples to provide greater spatial density in the three locations where data replacement is under consideration. Table 3-2 (now 3-1) was updated to reflect the revised sampling approach and identify which existing data may be replaced by newer data.

EPA Response (January 21, 2021)

EPA's conditional approval of the framework for evaluating data replacement as presented in PDI WP Section 1.4.1 should not be considered an approval of the results of the evaluation in future RD deliverables.

Willbridge Cove Group's Response (March 26, 2020)

Comment acknowledged.

From: Ron McLeod <Ron.McLeod@ALSGlobal.com>
Sent: Friday, May 14, 2021, 11:04 AM
To: Howard Boorse
Cc: Gauthier, Marilyn/PDX; Kidd, Bernice/RDD
Subject: RE: [EXTERNAL] - SOP Release for Willbridge Cove

ALS Canada Ltd dba ALS Environmental and ALS Life Sciences authorizes the transfer of these SOPs/documents to the US EPA and the inclusion of these documents into the QAPP along with the associated QAPP distribution.

Regards

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Portland Harbor Superfund Site
Willbridge Cove Project Area Remedial Design

Pre-Design Investigation Work Plan

Final

June 10, 2021

Prepared for the Willbridge Cove Group

Prepared by Jacobs (Willbridge Cove RD Supervising Contractor)
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For Submittal to EPA Region 10

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Acronyms and Abbreviations

Acronym	Definition
µg/kg	microgram(s) per kilogram
Anchor	Anchor QEA, LLC
ASAOC	Administrative Settlement Agreement and Order on Consent
bgs	below ground surface
bml	below mud line
bss	below sediment surface
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
cfs	cubic feet per second
CH2M	CH2M HILL Engineers, Inc.
Chevron	Chevron U.S.A., Inc.
cm	centimeter(s)
cm/yr	centimeter(s) per year
COC	contaminant of concern
CRD	Columbia River Datum
CUL	cleanup level
DDD	dichlorodiphenyldichloroethane
DDE	dichlorodiphenyldichloroethylene
DDT	dichlorodiphenyltrichloroethane
DDx	sum of six isomers associated with DDT, DDD, and DDE
DEQ	Oregon State Department of Environmental Quality
DQO	data quality objective
EPA	U.S. Environmental Protection Agency
ESD	Explanation of Significant Differences
FMD	future maintenance dredging
FS	Feasibility Study
FSP	Field Sampling Plan
HSP	Health and Safety Plan
HxCDF	hexachlorodibenzofuran
Jacobs	Jacobs Engineering Group Inc.
KMLT	Kinder Morgan Liquids Terminals, LLC
LIF	laser-induced fluorescence

Acronym	Definition
LNAPL	light non-aqueous phase liquid
McCall	McCall Oil and Chemical Corporation
MLW	mean low water
NAPL	non-aqueous phase liquid
NAVD88	North American Vertical Datum of 1988
OF	outfall
OHW	ordinary high water
PCB	polychlorinated biphenyl
PDI	Pre-Design Investigation
PeCDD	pentadioxin
Phillips 66	Phillips 66 Company
PHSS	Portland Harbor Superfund Site
Pre-RD	Pre-Remedial Design
PTW	principal threat waste
QA/QC	quality assurance/quality control
QAPP	Quality Assurance Project Plan
RAL	remedial action level
RD	Remedial Design
RI	Remedial Investigation
RM	river mile
ROD	Record of Decision
Shell	Shell Oil Company
SMA	sediment management area
SOW	Statement of Work
TCDD	tetrachlorodibenzo-p-dioxin
USACE	U.S. Army Corps of Engineers
USACE ERDC	U.S. Army Corp of Engineers, Engineer Research and Development Center
WCPA	Willbridge Cove Project Area
WP	Work Plan
yd^3	cubic yard

1. Introduction

In 2017, the U.S. Environmental Protection Agency (EPA) issued the Record of Decision (ROD) for the Portland Harbor Superfund Site (PHSS) (EPA 2017). The PHSS covers an area along the Willamette River from approximately river mile (RM) 1.9 to RM 11.8. The EPA has divided the PHSS into multiple areas for Remedial Design (RD). This document presents the Pre-Design Investigation (PDI) Work Plan (WP) for the Willbridge Cove Project Area (WCPA), which extends along the western shore of the Willamette River between approximately RM 7.5 and RM 8.1 and is located in Portland's Northwest Industrial District (Figure 1-1). The WCPA encompasses approximately 34.6 acres within the PHSS and is bordered by four upland properties adjacent to the river.

The Willbridge Cove Group, which consists of Chevron U.S.A., Inc. (Chevron), Kinder Morgan Liquids Terminals, LLC (KMLT), McCall Oil and Chemical Corporation (McCall), Phillips 66 Company (Phillips 66), and Shell Oil Company (Shell), entered into an Administrative Settlement Agreement and Order on Consent (ASAOC) for RD (Comprehensive Environmental Response, Compensation, and Liability Act [CERCLA] Docket No. 10-2020-0053) for the WCPA on January 31, 2020. The PDI WP is the second deliverable for the project, consistent with the Statement of Work (SOW) presented in the ASAOC, and has been prepared in accordance with the requirements and guidance in the following documents:

- The Willbridge Cove Project ASAOC (EPA 2020a)
- The PHSS ROD, subsequent Explanation of Significant Differences (ESD), Errata #1, and Errata #2 (EPA 2017, 2019, 2018, 2020b)
- *Remedial Design Guidelines and Considerations—Portland Harbor Superfund Site* (EPA 2020c)

The PDI WP is being submitted as a final document to EPA as requested in an April 5, 2021, email from Ben Leake of EPA to John Lang (Willbridge Cove Project Coordinator) and follows submittals of draft and revised draft work plans and exchanges of comment and response documents. The most recent response to comments document, which addresses comments included in EPA's conditional approval of the revised draft PDI Work Plan, is provided as Attachment 1 of this work plan.

1.1 Pre-Design Investigation Purpose

The purpose of the PDI is to identify and address data gaps to characterize and refine the preliminary sediment management areas (SMAs) within the WCPA for RD purposes. Data gathered during the PDI will be used to supplement the existing data to support refinement of SMA boundaries and evaluate the stability of sediment within the WCPA. Once the PDI data are available, the PDI and existing sample results will be evaluated in the context of the dredging history, current bathymetry, and the overall depositional nature of the WCPA, and some data replacement may be proposed in accordance with Section 1.4 of the RD Guidance (EPA 2020c). The PDI field investigation activities, the results of the SMA refinement, and stability evaluations will be documented in the PDI Evaluation Report.

1.2 Willbridge Cove Project Area Description

Four adjacent and currently operating petroleum storage and transfer terminals are directly upland to the WCPA: KMLT Willbridge Terminal, Chevron Willbridge Terminal, Phillips 66 Willbridge Terminal, and McCall Terminal. Each facility has a dock for petroleum product transfer to/from marine transport vessels within the WCPA. In addition, Tidewater Barge Lines, Inc. (Tidewater) leases the turning basin from McCall and parks spud barges in the area upstream of the Phillips 66 dock. The properties where KMLT Willbridge, Chevron Willbridge, and Phillips 66 Willbridge Terminals (known collectively as Willbridge

Terminals in some documents) operate are bisected by NW Front Avenue. The bulk storage facilities are inland of NW Front Avenue, between NW Front Avenue and Highway 30.

The PHSS ROD and ESD identified five preliminary SMAs for the WCPA (Figure 1-2) based on surface sediment data available at the time of the PHSS Feasibility Study (FS). The ROD and ESD divided the river into specific areas based on the current and anticipated future uses, which were the primary determinants of remedial technologies to be implemented at confirmed and refined SMAs. The WCPA contains dock structures and other facilities that support active shipping operations, and periodic maintenance dredging is required to maintain adequate water depths (Figure 1-2). The current authorized or operational depths are summarized in Table 1-1. Accordingly, the WCPA is dominated by future maintenance dredging (FMD) areas, but also includes intermediate and shallow water areas, as well as river banks (Figure 1-2).

The current and future uses of the WCPA, as well as the expected remedial approaches to areas of contaminated sediment, are key factors in evaluating whether enough data exist to characterize and refine SMAs for RD purposes. The following bullets outline the factors to be considered for each designated use and anticipated remedial technology in the WCPA:

- The anticipated remedial technology for contaminated sediment in FMD areas per Section 14.2.2 of the ROD is dredging to the depth of the site-wide remedial action level (RAL) concentrations or to a depth required to allow placement of a cap or backfill sufficient to be effective over the long term. Where RALs are achieved through dredging, placement of a residual layer will occur as soon as is practicable following dredging within the prism and surrounding area that may have been impacted by dredge residuals. Maintenance dredge depth requirements need to be considered during design and implementation of the remedy such that the final constructed elevation is below the maintained depth, including an over-dredge allowance or buffer zone.
- Several of the WCPA preliminary SMAs extend beneath overwater structures such as docks. The anticipated remedy for contaminated sediment in these areas is capping. Caps placed below or adjacent to overwater structures will consider the logistics of placing capping material below structures and any physical constraints adjacent to the structure, including sediment bed slope, current and future navigation uses, and propeller wash during design.
- Portions of three of the WCPA preliminary SMAs also extend into the intermediate regions between the FMD area and the river bank. The anticipated remedy for these areas is dredging to the depth required to achieve RALs and remove principal threat waste (PTW), or to a depth required to allow placement of cap or backfill material sufficient to be effective over the long term. The elevation of the top of the cap will be no higher than the pre-design elevation to avoid impacts to the floodway. EPA estimated the dredging depth required to accommodate a cap will generally be 5 feet.
- There are no ROD river banks identified in the WCPA. Other types of river banks may be included in the remedial action if contamination contiguous with contaminated river sediment is found during RD sampling. If river bank soil contamination is found, engineered caps or vegetation with beach mix will be placed as the final cover based on area-specific designs. If the contaminated soil contains PTW, or has potential for erosion, then the soil may be excavated and/or capped. Otherwise, the remedy will be to monitor.

The anticipated remedial technology for the remainder of the WCPA (outside of identified SMAs) is monitored natural recovery.

1.3 PDI Data Quality Objectives

The data gap evaluation process and plans for collection of additional data are based on site characteristics and data quality objectives (DQOs) developed for confirmation and refinement of SMAs in the WCPA and other RD considerations. Information used to develop the DQOs for this study consist of:

- Site characteristics, including hydrology and sedimentation rates for the WCPA, dredging history, and site features.
- Preliminary SMA boundaries and anticipated remedial technologies identified in the PHSS ROD (EPA 2017) and the ESD (EPA 2018).
- RD principles and recommended sampling design elements described in *Remedial Design Guidelines and Considerations – Portland Harbor Superfund Site* (EPA 2020c).
- Sediment data within the PHSS FS database and the 2018 Pre-Remedial Design (Pre-RD) investigation databases (further discussed in Section 2.2).
- River bank soil sample data collected at Willbridge Cove Group properties as part of source control evaluations (further discussed in Section 2.2).

Three DQO problem statements have been developed for the PDI based on these considerations:

1) The nature and extent of SMAs in the WCPA need to be sufficiently delineated and refined to support technology selection and RD. There are three decisions to be made using new PDI sample data and previously-collected (FS and Pre-RD) sample data obtained within and adjacent to the preliminary SMAs:
    a) Confirm the presence of an SMA.
    b) Determine the spatial distribution of contaminants of concern (COCs) relative to site-wide RALs and PTW thresholds in surface sediment.
    c) Determine the vertical distribution of COCs relative to site-wide RALs and PTW thresholds in subsurface sediment to a depth appropriate to support design of the applicable remedial technology for the area.
2) The physical and chemical stability of contaminated sediments at depth need to be sufficiently assessed to evaluate whether subsurface contamination has the potential to be exposed or recontaminate cleaner surface sediment. Stability will be evaluated using new PDI data collected in selected areas and combined with previously-collected sample data and other studies (e.g., bathymetric surveys).
3) The possible presence and extent of river bank contamination contiguous to preliminary SMAs needs to be sufficiently evaluated and refined to support technology selection and RD, if required.

The outcomes of the seven step DQO process for each problem statement are summarized in Tables 1-2, 1-3, and 1-4.

1.4 Data Evaluation Approaches

This section describes the data needs and evaluation approaches that will be used to address each of the DQO problem statements listed above. The data gaps evaluations presented in Sections 2.3, 2.4, and 2.5 of this PDI WP evaluate the sufficiency of existing data relative to these needs and identify gaps to be filled by the PDI sampling effort.

1.4.1 DQO 1 – SMA Refinement

The boundaries of the preliminary SMAs identified in the ROD and ESD will be confirmed and refined by identifying the existing samples that are still representative of current conditions, identifying new data that are needed, and comparing those sample results to site-wide RALs and PTW thresholds for focused COCs, and preparing maps and cross-sections depicting the results for inclusion in the PDI Evaluation Report.

The data set used for SMA refinement will consist of the existing FS and Pre-RD sediment databases and new sediment sample data from the PDI that will be collected and analyzed as described in Section 3.1. However, certain types of older sample results will be excluded or replaced in the PDI evaluation data set, as described below:

- Historical samples collected from locations and intervals that have been dredged will not be included in the PDI evaluation data set. The data gap evaluations in Section 2.3 of this WP describe these samples and the rationale for exclusion. This information will also be provided in the PDI Evaluation Report.
- Historical surface sediment samples collected in areas where there are adjacent, more-recent samples that indicate changed conditions will not be included in the PDI evaluation data set. These samples will be replaced by more-recent sample results from the immediate vicinity of the historical samples in accordance with Section 1.4 of the RD Guidance (EPA 2020c). As discussed in Section 3.1, three more recent surface sediment samples will generally be required to replace any older non-representative sample.
- Historical surface samples where RAL exceedances were significantly influenced by the inclusion of nondetect results in the sums (most commonly historical polychlorinated biphenyl [PCB] Aroclor data with elevated reporting limits). These samples will be replaced by more recent samples analyzed using methods with lower reporting limits.
- The PDI Evaluation Report will document the data replacement decision process and provide the rationale for excluding historical samples from the PDI evaluation data set. The data gaps evaluation in Section 2.3 of this WP describes the samples that are being considered for replacement and the sampling designs in Section 3.1 identify the proposed locations for the replacement samples.

1.4.2 DQO 2 – Physical and Chemical Stability of Buried Contamination

A multiple lines-of-evidence approach will be used to evaluate the physical stability of buried contamination in the WCPA, consistent with the *Technical Guidelines on Performing a Sediment Erosion and Deposition Assessment (SEDA) at Superfund Sites* (USACE ERDC 2014). The following information will be considered in this evaluation:

- Sediment accumulation rates based on bathymetric differencing analysis
- Local hydrodynamics and physical conditions, including erosion or deposition caused by wind- and wake-generated waves, currents, propeller wash, anchor drag, underwater structures, and other sources
- Surface sediment/subsurface sediment characteristics, including physical parameters and chemical concentration profiles and ratios developed from existing data and data representative of current conditions combined with new data acquired during this PDI required to address DQO 1
- Ratio of critical bed shear stress to total bed shear stress (e.g., assuming 2-year flood conditions).

The chemical stability evaluation will use existing data and data acquired during PDI sampling in a one-dimensional numerical contaminant transport model (CapSim)[1] to evaluate the potential for advection/diffusion of contaminants in buried sediment to recontaminate cleaner surface sediment.

The data gaps section for DQO 2 (Section 2.4) summarizes the existing and proposed PDI data for the WCPA relative to the physical and chemical stability evaluation data needs. The results of the physical and chemical stability evaluations will be documented in the PDI Evaluation Report.

1.4.3 DQO 3 – Nature and Extent of River Bank Contamination Contiguous with an SMA

The process for evaluating the nature and extent of river bank contamination contiguous with an SMA consists of first identifying the preliminary SMAs that are likely contiguous with a river bank and are likely to be included in the remedial footprint. Existing FS and Pre-RD data are used to make this determination. If existing surface sediment results for locations between the preliminary SMA boundary and the river bank are below the RALs and PTW thresholds, then the preliminary SMA is not contiguous and river bank characterization is not needed. If there are RAL and PTW threshold exceedances between the preliminary SMA boundary and the river bank, then river bank characterization is needed. The river bank data gaps evaluation in Section 2.5 identifies one river bank area may be contiguous with an SMA.

The river bank characterization process consists of comparing river bank surface soil sample results to site-wide CULs, RALs, PTW thresholds, and regional background values for metals. If COC concentrations in surface soil exceed Table 21 RAL or PTW thresholds, the archived subsurface soil from those respective locations will be analyzed for the Table 21 COCs (excluding chlorobenzene) to characterize the potential vertical extent of the river bank contamination. Maps and cross-sections (if needed) will be prepared depicting the horizontal and vertical extent of RAL and PTW threshold exceedances on the bank for inclusion in the PDI Evaluation Report.

1.5 Work Plan Organization

This document is organized as follows:

- **Section 1: Introduction**—Summarizes the purpose and DQOs for the WCPA PDI. This section also describes the organization of this document.
- **Section 2: Summary of Available Data and Site Conditions**—Summarizes the existing data within the WCPA directly related to the DQOs, describes the physical characteristics of the in-river area and the river banks, provides an evaluation of conditions in the preliminary SMAs, and identifies the data gaps that will be addressed by this investigation.
- **Section 3: Pre-Design Investigation Sampling Design**—Presents the sampling design for the PDI field effort.
- **Section 4: Sampling and Analysis**—Provides an overview of the sample collection and laboratory analytical methods that will be employed to achieve the project objectives.
- **Section 5: Data Management**—Presents an overview of the general field documentation and analytical data management requirements that will be followed during this investigation.
- **Section 6: Data Evaluation and Reporting**—Provides an overview of how the PDI data will be evaluated and reported.
- **Section 7: Schedule**—Presents a preliminary schedule for completing the PDI work.
- **Section 8: References**—Lists the references cited in this PDI WP.

[1] CapSim Version 3.8 developed by the Reible Research Group (Lampert and Reible [2009]; Reible [2014]; updated September 2019).

Tables and figures are located at the end of the text followed by appendixes. The following appendixes provide supporting documentation:

- **Appendix A**—Analytical Data Tables
- **Appendix B**—Field Sampling Plan (FSP)
- **Appendix C**—Quality Assurance Project Plan (QAPP)
- **Appendix D**—Health and Safety Plan (HSP), including an Emergency Response Plan
- **Appendix E**—Waste Management Plan

2. Summary of Available Data and Site Conditions

This section summarizes the available data for the WCPA sediments and river bank soil and identifies the data gaps that will be addressed in this PDI WP.

Section 3.2(a)(1) of the SOW in Appendix A of the ASAOC specifies data gap analysis of the following:

i. Surface and subsurface sediment contamination
ii. Surface water, sediment pore water, and groundwater data
iii. Bathymetry
iv. Flood-rise analysis; and
v. Non-aqueous phase liquid (NAPL) delineation, if applicable

This summary and associated data gap evaluation focuses on surface and subsurface sediment contamination and the physical and chemical stability of subsurface sediment within the WCPA. It also addresses potential river bank soil contamination adjacent to areas where an SMA is expected to be confirmed. Based on the available data and current understanding of WCPA conditions documented in the Draft Revised Sufficiency Assessment Report (Jacobs 2020), data gaps associated with other media, bathymetry, and NAPL are not anticipated. However, if other data gaps are identified, they will be addressed in a Supplemental PDI or in the RD WP. Data evaluations related to flood-rise will be performed as part of the RD after the revised SMA footprints and remedial technology assignments are known.

2.1 Physical Site Characteristics

The ROD for the PHSS (EPA 2017) divided the river into specific areas (Figure 1-2) that influence the selection of remedial technologies:

- Navigation Channel: The Willamette River federally authorized navigation channel has an authorized depth of 43 feet, plus 2 feet over-dredge allowance, from the Columbia River Datum (CRD), which is approximately -37.7 feet North American Vertical Datum 1988 (NAVD88) and varies in width. The channel extends from Willamette RM 0 to the Broadway Bridge at RM 11.6. The U.S. Army Corps of Engineers (USACE) indicates that channel maintenance continues to a depth of 40 feet (USACE 2020).
- FMD Areas: FMD areas are locations that are periodically dredged to allow continued vessel activity, such as docking and loading or unloading materials.
- Intermediate Region: The ROD defines this region as outside the horizontal limits of the navigation channel and FMD areas to the riverbed elevation of approximately -2 feet CRD, which is approximately +3.3 feet NAVD88 in this reach of the river (USACE 2014).
- Shallow Region: The shallow region is defined as shoreward of the riverbed elevation of approximately +3.3 feet NAVD88.
- River Bank Region: River banks are defined as areas from the top of bank down to the river.

The WCPA contains dock structures and other facilities that support active shipping operations and periodic maintenance dredging is required to maintain adequate water depths. Accordingly, the WCPA is dominated by FMD areas. The river bank region extends from the top of bank at approximately 35 feet NAVD88 to the water. The area between the FMD area and the river bank is comprised of a narrow region of intermediate and shallow water.

2.1.1 Hydrology and Sedimentation Rates

The WCPA is located between RM 7.5 and 8.1 on the west bank of the Willamette River, which widens markedly in this area resulting in mostly net depositional areas. Flow discharge measured at RM 7.8 during a high flow event (130,000 cubic feet per second [cfs]) with an average daily mean discharge of 33,000 cfs. Areas of sluggish flow or eddies are present closer to the western shoreline (EPA 2016a). In the nearshore areas of the WCPA, the flow is dampened by in-river structures and the cove itself, resulting in formation of eddies and quiescent conditions that facilitates deposition of suspended sediment.

PHSS bathymetric surveys were compared as part of the Remedial Investigation (RI)/FS and Pre-RD investigations to evaluate net sediment accumulation rates and identify erosional/depositional areas. An analysis of the sedimentation rate from 2009 to 2018 was performed by AECOM and Geosyntec (2019). The data were binned into the following three categories: depositional (greater than 2.5 centimeters [cm] per year [cm/yr]); neutral (-2.5 to 2.5 cm/yr); and erosional (less than -2.5 cm/yr). The results from this analysis and the associated maintenance dredge activities that occurred during this time period are shown on Figure 2-1. The figure shows that the area is predominantly net depositional except in the areas of maintenance dredging or vessel traffic as identified on Figure 2-1. Scour areas on the upstream side of the Phillips 66 dock are due to Tidewater Barge operations in this area.

2.1.2 Dredging History

The KMLT and Chevron berths were last dredged in 2011. The 2011 dredging depth for the upstream side of the KMLT dock was approximately -34 feet NAVD88, and about 26,100 cubic yards (yd^3) of sediment were removed. A 12-inch sand cover was placed over the dredge surface for a final elevation of approximately -33 feet NAVD88 (CH2M 2013). The areas on both sides of the Chevron dock were also dredged in 2011 to an average dredge elevation of -36.2 feet NAVD88. Approximately 13,560 yd^3 of sediment were removed from the berth on the north side of the dock, and about 12,390 yd^3 were removed from the berth on the south side of the dock. The McCall berth was previously dredged to remove 16,000 yd^3 of sand and silt material between 1993 and 1996 to a depth of -34.7 feet NAVD88 from the face of the dock to the edge of the federal navigation channel and to a depth of -19.7 feet NAVD88 along the backside of the dock (Anchor 2019). The McCall dock area dredging is ongoing (2020 to 2021) is expected to be completed in 2021. Other areas of the WCPA, such as the area shoreward of the McCall dock, the upstream Phillips 66 berth, and the downstream KMLT berth are net depositional with expected net accumulation rates of greater that 2.5 cm/year (Figure 2-1).

2.1.3 River Bank Slopes and Surface Conditions

The river banks within the WCPA consist of gently and moderately sloped banks that may include beaches below the ordinary high water (OHW) elevation and steeply sloped armored banks that extend from near the top of the river bank to below the waterline, as summarized below and shown on Figures 2-2 and 2-3. Note that the described conditions are from September 25, 2020 (low river stage) during rainy weather. From north to south, the observed conditions are as follows:

- Downstream of the KMLT dock, the bank is gently sloped near the river and consists of brownish red sand overlying cohesive clay/silt. The sand layer is approximately 12 inches thick near the OHW contour and thins to an inch or less near the water line. The area at the top of bank is well vegetated and stormwater runoff was observed coming from the vegetated area (not part of the KMLT operational area) onto the bank. The stormwater had cut a narrow rill into the sand layer and exposed the underlying silt/clay (Figure 2-2, photo 2).

- Between the KMLT dock and the Chevron dock, the bank nearest the water consists of sand overlying silt/clay (similar to the area downstream of the dock). Surface materials on the remainder of the bank consists of large riprap, asphalt, and gravel (Figure 2-2, photos 3 and 4).
- Between the Chevron and Phillips 66 docks, the bank is very steep and consists of large riprap from the water line to the OHW elevation. The riprap covers and surrounds the sheet-pile cutoff wall. The bank between OHW and the top of bank elevation is covered with coarse gravel and cobbles (Figure 2-2, photos 5 and 6).
- Upstream of the Phillips 66 dock in the vicinity of public outfall 22 (OF-22), the bank above the waterline is steep and covered with large riprap and gravel (Figure 2-2, photos 7 and 8). The sheet-pile cutoff wall around OF-22 is also covered and surrounded by riprap. Sand with scattered gravel was observed along the water line just upstream of the steep bank. The bank downstream of the OF-22 is gently sloped near the water line and mostly sand. The upper portion of the bank is heavily vegetated. Further to the south, WR-11 discharges to a sandy beach adjacent to the northernmost portion of the McCall property (Figure 2-3, photo 1).
- The bank along the remainder of the McCall property is relatively steep and covered by large riprap. This area is well armored with a very thin strip of sand and clay near the waterline. Vegetation was observed near the top of the bank (Figure 2-3, photos 2 and 3).

2.2 Summary of Existing Data

Data used to summarize the nature and extent of contamination in surface sediments and identify areas for further characterization within the WCPA were compiled from the following EPA-approved databases or tables downloaded from the Portland Harbor Data Portal (http://ph-public-data.com/):

- FS Database (http://ph-public-data.com/document/CDMSmith2018/).
- Portland Harbor PDI-Baseline Sample Database (Pre-RD) (http://ph-public-data.com/document/PHRD_2019/). This data set represents the Pre-RD and baseline data collected by Geosyntec and AECOM (2019). These data are referred to as "Pre-RD" data.

The summations included in the EPA-approved FS and Pre-RD databases follow the rules established during the FS. These datasets were reviewed and post-processed to ensure that multiple results for duplicates or splits collected from identical locations and intervals were resolved into a single value for each sample/analyte pair.

Figures 2-4 and 2-5 show the locations of surface and subsurface sediment samples used in the PDI data gaps evaluation. Table 2-1 and Table 2-2 list the sample results for focused COCs within the WCPA for the FS and the Pre-RD samples, respectively. In addition to the results, these tables provide sample dates, depths, and location information, including whether the sample is located within an area subject to periodic maintenance dredging. The sediment and river bank soil CULs, RALs, PTW thresholds for the PHSS COCs, and Oregon background soil concentrations for metals are summarized in Table 2-3. Tables in Appendix A provide full listings of available results for all available ROD Table 17 analytes and selected physical parameters in WCPA sediment samples.

Recent maintenance dredging areas within the WCPA are depicted on Figures 2-4 and 2-5 (note that these are only a subset of the authorized/operational areas shown on Figure 1-2, other areas are not routinely dredged). The KMLT and Chevron areas were last dredged in 2011 and dredging at the McCall dock is ongoing (2020-2021). The recent maintenance dredging is further discussed in Section 2.1.2. Locations of samples collected by KMLT, Chevron, and McCall at the authorized dredge depths are shown on Figure 2-6. The results for the KMLT, Chevron, and McCall samples have not been approved by EPA for use in SMA refinement but are considered as a line of evidence in support of the conceptual site model for

contaminant distribution, which shows cleaner more-recently deposited sediment overlying older more-contaminated subsurface sediment, and are described in Section 2.2.3.

This section provides a summary of the comparisons of surface and subsurface sediment focused COC sample results within and adjacent to the WCPA to PHSS CULs, RALs, and PTW thresholds. The results of these comparisons are indicated by color-coding on Tables 2-1 and 2-2.

2.2.1 Surface Sediment

The distribution of focused COCs and results of comparisons to CULs and RALs in surface sediment within and adjacent to the WCPA are shown on Figures 2-7 through 2-13. These figures use different symbols to depict the sources of the data such as the RI (squares), or the Pre-RD (circles), and different colors to depict the maximum sample concentration relative to CULs and RALs at each sample location (in some instances more than one surface interval was sampled or a location was sampled during more than one event). Nondetect results are indicated by black, detected results below the CUL by gray, detected results between the CUL and site-wide RAL by green, results above the site-wide RAL by yellow, and results above the PTW by orange.

The only focused COCs with exceedances of the RALs in surface sediment are total PCB Aroclors (Figure 2-7), the sum of the six isomers associated with dichlorodiphenyltrichloroethane (DDT), dichlorodiphenyldichloroethane (DDD), and dichlorodiphenyldichloroethylene (DDE) (DDx) (Figure 2-9), and 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) (Figure 2-11). The exceedance locations for PCB Aroclors are older FS samples that correspond with the five preliminary SMAs identified within the WCPA by the ROD. The DDx and 2,3,7,8-TCDD exceedances occur in single, isolated samples.

Surface sediment COC concentrations in the WCPA do not exceed any PTW thresholds for the additional Table 21 COCs (2,3,7,8-tetrachlorodibenzofuran, 1,2,3,4,7,8-hexachlorodibenzofuran [1,2,3,4,7,8-HxCDF], carcinogenic PAHs as benzo(a)pyrene equivalents, naphthalene, and chlorobenzene). The data are presented and screened in Appendix A, Tables A-1 and A-2.

2.2.2 Subsurface Sediment

The distribution of focused COCs and results of comparisons to CULs and RALs in subsurface sediment within and adjacent to the WCPA are shown on Figures 2-14 through 2-19. These figures use the same color-coding scheme described in Section 2.2.1 to depict the maximum subsurface sample concentration relative to CULs and RALs at each sample or core location. The vertical distribution of focused COCs in the vicinity of the preliminary SMAs is discussed further in Section 2.3. Outside of the preliminary SMAs, which are detailed in Sections 2.3.1 through 2.3.4, there are four other areas where the overall stability of the areas will be evaluated as part of the PDI Evaluation Report.

2.2.2.1 Shoreline area Behind McCall Dock

FS core location C413 is located between preliminary SMA 1 and the McCall shoreline. This core and its replicate had subsurface exceedances of the RALs for total PCBs (Figure 2-14) in all core intervals and DDx (Figure 2-15) in only the deepest interval of the replicate core. The surface interval at this location sampled during the FS and again during the Pre-RD had concentrations below the RALs.

2.2.2.2 Shoreward area of the Phillips 66 Slip

FS core locations C401 and C695 are located between preliminary SMA 3 and the Phillips 66 and McCall shoreline. These cores had subsurface exceedances of the RALs for total PCBs (Figure 2-14). The sample intervals with exceedances were on the order of 6 to 7 feet below the mudline when the samples were

collected (2004 and 2007, respectively). The significant depth below the sediment surface is consistent with the highly depositional nature of Willbridge Cove.

2.2.2.3 Immediately downstream of the Phillips 66 Dock

The area between the Phillips 66 dock (downstream side) and the Chevron maintenance dredge area includes several cores collected for dredge material characterization (DMMU1, DMMU2, DRB-1 and DRB-3). The analytical results for these cores were included in the FS dataset. Subsurface sediment samples from these four locations exhibited total PCB concentrations above the RALs (Figure 2-14). The cores collected from this area were either long composites (on the order of 10 feet or more) or were targeted to look at the anticipated post-dredge surface for the area. This area is expected to have similar characteristics as the area on the upstream side of the Phillips 66 dock near preliminary SMA 3 due to the similar dredging and deposition history.

2.2.2.4 Downstream of the KMLT Dock

The FS and ROD did not identify a preliminary SMA in this area. All surface sediment samples were below RALs for all six focused COCs (Figures 2-7 to 2-13). Subsurface samples in one or more locations exceeded the RALs for total PCBs (Figure 2-14), DDx (Figure 2-15), TCDD (Figure 2-17), and pentadioxin (PeCDD) (Figure 2-18). In the cores collected during the RI/FS, RAL exceedances were typically below depths of 5 feet. In the Pre-RD location (SC-S155), the sampling interval from 2.1 to 4.2 feet below the mudline exhibited nominal exceedances of the RALs for TCDD and PeCDD.

Subsurface samples from FS locations C690 and C377 and Pre-RD location S155 on the downstream end of the WCPA, exhibited subsurface concentrations of 1,2,3,4,7,8-HxCDF above the PTW threshold. These results were from sampling intervals 8 feet or more below the mudline in the FS locations and in a single interval from 2.1 to 4.2 feet below the mudline in location S155.

2.2.3 Summary of Samples Collected to Support Maintenance Dredging

Samples have been collected from the authorized depths in the KMLT, Chevron, and McCall maintenance dredge prisms to support the dredge permitting process (Figure 2-6). These results are briefly summarized in the following subsections but were not used to make decisions in the data gaps evaluation presented in Section 2.3.

2.2.3.1 Kinder Morgan

Sediment grab samples were collected from four locations within the Kinder Morgan dredge prism in October 2011 after the maintenance dredging was performed and prior to the placement of 1-foot thick sand layer. The samples were analyzed for volatile organic compounds, PAHs, DDx, PCBs, metals, and total petroleum hydrocarbons (as diesel range organics). The compounds with RALs (PAHs, DDx, and PCBs) are discussed briefly below:

- PAH compound were largely non-detected, and the three constituents that were detected (fluoranthene, phenanthrene and pyrene) were detected at concentrations less than 300 micrograms per kilogram (µg/kg).
- Total PCB Aroclors ranged from below the detection limit to 68 µg/kg (sums include one-half the detection limit for non-detected Aroclors). Where the field duplicate was collected, the parent sample had a result of 77 µg/kg and the duplicate was 49 µg/kg; consistent with the PHSS precedent, the average result of 63 is considered for this evaluation).

- Total DDx ranged from below the detection limit to 23 µg/kg (sums include one-half the detection limit for non-detected isomers).

2.2.3.2 Chevron

Sediment samples were collected from four location along the edges of the Chevron dredge prisms, and one location between the end of the dredge prism and the shoreline (S-3, Figure 2-6). These samples were collected in October 2012, after the maintenance dredging had been performed. The dredging permit did not require the placement of a sand layer. Samples were collected from 0 to 0.5-foot and 0.5 to 1-foot at locations S-2 and S-5. At locations S-1, S-3, and S-4 the samples were collected from the 0 to 1-foot and 1 to 2-foot intervals. The samples were analyzed for volatile organic compounds, DDX, PCBs (as Aroclors), PAHs, total petroleum hydrocarbons, and organotins:

- Total PAHs were detected at low concentrations (below CULs and RALs), except for at location S-3 in the 1- to 2-foot sampling interval.
- Total PCB Aroclors were detected at locations S-1, S-2, and S-3; however, the reporting limits were elevated, and the laboratory report noted potential matrix interferences.
- DDx isomers were not detected; however, reporting limits were elevated (ranging from 10 to 300 µg/kg).

2.2.3.3 McCall (2019)

Samples were collected in March 2019 to support the permitting for planned maintenance dredging riverward of the McCall dock. The bank side of the dredge prism, adjacent to the dock and adjacent to, or overlapping, preliminary SMA 1 is defined as dredge material management unit (DMMU) 10. Four samples representing the post-dredge surface were collected and analyzed for PHSS Table 21 parameters. A single RAL exceedance for 1,2,3,7,8-PeCDD was reported at location DMMU10-01Z (Figure 2-6). The RAL exceedance ratio was 1.6 and the other three post-dredge samples from DMMU 10 were below the site-wide RAL. The sediment characterization report (Anchor 2019) documented that the dioxin/furan concentration in the McCall dredge prism were consistent with concentrations observed in upriver background areas and the Downtown Reach of the Willamette River. The sediment characterization report (Anchor 2019) documented that the slope at DMMU 10 was stable relative to the adjacent authorized dredge depths; therefore, a sand cover layer in DMMU 10 was not required.

2.2.4 River Bank Soil

The river bank region is defined in the ROD (EPA 2017) as "areas from the top of bank down to the river that may be contaminated along the shoreline next to contaminated in-river shallow areas." Data used to summarize the nature and extent of contamination in-river bank soil samples were compiled from the following sources:

- Bank Soil Delineation Report Kinder Morgan Willbridge Terminal (Jacobs 2019)
- Erodible Soil Assessment Report, Kinder Morgan, Chevron, and ConocoPhillips Willbridge Terminals (Delta 2008)
- Updated Source Control Evaluation Report McCall Oil and Chemical Site (Anchor 2011)

Figures 2-20 and 2-21 show the locations of river bank soil samples used in the river bank evaluation. The analytical data for the river bank soil samples are included as Tables A-3a through A-3d in Appendix A.

The sediment and soil data are compared to the PHSS CULs, RALs, and PTW thresholds presented in Table 17 and Table 21 of the ROD and updated to reflect changes in the ESD (EPA 2019).

A brief discussion of the KMLT river bank soil results from 2008 and 2012 is warranted given the elevated concentrations of DDx found beneath the KMLT dock. The elevated concentrations beneath the KMLT dock are attributed to differences in the sediment composition beneath the dock, which is finer-grained and has higher organic content than the beach sands on either side of the dock. Because DDx compounds preferentially sorb to organic carbon in the soil, elevated concentrations appear to be associated with higher organic carbon content in soil beneath the dock. Pier pilings and riprap directly beneath the dock likely contribute to reducing wave and current energy, allowing finer material to accumulate. Additionally, there are no known release or transport mechanisms for DDx to migrate from the Terminal to the area beneath the dock.

There are no records or information to indicate DDT was transported, spilled, or released from the Willbridge dock. Although DDT was stored in Tank 4 at the Willbridge Terminal for a short period in the 1950s, site records indicate DDT was transported to the site in bags, mixed onsite, and transported offsite in trucks, therefore eliminating spillage off of the dock as a potential source of DDx compounds under the dock. Tank 4 is located approximately 1,000 feet from the dock area. There is no known transport mechanism for DDx to be transported from Tank 4 to the dock area. Soil investigations conducted in the Tank 4 area have delineated the extent of DDx impacts around Tank 4, and impacts appear to be localized around the tank. Additionally, a comparison of river bank soil DDx sample results and Tank 4 DDx sample results was performed to assess the nature and age of the DDx compounds. Specifically, the ratios of DDT to its degradation products (DDD and DDE) from the Tank 4 area and from the bank soil were compared. These ratios can be used as a rough estimate of the period of DDT application or release (Tavares et al. 1999). Based on the ratios of DDT to DDD and DDE, it appears the DDx detected in the Tank 4 area is an older release, and not related or associated with the DDx detected along the bank.

Follow-up soil sampling was performed in 2017 to further delineate the boundaries of the DDx-impacted soil below the dock. Approximately 6 yd^3 of soil were removed from beneath the dock in 2017 in a partially completed removal action. During excavation activities, petroleum hydrocarbon impacts were observed. Additional data were also obtained to determine hydrological conditions under and near the dock. This investigation was performed in 2018 and included 10 laser-induced fluorescence (LIF) borings to test for the presence of light non-aqueous phase liquid (LNAPL). Soil samples were collected from six soil borings. Surface samples and at least one subsurface sample were collected at all boring locations. DDx exceeded the CUL in five of the six surface samples, and none of the subsurface samples. Individual dioxin constituents exceeded RALs in five of the six surface locations and 2 of the 10 subsurface locations. One surface sample, located at 23 feet NAVD88 above the OHW elevation, exceeded the PTW criteria for 1,2,3,4,7,8-HxCDF. No surface samples exceeded the RAL for PCBs and the RAL for total PAHs were not exceeded in any sample location. The LIF investigation concluded LNAPL is present below residual saturation levels and cannot be hydraulically recovered or mobilized.

Additional removal measures are planned to remove shallow soil beneath the dock in 2021 (CH2M 2017). If an SMA is confirmed in this area and appears to extend to this portion of the river bank, the post-removal confirmation sample data will be considered in the RD for the area.

2.3 Data Requirements for Refinement of Preliminary Sediment Management Areas (DQO 1)

The following section provides a discussion related to the understanding of the current conditions within the WCPA to guide additional sampling to support refinement of SMAs in the WCPA as outlined in DQO 1. The preliminary SMAs for the WCPA are numbered 1 through 5 for this discussion.

The following evaluation focuses on areas within and immediately adjacent to preliminary SMAs where one or more focused COCs exceed a RAL in surface or subsurface sediment, and considers whether the FS

data are representative of current conditions based on dredging history, deposition and bathymetric changes, the results of more recent sampling, and sediment stability. These attributes will be considered in the sampling design for each preliminary SMA area presented in Section 3.

As indicated in Section 2.2.1, concentrations of three of the six focused COCs in surface sediment in the WCPA are below the RALs. The exceptions are total PCB Aroclors, DDx, and 2,3,7,8-TCDD, which exceed the RALs in one or more samples. The general distributions of these focused COCs are described below:

- **Total PCBs**—The distribution of total PCBs as summed Aroclors in surface sediment is shown on Figure 2-7. There are total PCB Aroclor results above the RAL within the WCPA that correspond with the five preliminary SMAs. Many of the samples with exceedances are located in areas that were dredged in 2011 (dredge prism outlines are shown on Figure 2-7). There are no RAL exceedances in the PCB congener dataset, which are primarily from the Pre-RD sampling (Figure 2-8).
- **Total DDx**—The distribution of total DDx in surface sediment is shown on Figure 2-9. There is one sample within the WCPA with an exceedance of the RAL (Sample G394). The sample is collocated with a total PCB Aroclor exceedance in one of the preliminary SMAs.
- **2,3,7,8-TCDD**—The distribution of 2,3,7,8-TCDD in surface sediment is shown on Figure 2-11. There is one sample within the WCPA with an exceedance of the RAL (sample S158). The sample is not collocated with any other focused COC exceedances and consists of a J-qualified value of 0.00064 µg/kg nominally above the RAL of 0.0006 µg/kg.

Cross-sections have been developed to present total PCB results relative to current and historical sediment surfaces. The cross-section locations are shown on Figure 2-22A and the respective cross-sections A-A′ through E-E′ are shown on Figures 2-22B through 2-22F. These cross-sections depict the 2018 bathymetry (assumed to be the near-current sediment surface) as a solid black line, the average achieved dredge elevation from 2011 as a dashed orange line where applicable, and the inferred bathymetric surfaces from sample elevations recorded during the FS and Pre-RD sampling events as a dashed brown line.

2.3.1 Preliminary SMA 1

Preliminary SMA 1, shown on Figure 2-23, is located on the shoreward side of the McCall dock and was established based on a RAL exceedance for PCB Aroclors in one FS surface sediment sample from 2004 (G407). The sample was collected at an elevation of -12 feet NAVD88 in an area that is depositional but is not dredged as it is on the shore side of the dock structure. During the Pre-RD, three surface samples were collected within this preliminary SMA (S164, B251, and S165) and three samples were collected adjacent to this SMA (B246, B257, and B253). Concentrations of total PCB congeners in all the Pre-RD samples from this area were below the RAL, including sample B251, which was less than 50 feet from G407. Additionally, total PCB Aroclor concentrations in surface and subsurface samples obtained from nearby FS cores at C698 and C403 were below the RAL. Replicate cores were collected at location C413, shoreward of the preliminary SMA boundary; the results are shown on Figure 2-23. The FS surface sample at this area was below the RAL and this finding was confirmed by the surface sample collected during the Pre-RD (B253); this location is outside of the preliminary SMA footprint.

The vertical profile for total PCBs across preliminary SMA 1 from the McCall river bank to the navigation channel is shown in Cross-Section A-A′ (Figures 2-22A and 2-22B). The cross-section depicts the 2018 bathymetry (assumed to be the near-current sediment surface) as a solid black line, and the inferred bathymetric surface from 2004 near G407 is shown as a dashed brown line. Based on comparison of the sample elevation reported in the FS database and the 2018 bathymetry, approximately 4.5 to 5 feet of sediment appears to have been deposited in the vicinity of this sample since 2004. However, there is

uncertainty associated with this comparison due to the degree of precision and accuracy associated with the recorded sample elevations and the measured GPS coordinates of the G407 sample.

The downstream Pre-RD sample S164 was collected at a similar elevation as G407. The Pre-RD data suggest the older FS surface result is unlikely to be representative of current conditions. Proposed sediment sampling is discussed further in Section 3.

2.3.2 Preliminary SMA 2

Preliminary SMA 2, shown on Figure 2-24, is located in the riverward portions of KMLT's slip and Chevron's downstream slip, which, due to the depositional nature of the cove, require regular maintenance dredging to maintain appropriate depths for vessel operations. The history of sampling, dredging, and deposition is critical to understanding how surface sediment conditions in this area have changed over time. Because of that history, the sediment represented by many of the FS surface samples used to identify the preliminary SMA is no longer present. The use of older PCB Aroclor results with elevated detection limits also needs to be considered as part of this evaluation. During the 2018 Pre-RD sampling, five surface grabs (S157, S158, S159, S161, and S162) and one core location (SC-S157) were collected from within this preliminary SMA. The data from the more recent sampling event is considered more indicative of current conditions, particularly within the dredged areas, as the Pre-RD samples measured the quality of sediment that had been deposited since the 2011 maintenance dredging event described in Section 2.1.2.

Preliminary SMA 2 was established based on PCB Aroclor results from surface sediment samples collected at four locations: AN-CTPD-02, AN-CTPD-03, AN-CTPD-06, and AN-CTPD-07 (white or yellow squares on Figure 2-24). Note that sample symbol colors depicted on the figure are determined by the maximum detected concentration in the surface and subsurface sediment intervals at each location. Two surface sediment intervals were collected from each AN-CTPD location in 2002, a 0 to 2 cm sample and a 0 to 15 cm sample. The reporting limits for the individual Aroclors in these 2002 samples were on the order of 23 to 60 µg/kg, resulting in total PCB sums greater than the RALs that are substantially influenced by nondetect values in all of these samples. However, as shown on Figure 2-24, the shallow (0 to 2 cm) interval at locations AN-CTPD-03 and AN-CTPD-06 were resampled for total PCB Aroclors with lower detection limits in 2009 and neither 2009 sample contained PCB Aroclors above the detection limit.

Samples AN-CTPD-02 and AN-CTPD-07, collected in 2002, are in areas that were dredged by Chevron in 2011 and the sediment represented by those samples is no longer present, as shown on Cross-Sections B-B′ and C-C′ (Figures 2-22A, 2-22C, and 2-22D). Concentrations of total PCB Aroclors in nearby 2008 pre-dredge cores used to characterize the post-dredging surface and underlying sediment in the dredge prism (subsurface samples at locations DMMU-1C and DMMU-2C) and a Pre-RD sample (S159) from similar elevations were below the RAL (Figure 2-24).

Cross-section profiles of total PCBs across preliminary SMA 2 from the head of the Chevron and Phillips 66 slips to the navigation channel and from upstream to downstream on the riverward portion of preliminary SMA 2 are shown in Cross-Sections B-B′, C-C′, and D-D′ (Figures 2-22A, 2-22C, 2-22D, and 2-22E, respectively).

The 2002 samples AN-CTPD-03 and AN-CTPD-06, which are within the preliminary SMA but outside of the dredge prism, were collected at elevations of -39 feet and -38 feet NAVD88, respectively, and both were located in apparent scour holes caused by docking vessels, as evidenced by the 2018 bathymetric surface (scour is sufficient to maintain operational depth in these areas without dredging). As shown on Cross-Sections C-C' and D-D′ (Figures 2-22D and 2-22E), the surface interval represented by the 2002 and 2009 AN-CTPD-03 samples appears to now be buried by 3 feet or more of sediment, and the location where the 2002 and 2009 AN-CTPD-06 samples were collected appears be at a similar elevation as the

2018 bathymetric surface. Given the very dynamic conditions in these areas due to ongoing deposition and periodic scouring, the 2002 results are not expected to be representative of current surface conditions. The total PCB concentration in Pre-RD sample S161 collected at a similar elevation (-38 feet NAVD88) near the AN-CTPD-06 samples did not exceed the RAL, and this more recent sample is anticipated to be representative of the area on the upstream side of the Chevron dock. Current conditions in the scour area downstream of the Chevron dock (near AN-CTPD-03) are unknown and represent a data gap.

A portion of the KMLT slip that was dredged in 2011 overlaps preliminary SMA 2. There were no FS samples in this area and total PCB congener concentrations in the surface and subsurface samples obtained from Pre-RD sampling locations S157 and S158 in this area were all below the RAL. The samples from the S157 core extended to a depth of 15.9 feet below sediment surface (bss). The exceedance of the RAL for 2,3,7,8-TCDD at S158 is J-qualified and, if rounded to four decimal places, would not exceed the RAL.

The available data discussed above, coupled with the dredging and depositional history, indicate that the FS data used to establish this preliminary SMA is no longer representative, either because the sediment that was sampled was removed by maintenance dredging or because the sampled areas are in very dynamic environments (scour holes). The 2018 Pre-RD results provide an indication of what the current conditions are; however, additional spatial coverage and some additional subsurface data are needed to refine this preliminary SMA. Proposed sediment sampling is discussed in Section 3.1.

2.3.3 Preliminary SMA 3

Preliminary SMA 3, shown on Figure 2-25, is located upstream of the Phillips 66 dock and was established during the FS based on RAL exceedances of PCB Aroclors in surface sediment samples collected at the following three locations: C532, G694, and SD117. Deeper samples, ranging from 1 to approximately 12 feet bss were collected from cores at C532, SD117, APS-3, and URB-1, which are located within the preliminary SMA boundary. The locations, concentrations, and sampling intervals where total PCBs exceed the RAL in subsurface sediment are identified on Figure 2-25. Other focused COCs analyzed in the FS cores listed previously did not exceed RALs in subsurface sediment. During the Pre-RD, a core was collected at location SC-163 to a depth of 12.7 feet; the surface interval at this location did not exceed any RALs. Subsurface sediment in all intervals collected exceeded the RALs for PeCDD and TCDD; two intervals also exceeded the RAL for total PCBs and the interval from 10 to 12.7 feet below mud line (bml) exceeded the PTW threshold for 1,2,3,4,7,8-HxCDF. Cross-Section E-E' (Figures 2-22A and 2-22F) presents a vertical profile of selected samples upstream of the Phillips dock. The available data suggests that at locations NA-3B, G694, S163, and C532, the surface elevation may not have changed substantially during the time between the FS sampling and the 2018 bathymetry survey; however, other portions of the area upstream of the Phillips 66 dock are net depositional with expected accumulation rates of greater that 2.5 cm per year (Figure 2-1), except in areas that may be subjected to anthropogenic erosion from barge traffic.

Results from the Pre-RD surface samples S163 and B241 are below the RAL; however, the current conditions in the central area of the slip near locations C532, G694, and SD117 are not well defined and additional subsurface data representing finer sampling intervals would be needed to inform RD. Proposed sediment sampling is discussed in Section 3.1.

2.3.4 Preliminary SMA 4 and Preliminary SMA 5

Preliminary SMA 4, shown on Figure 2-26, is located at the head of the Chevron slips and borders the Chevron river bank. This area was identified based on total PCB Aroclors above RALs in four surface sediment samples from three locations, G394, DMMU-3-B, and AN-CTPD-08. The concentration of DDx in

G394 also exceed the RAL. A deeper subsurface sample obtained from the core at DMMU-3-B also contained concentrations of total PCB Aroclors and DDx above the RALs. During the 2018 Pre-RD one surface sample was collected from within this preliminary SMA (S160).

Surface samples AN-CTPD-08A and AN-CTPB-08B were collected in 2002 and are within the area that was dredged in 2011, as shown on Cross-Section C-C' (Figure 2-22D). Concentrations of total PCBs in nearby dredge characterization samples from location DMMU-2-A, which represent the post-dredge surface and the interval immediately below the dredge prism and the Pre-RD surface sample S160, were nondetect or below the RAL and further represent the current surface conditions in this area.

FS sample locations G394 and DMMU-3-B are located in an undredged, steeply sloped area along the river bank between the Chevron and Phillips 66 docks. A sediment sample at GSP07W further up the slope contained total PCB congeners below the CUL and RAL. There are no recent surface sediment samples in this area. The locations of samples G394, DMMU-3-B, and GSP07W are also presented on Cross-Section C-C' (Figure 2-22D). Three samples of river bank soil adjacent to this area were collected and analyzed in 2007. DDx was detected at concentrations above the CUL, but below the RAL in these soil samples. PCBs were reported as not detected, but the detection limits for the soil sampling event were not documented. Preliminary SMA 5, also shown on Figure 2-26, is in the central to shoreward portion of Chevron's downstream slip and was established based on the total PCB Aroclor RAL exceedances in two samples obtained from the surface interval at AN-CTPD-01A in 2002. The area that was dredged in 2011, and the sediment represented by the 2002 samples is no longer present as shown in Cross-Section B-B' on Figure 2-22C. Concentrations of total PCBs in nearby core samples DMMU-1-A and DMMU-1-B collected to represent the post-dredge surface and the interval beneath the post-dredge surface were below the RALs. Location B239, sampled during the 2018 Pre-RD, is approximately 44 feet from the edge of this preliminary SMA boundary and the sediment at this location does not exceed applicable RALs.

Current conditions in this area are not well defined and there are limited bounding samples, constituting a data gap. Proposed sediment sampling is discussed in Section 3.1.

2.4 Data Requirements for Evaluating Physical and Chemical Stability of Buried Contamination (DQO 2)

As outlined in Section 1.4.2, the physical stability of the subsurface sediment will be evaluated throughout the WCPA to assess the potential for exposure of buried sediment by natural or anthropogenic forces. In addition, the chemical stability will be assessed to evaluate the potential for recontamination resulting from porewater diffusion and advection. The lines of evidence summarized in Section 1.4.2 will be developed using the existing data listed below, analyses presented in the PHSS FS, specifically Appendix C and Appendix D8 (EPA 2016b), and additional chemical and physical sediment data that will be collected as part of this PDI. Existing data and evaluations include:

- Existing bathymetric surveys and recent dredging records will be used to evaluate overall sediment accumulation rates and identify propeller scour areas. These surveys will include the river-wide 2004, 2009, and 2018 surveys, as well as multiple, high-resolution surveys performed by individual WCPA parties to support facility operations. Relevant data from the dredging events summarized in Section 2.1.2 in this WP will be incorporated into this analysis.
- Existing evaluations provided in the EPA FS related to wind and wake-generated waves and anchor drag will be reviewed in conjunction with recent bathymetry and current and anticipated future vessel use within the WCPA.
- Existing 2008 side-scan sonar data presented in the PHSS FS (EPA 2016b) and facility provided construction information will be used to evaluate debris and underwater structures.

- The combined chemical and physical property sediment data sets for relevant FS, Pre-RD, and PDI data will be used to inform updated evaluations of the subsurface to surface concentration ratios, evaluations of shear stress ratios, and CapSim modeling. Literature-based partitioning coefficients will be used as inputs to the CapSim modeling.

There are no data gaps specific to DQO 2 that need to be filled beyond the sediment samples that will be collected to address DQO 1 during this PDI.

2.5 Data Requirements to Assess the Nature and Extent of River Bank Contamination Contiguous with SMAs (DQO 3)

Two preliminary SMAs are located adjacent to WCPA river banks: Preliminary SMA 1 adjacent to the McCall river bank, and Preliminary SMA 4 adjacent to the Chevron and Phillips 66 river bank:

- The McCall river bank adjacent to Preliminary SMA 1 does not require characterization per the criteria outlined in Section 1.4.3. Existing data for surface sediment samples collected at G413, B253, 08R002, S164, G403, and B246, which are between the boundary of Preliminary SMA 1 and the McCall river bank, show no exceedances of the RALs and PTW thresholds (see Figure 2-23). Additionally, existing data from the McCall river bank show that contamination is not present in that area and the McCall facility has obtained a Source Control Decision (DEQ 2014) and Conditional No Further Action Determination (DEQ 2016, 2017). Total PCB Aroclor results for the McCall river bank soil samples were below the PHSS CUL of 9 µg/kg site-wide RAL (see Appendix A, Table A-3).
- The Chevron/Phillips 66 river bank adjacent to Preliminary SMA 4 requires characterization. There are no existing sample locations between the boundary of Preliminary SMA 4 and the river bank. Existing river bank soil sample results for the area are mostly from elevations near the top of bank with incomplete target analytes lists and elevated detection limits. Additional samples and other physical information are needed to characterize the nature and extent of possible river bank contamination, evaluate bank stability, and support possible RD. The sampling design for the Chevron/Phillips 66 river bank adjacent to Preliminary SMA 4 is discussed in Section 3.2.

3. Pre-Design Investigation Sampling Design

This section describes the sampling design developed to meet the DQOs established in Section 1.4. Section 3.1 describes the overall sediment sampling program and sampling designs for each preliminary SMA discussed in Section 2.3, and Section 3.2 details the river bank sampling adjacent to preliminary SMA 4 discussed in Section 2.5.

3.1 Sediment Sampling Design

This section provides the rationale for the sampling designs for SMA identification and refinement in each of the data gap areas. The overall sampling approach for each area is summarized in Table 3-1 and the proposed sample locations are shown on Figures 3-1 through 3-4.

3.1.1 Overall Design, Target Analytes, and Archiving Scheme

The sampling designs for each of the four preliminary SMAs incorporates the use of primary and step-out locations. The placement spacing of the primary and step-out locations, both relative to existing sample data and to each other, has been designed to constrain the natural neighbor interpolation and to reduce the influence of the associated smoothing algorithm that has resulted in the long, narrow preliminary SMAs in nearshore areas. The primary sampling locations are intended to determine the presence or absence of the preliminary SMAs, and the step-out locations are designed to further delineate the SMA, if needed. Generally, 150-foot grid spacing is used for characterization and step-out locations. Under-dock locations are generally centered under the dock structure. Target core depths are determined based on site use and water depths, the expected thickness of accumulated sediment, and authorized dredge elevation plus a 3- to 5-foot buffer below the dredge elevation (if dredging is the anticipated remedial technology).

Surface and subsurface sediment samples will be analyzed for ROD Table 21 compounds, except for chlorobenzene. The Portland Harbor RI Report indicated chlorobenzene was a constituent of interest in groundwater only at the Arkema facility (EPA 2016a), and sediment samples collected from the WCPA and analyzed for chlorobenzene during the RI were predominantly nondetect (Appendix A, Table A-1). Of the 31 results, chlorobenzene was detected in only one subsurface sample, with a result of 25 µg/kg, well below the PTW threshold of 320 µg/kg. Therefore, for the sampling effort outlined in this plan, sediment samples will not be submitted for analysis for chlorobenzene.

The primary and under-dock surface sediment grab sample locations for each data gap area will be submitted for immediate chemical analyses. Selected subsurface sediment sample intervals from primary cores in each data gap area will also be submitted for immediate chemical analyses.

The schedule for immediate analysis and archiving is dependent on the sample location and is described below:

- **Intermediate/shallow areas or not regularly dredged FMD areas[2] –** The top five sampling intervals will be analyzed immediately for the Table 21 parameters (surface composite grab plus four 1-foot sample intervals from 1 to 5 feet below mudline).

[2] Current elevations in FMD areas that are not regularly dredged are within 2 ft of the operational elevation. Therefore, characterizing the upper 5 feet of sediment will bracket the operational elevation.

- **Regularly-dredged FMD areas** – Immediately analyze surface composite grab and the four 1-foot intervals bracketing the operational elevation[3]. The surface grab will be used to assess current conditions and is expected to be representative of recently deposited sediment above the operational elevation. The four intervals bracketing the operational elevation will provide the data needed to understand whether additional (deeper) dredging and/or capping may be needed in the regularly-dredged FMD areas at or below the operational depth.

The remaining 1-foot intervals from the primary cores will be archived (i.e., held frozen) for possible later analysis. Proposed locations are shown on Figures 3-1 through 3-4. The proposed analytical and archiving scheme for each data gap area is presented in Table 3-2.

The step-out surface grabs and cores will be collected during the same field event as the primary samples. All step-out samples will be archived for possible later analyses according to the decision rules described below:

1) If the primary samples have concentrations below RALs and PTW thresholds for all Table 21 analytes, then samples from step-out locations will not be analyzed and deeper archived samples from that primary core will not be analyzed.
2) If one or more of the primary samples submitted for immediate analysis have results above the RALs or PTW thresholds, then the samples from the immediately adjacent step-out locations will be released and analyzed for Table 21 analytes (except chlorobenzene). The samples from the step-out locations will be analyzed using the same sequence as the primary locations in that for intermediate, shallow, or not regularly dredged areas, the surface grab composite and four core sample intervals from 1 to 5 feet below the sediment surface will be submitted for analysis. Similarly, if samples from a step-out location in a regularly dredged area are to be released from archive, the surface grab composite and the four intervals bracketing the operational depth will be released for analysis.
3) Data from step-out locations will be assessed using the same decision logic as in Steps 1 and 2, above, to determine if additional step-outs need to be analyzed.

Additional archived subsurface intervals will be analyzed according to the decision rules for primary cores described below:

- In FMD areas that are regularly dredged, it is necessary to know whether contamination above RALs and PTW thresholds is present below the operational or authorized dredge elevation to determine the need for placement of cover material or a cap following dredging. Therefore, if COC concentrations exceed a RAL or PTW threshold in the deepest sample interval below the operational elevation, then the archived samples from the next depth interval in that core will be released and analyzed for Table 21 analytes (except chlorobenzene) at the operational elevation. If that interval also exceeds the RAL or PTW threshold, the next interval will be released and analyzed and so on until the depth of contamination is reached or the bottom interval of the core is analyzed.
- In FMD areas that are not regularly dredged and areas outside of the FMD area, it is necessary to know the depth of contamination relative to RALs and PTW thresholds for remedial technology selection and design purposes; therefore, if concentrations of COCs in the 3 to 5 foot sampling intervals from a core exceed a RAL or PTW threshold, then the archived samples from the next depth interval from that core will be released and analyzed for Table 21 analytes (except chlorobenzene). If that interval also exceeds the RAL or PTW threshold, the next interval will be released and analyzed and so on until the depth of contamination is reached or the bottom interval of the core is analyzed.

[3] Operational elevation is equivalent to the permitted dredge elevation in the regularly dredged FMD areas.

3.1.2 Preliminary SMA 1

This preliminary SMA was identified based on a single FS data point that may no longer be representative of current conditions, as summarized in Section 2.3.1. The 2018 Pre-RD data collected within and adjacent to this preliminary SMA suggest surface conditions have improved; however, additional data are needed to verify the presence or absence of the SMA, and, if it is present, to delineate its areal and vertical extent to inform RD.

The proposed sampling approach is shown on Figure 3-1. This area is primarily an intermediate zone, with some portions within an FMD Area. Sample location G407 will be reoccupied, with a primary core and collocated surface grab collected at that location (PD03), and two primary surface grabs (PD02 and PD04) will be placed near the primary location PD03 to provide three samples for potential replacement of the approximately 15-year old data from G407.

The proposed step-out locations consider the existing FS and Pre-RD data and include three locations upstream, one location between PD02, PD03, and PD04 and the shoreline, and one downstream of the primary sample locations. Step-out locations are not needed on the channel side of preliminary SMA 1 because existing Pre-RD and FS samples show no exceedances of RALs (additionally, the dredge characterization data collected to support the maintenance dredging at the McCall dock indicated a lack of impact in the riverward area) and only one step-out location is positioned downstream of the primary location because there are three existing data points that will be used in the combined data set (C403/G403, B246, and S164). The sample design incorporates existing core data from C403/C698. Cores will be collected to 10 feet bml (or refusal) for possible delineation of the depth of contamination to support removal depth determination and cap design, if needed. Deeper samples are not needed for RD based on a presumed technology assignment of capping.

The step-out locations are positioned on a 150-foot sample grid along the shoreline side of the preliminary SMA to inform the conditions along the remaining area of the preliminary SMA. If one or more RAL exceedances are found in any of the primary samples listed below, then the samples from the corresponding step-out locations will be analyzed following the protocol outlined in Section 3.1.1:

- Primary location (PD02, PD03, or PD04) – Step-out locations PD01, PD05, and PD06
- Step-out location PD06 (if analyzed) – Step-out location PD07
- Step-out location PD07 (if analyzed) – Step-out location PD08

If RAL exceedances are found at PD01, an additional step-out is not required because Pre-RD location S164 can be used to bound the SMA in a northerly direction.

The surface grab composite and sample intervals from 1 to 5 feet below the sediment surface from the primary core location will be analyzed immediately. Deeper intervals will be archived, with the sample intervals analyzed pending results of the initial analysis as described in Section 3.1.1. The combined data set, including FS samples (C403, C698), Pre-RD samples (B246, S164, B251, S165, B253), and additional samples analyzed during this PDI (including primary locations PD02, PD03, and PD04 and step-out samples, if analyzed) will be used to evaluate this area (Table 3-1, Figure 3-1).

3.1.3 Preliminary SMA 2

This preliminary SMA was identified based on FS data, that based on dredging history and the dynamic environment, may no longer be representative of current conditions, as summarized in Section 2.3.2. The 2018 Pre-RD data collected within this preliminary SMA suggest surface conditions have improved; however, additional spatial coverage and subsurface data are needed to verify the presence or absence of the SMA, and, if it is present, delineate its areal and vertical extent to inform RD.

The proposed sampling approach is shown on Figure 3-2. Preliminary SMA 2 spans:

1) Dock structures
2) Areas within the FMD area that are not routinely dredged because scour due to propeller wash maintains the depths at or below the required operational depth
3) Areas within the FMD area that are routinely dredged due to ongoing deposition
4) Areas that are within an FMD area that has not been recently dredged, and given current operational depths and bathymetry, maintenance dredging is not anticipated in the near-future

The sampling design to address each of these areas is discussed in the subsections below.

3.1.3.1 Under Dock Areas

Four primary surface grab samples (as three-point composites) will be collected from under the docks where the preliminary SMA crosses dock structures (PD12, PD16, PD20, and PD26). These samples will be analyzed immediately.

3.1.3.2 Scour Areas

The areas near the end of the Chevron dock are within the FMD area that is not routinely dredged because scour due to propeller wash maintains the depths at or below the required operational depth. Two historical samples in the FS database (AN-CTPD-03 and AN-CTPD-06) exhibited total PCB concentrations above the RALs. Based on the dynamic conditions in this area, additional data are needed to verify current conditions. The proposed locations are generally positioned on a 150-foot sample grid and consider the more recent data collected during the 2018 Pre-RD (S158 and S161). FS location AN-CTPD-03 will be reoccupied and a core and associated surface grab will be collected (PD14). Two additional primary surface grabs (PD13 and PD15) will be placed immediately upstream and downstream of PD14 to provide three samples for potential replacement of AN-CTPD-03 data. Near location AN-CTPD-06, the Pre-RD sample S161 will be used in conjunction with two additional primary surface grab samples (PD17 and PD18) one reoccupying and one adjacent to AN-CTPD-06 to verify current conditions and potentially replace AN-CTPD-06. PD18 will also include a co-located core. At primary sampling locations, the surface grab sample will be analyzed immediately. The four intervals including and below the operational elevation from the primary core locations will also be analyzed immediately, as described in Section 3.1.1

Step-out core and surface grab locations are positioned on the channel side of the preliminary SMA in this area (PD09 and PD10). The cores will be driven to 10 feet bml (or refusal) for possible delineation of the depth of contamination, if needed. The locations and intervals to be analyzed in the step-out cores will depend on which samples in the corresponding primary core or grab locations (if any) exceed RALs (see Section 3.1.3.5).

3.1.3.3 Routinely Dredged Areas

In the areas that are routinely dredged (that is, areas within the 2011 dredge footprints shown on Figure 3-2), existing Pre-RD locations S158, SC-S157, S157, and S159 were used to inform the additional sampling needed. Two primary surface grab and core sampling locations (PD23 and PD28) are within the boundary of the preliminary SMA to verify current conditions. Three step-out locations (PD24, PD25, and PD27) have been positioned around the periphery of this preliminary SMA to further delineate the conditions if this portion of the SMA is confirmed to be present. The cores in this area will be driven to 10 feet bml (or refusal) for possible delineation of the depth of contamination, if needed. At primary sampling locations, the surface grab sample will be analyzed immediately. The four intervals at and below the operational elevation from the primary core locations will also be analyzed immediately, as described

in Section 3.1.1. The locations and intervals to be analyzed in the step-out cores will depend on which samples in the corresponding primary core or grab locations (if any) exceed RALs (see Section 3.1.3.5).

3.1.3.4 Areas Within FMD Not Routinely Dredged

The area on either side of the Phillips 66 dock is within an FMD area that has not been recently dredged, and given current operational depths and bathymetry, maintenance dredging is not anticipated. One primary core with surface grab is located upstream and one primary core with surface grab is located downstream of the Phillips 66 dock (PD19 and PD21) on an approximate 150-foot spacing. The placement is partially based on the results from the 2018 sample S162, which were below RALs. Three step-out cores with surface grabs are included, with two towards the Navigation Channel (PD10 and PD11) and the third located upstream (PD22).

Cores will be extended to 10 feet bml for possible delineation of the depth of contamination to support removal depth determination and cap design, if needed. Deeper samples are not needed for RD based on the anticipated future uses, operational depths, and a presumed technology assignment of capping (at a depth sufficient to be protective in the long-term). The top surface grab composite and sample intervals from 1 to 5 feet below the surface in the primary cores will be analyzed immediately. Deeper intervals will be archived, with the sample intervals analyzed pending results of the initial analysis, as described in Section 3.1.1. The locations and intervals to be analyzed in the step-out cores will depend on which samples in the corresponding primary core or grab locations (if any) exceed RALs (see Section 3.1.3.5).

3.1.3.5 Primary and Step-out Pairing and Overall SMA 2 Dataset

The following summary presents the primary and step-out location pairings for preliminary SMA 2. If one or more RAL exceedances are found in the primary sample locations listed, then the samples from the corresponding step-out locations will be analyzed following the protocol outlined in Section 3.1.1:

- Primary locations PD13, PD14, or PD15 – Step-out location PD09
- Primary locations PD17 or PD18 – Step-out location PD10 and PD24
- Primary location PD19 – Step-out location PD10
- Primary location PD23 or PD28 – Step-out locations PD24, PD25, and PD27
- Primary location PD21 – Step-out locations PD11 and PD22

The combined data set, including Pre-RD samples (S157, SC-S157, S158, S159, S161, S162) and additional samples analyzed during this PDI (including primary locations PD12 through PD21, PD23, PD26, and PD28 and step-out samples, if analyzed) will be used to evaluate this area (Table 3-1, Figure 3-2).

3.1.4 Preliminary SMA 3

This preliminary SMA was identified based on FS data, but Pre-RD subsurface sediment data also support the possible presence of an SMA in the area, as described in Section 2.3.3. However, current conditions in the central area of the slip near locations C532, G694, and SD117 are not well defined and additional surface and subsurface sediment samples are needed to refine SMA boundaries and inform RD.

The proposed sampling approach is shown on Figure 3-3. This area is in the FMD area (Figure 1-2), with a portion in the intermediate zone. The area has not been dredged recently, and given current operational depths and bathymetry, maintenance dredging is not anticipated in the near term. Pre-RD samples SC-163/S163 and B241 were incorporated into the sample design. One primary surface grab (PD34) will be collected under the Phillips 66 dock in this area and four primary cores with surface grabs (PD31, PD32, PD35, PD36) are located within or immediately adjacent to the preliminary SMA and are centered

on a 150-foot grid. Cores will be collected to 15 feet bml (or refusal) due to thicker sediment deposits in this area so that depth of contamination can be delineated, if needed. The six proposed step-out cores with surface grabs (PD29, PD30, PD33, PD37, PD38, PD39) are also generally placed using the 150-foot grid around the primary locations. Four of these step-out locations (PD33, PD37, PD38, PD39) are in the zone closer to the shoreline will be collected to a depth of 15 feet. The two step-out locations (PD29 and PD30) between Preliminary SMA 3 and Preliminary SMA 2 will have cores collected to a depth of 10 feet or refusal. These two step-out locations will also inform conditions near preliminary SMA 2, if needed.

If one or more RAL exceedances are found in the primary sample locations in this area, then the immediately adjacent step-out location or locations will be analyzed, as described below and in Section 3.1.1:

- Primary location PD31 – Step-out location PD29
- Primary location PD32 – Step-out locations PD30 and PD33
- Primary location PD36 – Step-out locations PD37 and PD39
- Primary location PD35 – Step-out location PD38

The surface grab and top four core intervals at the primary locations (i.e., the top 5 feet) will be analyzed immediately. Deeper intervals will be archived, with the sample intervals analyzed pending results of the initial analysis, as described in Section 3.1.1. Samples from step-out locations will be held frozen and released for analysis, as described in Section 3.1.1. The under-dock surface sample will be analyzed immediately.

The combined data set, including FS (C532), Pre-RD (S163, SC-S163, B241) and additional samples analyzed during this PDI (including primary locations PD34, PD31, PD32, PD35, PD36 and step-out samples, if analyzed) will be used to evaluate this preliminary SMA (Table 3-1, Figure 3-3).

3.1.5 Preliminary SMA 4 and SMA 5

These preliminary SMAs were identified based on FS data and, based on dredging history, may no longer be representative of current conditions in some areas, as summarized in Section 2.3.4. Current conditions at Preliminary SMA 4 are not well defined and there are limited bounding samples with surface or subsurface data in the vicinity of the RAL exceedances at G394 and DMMU-3-B. Additional surface samples and subsurface samples are needed to refine Preliminary SMA 4 and inform RD. Preliminary SMA 5 is located fully within the 2011 dredging prism and additional data are needed to verify the presence or absence of the SMA, and, if present, to delineate its extent to inform RD.

The proposed sampling approach is shown on Figure 3-4. A portion of Preliminary SMA 4 and all of Preliminary SMA 5 are within the 2011 dredge prisms (FMD area); the remainder of preliminary SMA 4 is in the intermediate zone.

To address Preliminary SMA 4, one primary under dock grab (PD43) and seven primary cores with surface grabs (PD42, PD44, PD47, PD48, PD50, PD52, and PD53) are located within the preliminary SMA. One additional primary core with surface grab is located near the Phillips 66 dock (PD46). Sampling locations are positioned on a nominal 150-foot grid. Two primary cores and surface grabs (PD50, PD52) will be positioned near the existing FS samples G394 and DMMU-3-B. Two primary cores and grabs will be collected at a similar elevation on the downstream side of the Chevron dock (PD47, PD48) and two primary grab/cores will be collected near the edge of the preliminary SMA within the dredged area (PD42, PD44). One step-out core with surface grab is located riverward, between the docks (PD45). One primary core with surface grab is located adjacent to the Phillips 66 dock, near the shore (PD53). A step-out core with surface grab is located at the downstream end of the preliminary SMA near the shore to confirm the extent in that direction (PD40). Cores will be extended to 10 or 15 feet bml, or refusal, for possible

delineation of the depth of contamination, if needed. Two step-out surface grabs (PD49, PD51) will also be located in the near shore area (a steep intermediate zone) to potentially help bound the previous RAL exceedances at samples G394, and DMMU-3-B further up the slope, if needed.

Refusal is anticipated in the cores placed parallel to the shoreline due to the heavy armoring present in that area. In areas outside of the 2011 dredge prisms, the surface grab and top four core intervals at the primary locations (i.e., the top 5 feet) will be analyzed immediately. At the locations within the 2011 dredge prism, samples from the surface and the four intervals including and below the operational elevation will be analyzed immediately, as described in Section 3.1.1. The remaining intervals will be held frozen and released for analysis as described in Section 3.1.1. Step-out samples will be held frozen and released for analysis as described in Section 3.1.1.

One primary core with surface grab (PD41) is located within the area identified as preliminary SMA 5 to evaluate current conditions. The target location is adjacent to the location of sample AN-CTPD-01, which was removed during dredging in 2011. Samples from the surface and the four intervals including and below the operational elevation will be analyzed immediately, as described in Section 3.1.1. The remaining intervals will be held frozen and released for analysis as described in Section 3.1.1.

The following summary presents the primary and step-out location pairings for this area. If one or more RAL exceedances are found in the primary sample locations listed, the samples from the corresponding step-out locations will be analyzed following the protocol outlined in Section 3.1.1:

- Primary location PD41 or PD47 – Step-out location PD40
- Primary location PD44 or PD46 – Step-out location PD45
- Primary location PD52 – Step-out locations PD51
- Primary location PD50 – Step-out locations PD49 and PD51

The combined data set, including Pre-RD (S160, B239) and additional samples analyzed during this PDI (including primary locations PD41, PD42, PD43, PD44, PD46, PD47, PD48, PD50, PD52, and PD53, and step-out samples, if analyzed) will be used to evaluate the two preliminary SMAs (Table 3-1, Figure 3-4).

3.2 River Bank Sampling Design

As described in Section 2.5, the river bank between the Chevron and Phillips 66 slips adjacent to preliminary SMA 4 will be characterized (Figure 3-5). Concentrations of Table 17 COCs (except chlorobenzene and tributyltin) in surface soil samples will be measured to help evaluate recontamination potential from the river bank soil if there are areas where surface soil does not exceed RAL or PTW thresholds. If surface soil exceeds Table 21 RAL or PTW thresholds, the archived subsurface soil from those respective locations will be analyzed for the Table 21 COCs to characterize the potential vertical extent of the river bank contamination. If COC concentrations in soil samples do not exceed the Table 21 RAL or PTW thresholds, the data will be compared to CULs and Oregon background soil concentrations to help evaluate recontamination potential from the river bank soil.

The primary objective of this sampling is to characterize the potential areal extent of contaminant concentrations exceeding the RALs or PTW thresholds along this approximate 500-foot portion of the WCPA river bank. Data are needed from the surface and subsurface soil/sediment between the mean low water (MLW) and top of bank elevations adjacent to preliminary SMA 4 to a depth sufficient to support RD (5 feet). The sampling design includes two transects along the bank following the MLW and OHW elevations. River bank surface soil samples will be collected approximately every 100 feet along each transect, as shown on Figure 3-5. This spacing is intended to provide adequate coverage for contaminant delineation along the different inundation zones. Due to the presence of large riprap and steep slopes, sample locations presented are preliminary and will be adjusted based on field conditions encountered. A

50-foot long area in either direction, along the same contour elevation, will be evaluated during the field event to identify the area that will most likely allow for sample collection.

River bank surface soil samples will be analyzed for the ROD Table 17 and Table 21 RAL compounds, except for tributyltin and chlorobenzene. Chlorobenzene is a constituent of interest only in groundwater at the Arkema facility (EPA 2016a). The criteria for excluding chlorobenzene was discussed in Section 3.1. Additionally, the analytical testing suite for surface soil samples does not include tributyltin as it is not a constituent of concern within the WCPA based on the conceptual site model and existing sediment sampling results. Tributyltin was introduced as anti-fouling additive to paint in the late 1960s and postdates creation of Willbridge Cove between 1957 and 1966. RI and Pre-RD analytical data from the WCPA indicate that concentrations of tributyltin in sediment are well below the 3,080 µg/kg CUL. An evaluation of the available tributyltin results within the FS and Pre-RD data sets (Appendix A) indicates that of the nine Pre-RD surface sediment samples collected within the cove and analyzed for tributyltin, the constituent was detected in seven of the nine samples, at concentrations of 19 µg/kg or less. Within the FS data set, tributyltin was analyzed in 51 samples and detected in 24; the detected values were 83 µg /kg or less. Additionally, the Portland Harbor Remedial Investigation (RI) Report (EPA 2016) documented that complete or likely complete pathways for tributyltin were only identified at Cascade General and Gunderson, which are not part of the WCPA.

Soil samples will be collected at 1-foot depth intervals to refusal or a maximum depth of 5 feet bgs. Samples from the 0- to 1-foot-bgs interval will be submitted for chemical analysis of ROD Table 17 and Table 21 COCs (excluding chlorobenzene and tributyltin). Subsurface samples will be collected and archived for later analysis of the Table 21 COCs to support potential RD decisions (for example, excavation depth and cap design), if needed.

Soil properties such as grain size distribution, geotechnical properties, and river bank geometry (e.g., bank height, MLW and OHW elevations, and bank angle) are needed to evaluate the bank conditions for stability analysis if there is a need to assess recontamination potential in the future. Based upon the analytical results, select soil samples may be analyzed for grain size to support a more detailed river bank erodibility evaluation if one is needed to assist RD. The proposed sampling approach to fill the river bank data gaps are presented in Table 3-1 and the analytical schedule and archiving scheme for the river bank soil is presented in Table 3-3.

4. Sampling and Analysis

This section provides an overview of the procedures to be used for river bank and sediment sampling efforts. The FSP, field sampling forms, and relevant field standard operating procedures (SOPs) are included in Appendix B. The QAPP for the laboratory analytical data is included in Appendix C. General soil and sediment sample collection procedures are described below. Detailed collection procedures, including pertinent specifications for location positioning, required field data needs, sample containerization and preservation, and sample nomenclature are provided in the FSP and associated SOPs (Appendix B) and the QAPP (Appendix C).

4.1 Sediment Sampling and Analysis for SMA Refinement

Surface sediment samples (0 to 30 cm or 0 to 1 foot) will be collected as grab samples using a Ponar grab, Eckman dredge, power grab, or similar sampling device from areas that are not under dock structures. The surface samples will be collected as three-point composites.

Under-dock surface sediment samples (0 to 30 cm or 0 to 1 foot) will also be collected as three-point composites around the target sample location. The subsamples will be obtained by divers using hand tools (shovels, trowels, spoons) and the composites will be created on-shore or on the boat deck from the diver-recovered subsamples.

Sediment core samples will be collected using vibracores by a qualified subcontractor. The sampling team will maneuver to safely get as close as practicable to the proposed locations. If structures or barges are present, the as-sampled location may be offset as long as it remains within the target grid cell(s). Where possible, the target sampling location may be photographed with an underwater camera and probed with a thin metal rod to determine whether sediment is present and estimate the thickness. The photographs and probing can also help to identify debris that may impede core collection. If debris, riprap, or other submerged obstructions are present at a target location, the location will be moved to the nearest accessible location as long as it remains within the target grid cell. The target core length (up to 15 ft bml) in each area is defined in Section 3. The cores will be sectioned, processed, and sampled in 1-foot (30-cm) increments. The samples will be analyzed and/or archived based on the analytical schedule presented in Table 3-2.

The FSP (Appendix B) presents acceptance criteria for the sediment cores and grab samples and provides additional details about the processing and subsampling procedures, as well as procedures to maintain positioning.

4.2 River Bank Sampling

Soil borings will be advanced using hand auger techniques to a vertical depth of 5 feet bgs. The top layer of vegetation and gravel (if any) will be temporarily removed in an area at least 6 inches in diameter. If riprap or other surface obstructions are present at a target location, the location will be moved to the nearest accessible location along the target elevation contour.

Bank soil samples will be collected continuously on 1-foot (30 cm) sampling intervals to a depth of 5 feet. Soil samples will be collected using decontaminated (or single-use) hand tools, including shovels, trowels, spoons, and hand augers.

4.3 Analytical Requirements

Tables 3-2 and 3-3 and Sections 3.1 and 3.2 describe the analytical approach for the PDI sediment and river bank soil samples. The analysis of soil and sediment samples may include the following chemicals and associated methods:

- ROD Table 17/21 COCs (except chlorobenzene and tributyltin):
  - 16 Priority Pollutant PAHs and 2-methylnaphthalene (EPA Method 8270C or D SIM)
  - PCB congeners (EPA Method E1668; bank surface soils) or PCB Aroclors (EPA Method 8082; sediments and subsurface bank soils)
  - Dioxin/furan congeners (EPA Method E1613)
  - Pesticides[4] by E1699 or E1699M
  - Metals, including mercury by SW6010B or C and SW7471A or B
  - TPH-DRO by NWTPH-Dx
  - Bis(2-ethylhexyl)phthalate (BEHP) by low level SW8270C or D-SIM
- Physical Parameters:
  - Grain size (ASTM International Standard D7928 and D6913)
  - Total Organic Carbon (EPA Method SW9060)

4.4 Quality Assurance and Quality Control

Field quality assurance and quality control (QA/QC) sample requirements reflect the specific media collected and analyses required. The QA/QC requirements and frequencies are generally as follows:

- **Field duplicates:** 1 per 20 field samples (5 percent) on primary sample locations
- **Matrix spike/matrix spike duplicate:** 1 per 20 field samples (5 percent) on primary sample locations
- **Equipment blanks**: Equipment blanks will be collected for reusable sampling equipment for each matrix sampled and will be collected at a rate of one per week per matrix per equipment; equipment blanks will not be collected for single-use or dedicated equipment
- **Temperature blanks**: 1 per cooler

The FSP (Appendix B) and QAPP (Appendix C) provide instruction on how the field QA/QC samples are to be prepared.

4.5 Health and Safety

All site fieldwork will be performed in accordance with the Jacobs Engineering Group Inc. (Jacobs) Health and Safety corporate standards. The HSP (Appendix D) will be reviewed annually and updated as needed to address new site conditions or tasks. The HSP contains current guidance for working safely during the time of COVID-19 and will be updated to include a dive safety plan and to reflect any changes that occur prior to the commencement of fieldwork. The HSP also includes an Emergency Response Plan for the WCPA.

[4] Pesticides to be reported will include the six DDT isomers, Aldrin, alpha-Chlordane, cis-Nonachlor, Dieldrin, gamma-BHC (Lindane), gamma-Chlordane, Oxychlordane, and trans-Nonachlor. Method 1699M will be used for the six DDT isomers in sediments.

5. Data Management

This section provides an overview of the field and analytical data management procedures. The FSP (Appendix B) and QAPP (Appendix C) provide additional instruction for the project data management requirements.

5.1 Field Documentation

All field activities will be recorded in a field logbook, using activity-specific forms, or using electronic data capture. The FSP (Appendix B) and the associated SOPs provide additional detail and direction related to field documentation requirements.

5.2 Data Management and Validation

Laboratory analytical data acquired during this investigation will be reviewed and validated by the project chemist to assure the data quality criteria set forth in the QAPP (Appendix C) are met and that the data are usable for their intended purpose.

5.3 Data Treatment

Data handling, such as the summations of totals and handling of field splits or field replicates will be managed in accordance with current EPA requirements and/or agency-accepted PHSS precedent. The locations of the surface sediment composite samples will be represented by the centroid of the three individual grabs comprising the composite sample.

6. Data Evaluation and Reporting

The analytical results from the PDI will be used to evaluate current conditions and supplement or supersede existing FS and Pre-RD sediment data and together, these data will support identification and refinement of SMA boundaries within the WCPA. These evaluations will be based on comparison of sediment analytical results to the RALs and PTW thresholds in accordance with the RD Guidelines and Considerations (EPA 2020c). The results of the SMA identification and refinement evaluations, including sample replacement and stability evaluation outcomes and river bank soil results, will be documented in the PDI Evaluation Report.

The PDI Evaluation Report will include:

- Summary of investigations performed
- Photographs documenting work conducted
- Summary of validated data
- Laboratory data reports and data validation reports
- Figures summarizing investigation results
- Narrative interpretation of data and results
- Recommendations for replacement of historical data from locations G407, AN-CTPD-03, and AN-CTPD-06 (based on results of a minimum of three more recent samples for each location)
- Results of physical and chemical stability analysis of areas with subsurface RAL exceedances
- Conclusions and recommendations on whether the data are sufficient to complete the Basis of Design Report

7. Schedule

Figure 7-1 provides a preliminary schedule. The start date for fieldwork will be dependent upon agency concurrence with this WP, subcontractor availability, and favorable weather windows. The following assumptions were used in developing the PDI schedule:

- Contracting and planning includes obtaining subcontractor bids and contracts for laboratories, marine services, and other subcontractors, and obtaining access agreements, permits, and/or in-water work variances (as needed).
- Fieldwork is anticipated to take approximately 5 weeks for the sediment sampling and 1 week for the river bank sampling. River bank sampling will be conducted in fall 2021 during low river stage.
- Laboratory analysis will be on an approximate 4-week turnaround time because the PCB and dioxins/furan analyses have the potential for matrix interferences and the need for re-extractions or re-analyses.
- Data validation is expected to take 4 weeks from the receipt of electronic data sets.
- The durations for the laboratory analysis and data validation are extended to account for the analysis of the step-out locations.
- The schedule assumes EPA review of draft documents is approximately 12 weeks (approximately 90 calendar days) and that revisions are due 45 calendar days after EPA review comments are received per the Willbridge Cove Project ASAOC.

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Tables

Table 1-1. Authorized or Operational Depths for WCPA Berths

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Berth	Authorized or Operational Depth	
	CRD (ft)	NAVD88 (ft)
KMLT – Downstream of Dock	-34	-28.7
KMLT – Upstream of Dock	-38.5	-33.2
Chevron – Both sides of Dock	-40	-34.7
Phillips 66 – Downstream of Dock	-25	-19.7
Phillips 66 – Upstream of Dock	-20	-14.7
McCall – Length of Dock	-38	-32.7

Notes:
Chevron - Chevron U.S.A., Inc.
CRD – Columbia River Datum
KMLT – Kinder Morgan Liquid Terminals, LLC.
ft – feet
McCall – McCall Oil and Chemical Corporation
NAVD88 – North American Vertical Datum of 1988
Phillips – Phillips 66 Company
WCPA – Willbridge Cove Project Area

Table 1-2. Summary of DQO Process – Problem Statement 1

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Steps	Description
Step 1 Problem Statement	The preliminary SMAs need to be sufficiently delineated and refined to support technology selection and remedial design.
Step 2 Identify the Decision/Principal Study Questions	Do additional data collected within and adjacent to the preliminary SMAs combined with existing, representative data indicate that an SMA is present and, if so, what is its footprint and vertical extent contamination?
Step 3 Identify Inputs to the Study	• Current bathymetric data • Preliminary SMA boundaries • Anticipated remedial technology for area • Authorized or operational dredge elevations for FMD areas • Locations and depths of new and existing samples • Concentrations of Table 21 COCs (except chlorobenzene) in new and existing surface and subsurface sediment samples • Sediment grain size • Dredging permits and records • PHSS site-wide RALs and PTW thresholds • Expected slopes and substrate materials in sampling areas • Construction information for docks and overwater structures
Step 4 Define Boundaries of the Study	**Spatial Boundaries – Areal Extent:** • Surface and subsurface sediment within and adjacent to preliminary SMA boundaries **Spatial Boundaries – Vertical Extent:** • FMD Areas – cores from surface to operational elevation or authorized dredge elevation plus buffer (up to 5 feet) • Beneath Structures – surface sediment only • Intermediate Region – cores from surface to maximum expected remedial dredge depth plus buffer (up to 5 feet) to characterize material that would potentially be capped. • Shallow Region – cores from surface to maximum expected remedial dredge depth plus buffer (up to 5 feet) to characterize material that would potentially be capped.

Steps	Description
Step 5 Develop a Decision Rule	• Confirmation of SMA - If Table 21 COC concentrations in surface sediment samples from the combined data set within and adjacent to the preliminary SMA boundary are below site-wide RALs and PTW thresholds, then no SMA is present. Otherwise, an SMA is present and will be delineated for remedial design purposes. • Determine areal extent (footprint) of SMA – if Table 21 COC concentrations in surface sediment samples collected around the RAL/PTW threshold exceedance location(s) are below site-wide RALs/PTW thresholds, the areal extent of the SMA has been delineated. • Characterize the vertical extent of contamination to depth required for RD – if COC concentrations in two consecutive sample intervals beneath the RAL/PTW exceedance location(s) are below all applicable RALs and PTW thresholds, then the vertical extent of contamination has been delineated. If the expected remedial technology in an area includes capping, full delineation of the depth of contamination is not necessary, but characterization of subsurface sediment will be required to support cap design evaluations or to demonstrate stability of the buried sediment. Delineation of the depth of contamination is needed in instances where dredging alone is the anticipated remedial alternative. • Subsurface exceedances of RALs and PTW thresholds in areas without surface exceedances will be evaluated using the overall sediment stability protocol (Table 1-3 in Revised PDI Work Plan).
Step 6 Specify Tolerable Limits on Decision Errors	New analytical data will meet EPA specifications for precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS) and analytical method requirements.
Step 7 Optimize the Design	The data gaps evaluations documented in Section 2 and the sampling designs presented in Section 3 of this work plan consider existing FS and Pre-RD data relative to the following design elements to identify the additional surface sediment and core samples to be collected during the PDI: • Surface and subsurface sample locations are generally spaced on a nominal 150- by 150-foot grid, where applicable. In some areas, closer spacing is proposed to address data gaps. Step-out samples will also be collected and held frozen and will be analyzed if needed to bound RAL or PTW exceedances in primary locations (see Section 3). • Core depths and sampling intervals will be determined based on anticipated future uses and operational depths and the anticipated technology to be applied (See Step 4). • Surface sediment samples (0 to 30 cm or 0 to 1 foot) will be collected as grab samples using a Ponar grab, Eckman dredge, power grab, or similar sampling device from areas that are not under dock structures. The surface samples will be collected as three-point composites. • Under-dock surface sediment samples (0 to 30 cm or 0 to 1 foot) will also be collected as three-point composites around the target sample location. The subsamples will be obtained by divers using hand tools (shovels, trowels, spoons) and the composites will be created on-shore or on the boat deck from the diver-recovered subsamples. • Subsurface sediment will be collected with a Vibracore or similar method that allows penetration to the required depth, collects sediment samples at sampling intervals of at least 30 centimeters (1 foot), recovers 80% of the sampling interval, and establishes the vertical extent of contamination. • The PDI samples will be analyzed for Table 21 COCs (except chlorobenzene). Grain size distribution and TOC will also be analyzed in selected samples.

Notes:
cm - centimeter
COC – contaminant of concern
DQO – data quality objective
EPA – U.S. Environmental Protection Agency
FMD – future maintenance dredge
PARCCS – precision, accuracy, representativeness, comparability, completeness, and sensitivity
PDI – Pre-Design Investigation
PHSS – Portland Harbor Superfund Site
PTW – principal threat waste
RAL – remedial action level
RD – remedial design
SMA – sediment management area
TOC – total organic carbon

Table 1-3. Summary of DQO Process – Problem Statement 2

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Steps	Description
Step 1 Problem Statement	The physical and chemical stability of contaminated sediments at depth need to be sufficiently assessed to evaluate whether subsurface contamination has the potential to be exposed or recontaminate cleaner surface sediment.
Step 2 Identify the Decision/Principal Study Questions	Does buried contamination in the WCPA have the potential to be exposed or to recontaminate cleaner surface sediment through advection and dispersion in sediment porewater?
Step 3 Identify Inputs to the Study	• Bathymetric differencing information (historical and current bathymetric data) • Sediment slopes and armoring materials • Sediment grain size and geotechnical properties (porosity, bulk density) • Porewater upwelling velocity (range of site-specific values from Appendix C2 of the Portland Harbor RI [EPA 2016a] will be used) • Existing data in the Portland Harbor FS (EPA 2016b) will be used to for estimates of natural and anthropogenic forces, bottom shear stresses, and critical shear stresses. • Authorized or operational dredge elevations for FMD areas • Locations and depths of new and existing samples • Concentrations of Table 21 COCs (except chlorobenzene) and TOC in new and existing subsurface sediment samples • PHSS CULs, RALs, and PTW thresholds • Literature-based organic carbon coefficients (K_{oc}) for Table 21 COCs detected in subsurface sediment
Step 4 Define Boundaries of the Study	Subsurface sediment with Table 21 COC concentrations above RALs or PTW thresholds in locations within and outside of preliminary SMA boundaries and elsewhere in the WCPA.
Step 5 Develop a Decision Rule/Analytical Approach	The physical stability of contaminated subsurface sediment in areas of possible natural and anthropogenic erosion will be evaluated using multiple lines of evidence that consider bathymetry, slope stability, sedimentation rates, grain size, geotechnical information, and the comparison of critical and bed shear stresses. The chemical stability of contaminated subsurface sediment will be evaluated in the CapSim Model (Lampert and Reible 2009) using Table 21 COC and TOC concentrations in subsurface sediment, porosity and bulk density information, K_{oc} values for detected COCs, and the estimated porewater upwelling velocity.
Step 6 Specify Tolerable Limits on Decision Errors	New analytical data will meet EPA specifications for PARCCS and analytical method requirements.

Steps	Description
Step 7 Optimize the Design	The same grain size and chemical concentration data used to characterize and refine SMAs under DQO 1 will be used in the subsurface sediment stability evaluation. This includes the combined FS, Pre-RD, and PDI data sets, along with existing bathymetric surveys.

Notes:
COC – contaminant of concern
CUL – cleanup level
DQO – data quality objective
EPA – U.S. Environmental Protection Agency
FMD – future maintenance dredge
FS – Feasibility Study
PARCCS – precision, accuracy, representativeness, comparability, completeness, and sensitivity
PDI – Pre-Design Investigation
PHSS – Portland Harbor Superfund Site
PTW – principal threat waste
RAL – remedial action level
Pre-RD – Pre-Remedial Design Investigation
SMA – sediment management area
TOC – total organic carbon
WCPA – Willbridge Cove Project Area

Table 1-4. Summary of DQO Process – Problem Statement 3

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Steps	Description
Step 1 Problem Statement	The possible presence of river bank contamination contiguous with in-water SMAs needs to be evaluated and refined to support technology selection and remedial design.
Step 2 Identify the Decision/Principal Study Questions	Is river bank contamination contiguous with sediment contamination at SMAs, and if so, what is the areal and vertical extent of river bank contamination?
Step 3 Identify Inputs to the Study	• Current bathymetric data • Preliminary SMA boundaries • Anticipated remedial technology for SMAs and contiguous river banks • Locations and depths of new and existing sediment and river bank soil samples • Concentrations of Table 21 COCs (except chlorobenzene) in new and existing surface and subsurface soil and sediment samples • Concentrations of Table 17 COCs (except chlorobenzene and tributyltin) in new and existing surface soil samples (these data will be collected to help evaluate recontamination potential from the river bank soils if there are areas where COC concentrations in surface soil do not exceed RAL or PTW thresholds). • Soil and sediment grain size and geotechnical properties • PHSS site-wide CULs and Oregon background metals concentrations for Portland Basin (DEQ, 2013) • PHSS RALs and PTW thresholds for Table 21 COCs • Slopes and armoring materials along river bank • Height of bank, mean low and ordinary high water elevations, and bank angle • Locations and construction information for docks, buried utilities, remediation systems, and other over- and on-bank structures
Step 4 Define Boundaries of the Study	**Spatial Boundaries – Areal Extent:** • Surface and subsurface soil between mean low water and top of river banks that are contiguous with SMAs. **Spatial Boundaries – Vertical Extent:** • Cores from surface to maximum expected removal depth to characterize material that would potentially be capped. Based on the bank slopes and existing infrastructure in this area, it is expected that any removal would be less than 5 feet and residual material would need to be capped.

Steps	Description
Step 5 Develop a Decision Rule	Characterize river bank surface soil relative to site-wide CULs (ROD Table 17) and RALs or PTW thresholds (ROD Table 21), as well as background metals concentrations, as applicable. • If COC concentrations in surface soils exceed Table 21 RAL or PTW thresholds, the archived subsurface soil samples from those respective locations will be analyzed for the Table 21 COCs to characterize the vertical extent of the river bank contamination. If Table 21 COC concentrations in two consecutive soil sample intervals beneath the RAL/PTW exceedance location(s) are below RALs and PTW thresholds, the vertical extent of the river bank contamination has been delineated. However, the expected remedial technology for river banks is excavation to a sufficient depth to install a cap (up to 5 feet or as deep as feasible given local conditions) and capping, so full delineation of the depth of contamination is not necessary.
Step 6 Specify Tolerable Limits on Decision Errors	New analytical data will meet EPA specifications for PARCCs and analytical method requirements.
Step 7 Optimize the Design	The data gaps evaluations documented in Section 2 and the sampling design in Section 3 of this work plan consider existing sediment and river bank soil data relative to the following design elements to identify surface soil and core samples to be collected during the PDI: • Soil sampling locations are generally spaced at 100-foot intervals on two transects (one at mean low water, the other at ordinary high water) paralleling the shoreline adjacent to the SMA. The presence of heavy armoring (riprap and large gravel) on steep slopes, as well as stormwater and source control infrastructure on the upper portion of the river bank, will influence whether sampling can be accomplished at all designated locations. Locations may be adjusted by up to 50 feet on either side of the designated location along the same elevation contour, to the extent practicable, so that the appropriate material (i.e., <2 mm diameter material) is sampled. Results for river bank soil samples collected prior to placement of riprap or construction of stormwater and source control infrastructure may also be used to augment the PDI data as appropriate. • Borehole depth and sampling intervals will be determined based on the anticipated technology to be applied (to a depth of 5 feet to allow for capping, if needed). • Bank soil will be collected with a hand auger or similar method that allows penetration to the required depth, collection of sediment samples at sampling intervals of at least 30 centimeters (1 foot), recovery of 80% of the sampling interval, and the establishment of the vertical extent of contamination sufficient to support the remedial design. • Surface soil samples will be analyzed immediately for Table 17/21 COCs (except chlorobenzene and tributyltin) using methods with detection limits that will allow for comparison to sitewide CULs, metals background, RALs, and PTW thresholds. • Grain size distribution will also be analyzed in selected samples.

Notes:
1. Although CULs are not decision criteria for this DQO, analytical data with detection limits consistent with CULs may be needed for a future recontamination potential evaluation if COCs in soil are below the RALs.
COC – contaminant of concern
CUL – cleanup level
DQO – data quality objective
EPA – U.S. Environmental Protection Agency
mm - millimeter
PARCCS – precision, accuracy, representativeness, comparability, completeness, and sensitivity
PDI – Pre-Design Investigation
PHSS – Portland Harbor Superfund Site
PTW – principal threat waste
RAL – remedial action level
RD – remedial design
SMA – sediment management area

Table 2-1. FS Results for Samples Collected Within WCPA - Focused COCs

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Location ID	Sample Name	Sample Type	Sample Date	Dredge Area	Depth Class	Start Depth	End Depth	Depth Units	X	Y	Elevation (NAVD88) ft NAVD88	1,2,3,7,8-PeCDD ug/kg		2,3,4,7,8-PeCDF ug/kg		2,3,7,8-TCDD ug/kg		DDx - 6 Isomers (Calculated U = 1/2) ug/kg		Total PAH (Calculated U = 1/2) ug/kg		Total PCB Aroclors (Calculated U = 1/2) ug/kg		Total PCB Congener (Calculated U = 1/2) ug/kg	
											Sediment/Bank Soil CUL	**0.0002**		**0.0003**		**0.0002**		**6.1**		**23,000**		**9**		**9**	
											Sitewide RAL	**0.0008**		**0.2**		**0.0006**		**160**		**30,000**		**75**		**75**	
											PTW Threshold	**0.01**		**0.2**		**0.01**		**7,050**		**NA**		**200**		**200**	
Surface Sediment Samples within WCPA																									
WLCCWI08ANCTPD03	WLCCWI08ANCTPD03	N	5/29/2009	Riverward end of Chevron Slip (not dredged in 2011)	Surface Sediment	0	2	cm	7629152	700952	-39	--		--		--		--		--		4	UT	--	
WLCCWI08ANCTPD06	WLCCWI08ANCTPD06	N	5/29/2009	Riverward end of Chevron Slip (not dredged in 2011)	Surface Sediment	0	2	cm	7629255	700767	-38	--		--		--		--		--		3.9	UT	--	
WLCCWI08DMMU1A	WLCCWI08DMMU1A	N	5/29/2009	2011 Chevron Dredge Area	Surface Sediment	0	2	cm	7628747	700605	-35	--		--		--		--		--		***19***	***T***	--	
WLCCWI08DMMU1B	WLCCWI08DMMU1B	N	5/29/2009	2011 Chevron Dredge Area	Surface Sediment	0	2	cm	7628834	700677	-33	--		--		--		--		--		***20***	***T***	--	
WLCCWI08DMMU2A	WLCCWI08DMMU2A	N	5/29/2009	2011 Chevron Dredge Area	Surface Sediment	0	2	cm	7628879	700461	-33	--		--		--		--		--		4	UT	--	
WLCCWI08DMMU2B	WLCCWI08DMMU2B	N	5/29/2009	2011 Chevron Dredge Area	Surface Sediment	0	2	cm	7628979	700542	-33	--		--		--		--		--		3.9	UT	--	
WLCCWI08DMMU2C	WLCCWI08DMMU2C	N	5/29/2009	2011 Chevron Dredge Area	Surface Sediment	0	2	cm	7629098	700580	-33	--		--		--		--		--		***17***	***T***	--	
WLCCWI08DMMU3A	WLCCWI08DMMU3A	N	5/29/2009	2011 KMLT Dredge Area	Surface Sediment	0	2	cm	7628650	700651	-24	--		--		--		--		--		***19***	***T***	--	
WLCCWI08DMMU3B	WLCCWI08DMMU3B	N	5/29/2009		Surface Sediment	0	2	cm	7628779	700408	-18	--		--		--		--		--		***560***	***T***	--	
WLCCWI08DMMU3D	WLCCWI08DMMU3D	N	5/29/2009		Surface Sediment	0	2	cm	7628873	700325	-13	--		--		--		--		--		3.9	UT	--	
WLCCWI08DMMU3E	WLCCWI08DMMU3E	N	5/29/2009		Surface Sediment	0	2	cm	7628981	700402	-24	--		--		--		--		--		***19***	***T***	--	
WLCWTG02ANCTPD01	WLCWTG02ANCTPD01A	N	7/16/2002	2011 Chevron Dredge Area	Surface Sediment	0	2	cm	7628756	700622	-35	--		--		--		***19***	***T***	1,400	T	***120***	***JT***	--	
WLCWTG02ANCTPD02	WLCWTG02ANCTPD02A	N	7/16/2002	2011 Chevron Dredge Area	Surface Sediment	0	2	cm	7628958	700792	-34	--		--		--		***15***	***T***	1,100	T	***110***	***JT***	--	
WLCWTG02ANCTPD03	WLCWTG02ANCTPD03A	N	7/16/2002	Riverward end of Chevron Slip (not dredged in 2011)	Surface Sediment	0	2	cm	7629152	700954	-39	--		--		--		***14***	***AT***	1,700	T	***130***	***JT***	--	
WLCWTG02ANCTPD06	WLCWTG02ANCTPD06A	N	7/16/2002	Riverward end of Chevron Slip (not dredged in 2011)	Surface Sediment	0	2	cm	7629255	700769	-38	--		--		--		***18***	***T***	1,400	T	***130***	***JT***	--	
WLCWTG02ANCTPD07	WLCWTG02ANCTPD07A	N	7/16/2002	2011 Chevron Dredge Area	Surface Sediment	0	2	cm	7629084	700617	-34	--		--		--		***7.3***	***AT***	880	T	***120***	***JT***	--	
WLCWTG02ANCTPD08	WLCWTG02ANCTPD08A	N	7/16/2002	2011 Chevron Dredge Area	Surface Sediment	0	2	cm	7628892	700455	-33	--		--		--		***8.4***	***AT***	1,100	T	***130***	***JT***	--	
LW2-NA3A	NA-3A-0005	N	10/22/2004		Surface Sediment	0	5	cm	7629429	700328	-21	--		--		--		--		--		--		--	
WLCCWI08DMMU1C	WLCCWI08DMMU1C	N	5/29/2009	2011 Chevron Dredge Area	Surface Sediment	0	5	cm	7628936	700759	-33	--		--		--		--		--		***18***	***T***	--	
LW2-GBT020	LW2-GBT020	N	12/6/2005		Surface Sediment	0	10	cm	7628495	701060	-13	***0.000494***	***J***	***0.000748***	***J***	***0.000226***	***J***	***150***	***T***	360	JT	--		***19***	***JT***
LW2-GBT021	LW2-GBT021	N	12/13/2005		Surface Sediment	0	10	cm	7629274	700117	-6	0.000053	J	0.00007	J	0.000011	U	***8.4***	***JT***	430	T	--		***26***	***T***
WLCASF97S001	WLCASF97S001W4159	N	6/12/1997		Surface Sediment	0	10	cm	7630379	699598	-9	--		--		--		--		730	T	--		--	
WLCASF97S002	WLCASF97S002W4158	N	6/12/1997		Surface Sediment	0	10	cm	7630047	700105	-32	--		--		--		--		420	T	--		--	
WLCASF97S003	WLCASF97S003W4157	N	6/12/1997		Surface Sediment	0	10	cm	7629493	700297	-17	--		--		--		--		1,100	T	--		--	
WLCASF97S004	WLCASF97S004W4156	N	6/12/1997		Surface Sediment	0	10	cm	7629322	700277	-23	--		--		--		--		1,700	T	--		--	
WLCASF97S005	WLCASF97S005W4155	N	6/12/1997		Surface Sediment	0	10	cm	7629019	700128	-5	--		--		--		--		9,000	T	--		--	
WLCASF97S006	WLCASF97S006W4154	N	6/12/1997		Surface Sediment	0	10	cm	7628791	700365	-2	--		--		--		--		1,000	T	--		--	
WLCASF97S008	WLCASF97S008W4152	N	6/12/1997		Surface Sediment	0	10	cm	7629266	700704	-35	--		--		--		--		800	T	--		--	
WLCASF97S009	WLCASF97S009W4172	N	6/11/1997		Surface Sediment	0	10	cm	7628648	700612	-32	--		--		--		--		1,400	T	--		--	
WLCASF97S010	WLCASF97S010W4171	N	6/11/1997		Surface Sediment	0	10	cm	7629081	700977	-34	--		--		--		--		1,400	T	--		--	
WLCASF97S011	WLCASF97S011W4170	N	6/11/1997		Surface Sediment	0	10	cm	7628514	700853	-19	--		--		--		--		2,800	T	--		--	
WLCASF97S012	WLCASF97S012W4168	N	6/11/1997		Surface Sediment	0	10	cm	7628858	701245	-20	--		--		--		--		680	T	--		--	
WLCASF97S012	WLCASF97S012W4169	FR	6/11/1997		Surface Sediment	0	10	cm	7628858	701245	-20	--		--		--		--		680	T	--		--	
WLCOFJ022201	WLCOFJ02220122010	N	10/21/2002		Surface Sediment	0	10	cm	7629006	700111	-2	--		--		--		2.5	UJT	450	JT	--		--	
WLCOFJ022206	WLCOFJ02220622060	N	10/21/2002		Surface Sediment	0	10	cm	7629040	700177	-19	--		--		--		***10.1***	***JT***	1,600	JT	--		--	
WR-WSI98SD104	WR-WSI98SD1040	N	9/23/1997		Surface Sediment	0	10	cm	7628572	701218	-1	--		--		--		--		600	T	--		--	
WR-WSI98SD105	WR-WSI98SD1050	N	9/23/1997		Surface Sediment	0	10	cm	7628878	701254	-20	--		--		--		--		640	T	--		--	
WR-WSI98SD107	WR-WSI98SD1070	N	9/23/1997		Surface Sediment	0	10	cm	7628533	700849	-19	--		--		--		--		2,000	T	--		--	
WR-WSI98SD110	WR-WSI98SD1100	N	9/23/1997		Surface Sediment	0	10	cm	7629093	700983	-35	--		--		--		--		1,000	T	--		--	
WR-WSI98SD113	WR-WSI98SD1130	N	9/23/1997		Surface Sediment	0	10	cm	7629298	700704	-35	--		--		--		--		940	T	--		--	
WR-WSI98SD114	WR-WSI98SD1140	N	9/23/1997		Surface Sediment	0	10	cm	7629023	700135	-8	--		--		--		--		2,000	NT	--		--	
WR-WSI98SD115	WR-WSI98SD1150	N	9/23/1997		Surface Sediment	0	10	cm	7629434	700515	-24	--		--		--		--		430	T	--		--	
WR-WSI98SD117	WR-WSI98SD1170000CC	N	9/23/1997		Surface Sediment	0	10	cm	7629340	700260	-22	--		--		--		***8.6***	***AJT***	930	T	***96***	***T***	--	
WR-WSI98SD118	WR-WSI98SD1180	N	9/23/1997		Surface Sediment	0	10	cm	7629521	700296	-16	--		--		--		--		2,300	JT	--		--	
WR-WSI98SD123	WR-WSI98SD1230	N	9/24/1997		Surface Sediment	0	10	cm	7630398	699583	-8	--		--		--		--		340	T	--		--	
WLCOFJ022204	WLCOFJ02220422040	N	10/21/2002		Surface Sediment	0	12	cm	7629090	700117	-16	--		--		--		4.01	UJT	1,900	JT	--		--	
WLCOFJ022205	WLCOFJ02220522050	N	10/21/2002		Surface Sediment	0	12	cm	7629070	700151	-18	--		--		--		***16.4***	***JT***	6,400	JT	--		--	
WLRWTF98ATXSD3	WLRWTF98ATXSD3ATXSD3	N	12/18/1998		Surface Sediment	0	12	cm	7628416	701026	-8	--		--		--		***14***	***AT***	--		--		--	
WLRWTF98ATXSD4	WLRWTF98ATXSD4ATXSD4	N	12/18/1998		Surface Sediment	0	12	cm	7628460	700769	5	--		--		--		6.7	AUT	73	T	--		--	
WLRWTF98ATXSD5	WLRWTF98ATXSD5ATXSD5	N	12/18/1998		Surface Sediment	0	12	cm	7628584	700620	5	--		--		--		6.7	AUT	500	UJT	--		--	
WLRWTF98HEVSD1	WLRWTF98HEVSD1HEVSD1	N	12/17/1998		Surface Sediment	0	12	cm	7628832	700321	1	--		--		--		--		500	UJT	--		--	
WLRWTF98HEVSD2	WLRWTF98HEVSD2HEVSD2	N	12/17/1998		Surface Sediment	0	12	cm	7628783	700396	-15	--		--		--		3.35	AUT	500	UJT	--		--	
WLRWTF98HEVSD3	WLRWTF98HEVSD3HEVSD3	N	12/17/1998		Surface Sediment	0	12	cm	7628708	700471	0	--		--		--		3.35	AUT	500	UJT	--		--	
WLRWTF98TOSSD1	WLRWTF98TOSSD1TOSSD1	N	12/17/1998		Surface Sediment	0	12	cm	7628882	700247	6	--		--		--		--		500	UJT	--		--	
WLRWTF98TOSSD2	WLRWTF98TOSSD2TOSSD2	N	12/17/1998		Surface Sediment	0	12	cm	7628955	700135	7	--		--		--		--		125	T	--		--	
WLRWTF98TOSSD3	WLRWTF98TOSSD3TOSSD3	N	12/17/1998		Surface Sediment	0	12	cm	7629084	700168	-12	--		--		--		--		3,400	JT	--		--	
WLRWTF98TOSSD4	WLRWTF98TOSSD4TOSSD4	N	12/17/1998		Surface Sediment	0	12	cm	7629054	699987	13	--		--		--		--		6.7	UT	--		--	
WLRWTF98TOSSD5	WLRWTF98TOSSD5TOSSD5	N	12/18/1998		Surface Sediment	0	12	cm	7629232	699945	9	--		--		--		--		350	JT	--		--	
LW2-B019	LW2-B019	N	7/26/2004		Surface Sediment	0	15	cm	7628948	700051	9	0.000197	J	0.000141	U	0.00004	J	3.54	JNT	24	JT	2.1	UT	--	
LWG0108R002SDS015C00	LWG0108R002SDS015C00	N	10/17/2002		Surface Sediment	0	15	cm	7630259	699666	1	--		--		--		1.3	UT	420	T	***36***	***T***	--	
WLCOFJ022202	WLCOFJ02220222020	N	10/21/2002		Surface Sediment	0	15	cm	7629048	700077	-3	--		--		--		***13.6***	***JT***	2,400	JT	--		--	
WLCOFJ022203	WLCOFJ02220322030	N	10/21/2002		Surface Sediment	0	15	cm	7628994	700157	-4	--		--		--		***19.4***	***JT***	***28,000***	***T***	--		--	
WLCOFJ0222031	WLCOFJ022203122031	FR	10/21/2002		Surface Sediment	0	15	cm	7628991	700159	-4	--		--		--		***23.8***	***JT***	1,760	JT	--		--	
WLCWTG02ANCTPD01	WLCWTG02ANCTPD01B	N	7/16/2002	2011 Chevron Dredge Area	Surface Sediment	0	15	cm	7628756	700622	-35	--		--		--		***27***	***T***	1,900	T	***110***	***JT***	--	

Table 2-1. FS Results for Samples Collected Within WCPA - Focused COCs

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Location ID	Sample Name	Sample Type	Sample Date	Dredge Area	Depth Class	Start Depth	End Depth	Depth Units	X	Y	Elevation (NAVD88) ft NAVD88	1,2,3,7,8-PeCDD ug/kg		2,3,4,7,8-PeCDF ug/kg		2,3,7,8-TCDD ug/kg		DDx - 6 Isomers (Calculated U = 1/2) ug/kg		Total PAH (Calculated U = 1/2) ug/kg		Total PCB Aroclors (Calculated U = 1/2) ug/kg		Total PCB Congener (Calculated U = 1/2) ug/kg	
										Sediment/Bank Soil CUL		**0.0002**		**0.0003**		**0.0002**		**6.1**		**23,000**		**9**		**9**	
										Sitewide RAL		**0.0008**		**0.2**		**0.0006**		**160**		**30,000**		**75**		**75**	
										PTW Threshold		**0.01**		**0.2**		**0.01**		**7,050**		**NA**		**200**		**200**	
WLCWTG02ANCTPD02	WLCWTG02ANCTPD02B	N	7/16/2002	2011 Chevron Dredge Area	Surface Sediment	0	15	cm	7628958	700792	-34	--		--		--		***7.7***	***AT***	690	T	***91***	***JT***	--	
WLCWTG02ANCTPD03	WLCWTG02ANCTPD03B	N	7/16/2002	Riverward end of Chevron Slip (not dredged in 2011)	Surface Sediment	0	15	cm	7629152	700954	-39	--		--		--		***13***	***T***	460	T	***140***	***T***	--	
WLCWTG02ANCTPD06	WLCWTG02ANCTPD06B	N	7/16/2002	Riverward end of Chevron Slip (not dredged in 2011)	Surface Sediment	0	15	cm	7629255	700769	-38	--		--		--		***11***	***AT***	1,100	T	***110***	***JT***	--	
WLCWTG02ANCTPD07	WLCWTG02ANCTPD07B	N	7/16/2002	2011 Chevron Dredge Area	Surface Sediment	0	15	cm	7629084	700617	-34	--		--		--		***10***	***AT***	320	T	***92***	***JT***	--	
WLCWTG02ANCTPD08	WLCWTG02ANCTPD08B	N	7/16/2002	2011 Chevron Dredge Area	Surface Sediment	0	15	cm	7628892	700455	-33	--		--		--		***11***	***T***	1,100	T	***120***	***JT***	--	
LW3-GSP07W	LW3-GSP07W	N	10/15/2007		Surface Sediment	0	19	cm	7628812	700370	-14	0.0000989	U	0.0000859	U	0.000109	U	0.98	JNT	60	JT	--		2	JT
LW2-G389	LW2-G389	N	10/29/2004		Surface Sediment	0	20	cm	7628548	700673	4	--		--		--		1.29	JNT	6.3	JT	2.7	UT	--	
LW3-G694	LW3-G694	N	11/16/2007		Surface Sediment	0	20	cm	7629340	700318	-23	--		--		--		***15***	***JT***	1,300	T	***89***	***JT***	--	
LW2-G399	LW2-G399	N	8/27/2004		Surface Sediment	0	22	cm	7629573	700201	-1	--		--		--		***12***	***JNT***	2,300	T	--		***38***	***JT***
W-09-A-PG	LWG2-PG-W9A	N	11/30/2005		Surface Sediment	0	23	cm	7628899	700250	3	--		--		--		--		12	JT	--		--	
LW2-G394	LW2-G394	N	8/27/2004		Surface Sediment	0	25	cm	7628836	700366	-25	--		--		--		***459***	***JNT***	5,100	T	***790***	***JT***	--	
LW2-G404	LW2-G404	N	8/27/2004		Surface Sediment	0	25	cm	7629091	700096	-9	--		--		--		***11***	***JNT***	1,200	T	***53***	***JT***	--	
LW2-G403	LW2-G403	N	10/22/2004		Surface Sediment	0	26	cm	7629824	700114	-4	0.000024	U	0.00006	J	0.00002	U	1.5	JNT	210	JT	2.35	UT	--	
LW2-G401	LW2-G401	N	10/22/2004		Surface Sediment	0	27	cm	7629036	700154	-10	--		--		--		***11.8***	***JNT***	4,200	T	--		***29***	***JT***
LW2-G413	LW2-G413	N	10/22/2004		Surface Sediment	0	27	cm	7630351	699536	2	--		--		--		0.99	JNT	120	JT	***55.5***	***JT***	--	
LW3-G692-2	LW3-G692-2	FR	12/4/2007		Surface Sediment	0	28	cm	7628730	701048	-20	0.000154	JT	***0.000569***	***JT***	0.000019	UT	***16***	***JT***	650	T	--		***21***	***JT***
LW2-G407	LW2-G407	N	9/9/2004		Surface Sediment	0	29	cm	7630154	699834	-12	--		--		--		4.8	JNT	340	T	***100***	***JT***	--	
LW2-C532	LW2-C532-A	N	10/18/2005		Surface Sediment	0	30	cm	7629257	700289	-21	--		--		--		***14***	***JNT***	800	T	***150***	***T***	--	
LW3-G692-1	LW3-G692-1	N	12/4/2007		Surface Sediment	0	30	cm	7628730	701055	-20	0.0000181	U	0.000185	J	0.0000155	U	***23***	***T***	750	T	--		***26***	***JT***
W-09-C-PG	LWG2-PG-W9C	N	11/30/2005		Surface Sediment	0	30	cm	7628988	700306	-20	--		--		--		--		580	T	--		--	
WLCDRD05PG074	WLCDRD05PG07474	N	5/25/2005		Surface Sediment	0	30	cm	7628952	701290	-24	--		--		--		***18***	***JNT***	400	JT	***46***	***T***	--	
LW2-NA3B	NA-3B-0034	N	10/22/2004		Surface Sediment	0	34	cm	7629433	700322	-20	--		--		--		***13***	***JNT***	1,100	JT	***68.9***	***JT***	--	
Subsurface Sediment Samples within WCPA																									
LW2-C377	LW2-C377-B	N	11/10/2004		Subsurface Sediment	30	153	cm	7628521	701143	-5	0.000045	U	0.000191	J	0.000045	J	***62***	***JNT***	330	T	***39.6***	***JT***	--	
LW2-C377	LW2-C377-C	N	11/10/2004		Subsurface Sediment	153	273	cm	7628521	701143	-5	0.000132	J	***0.001002***	***J***	0.000067	J	***99***	***JNT***	430	T	***280***	***T***	--	
LW2-C377	LW2-C377-D	N	11/10/2004		Subsurface Sediment	273	369	cm	7628521	701143	-5	***0.000489***	***J***	***0.00614***		***0.000296***	***J***	--		2,100	T	--		--	
LW2-C377	LW2-C377-E	N	11/10/2004		Subsurface Sediment	369	482	cm	7628521	701143	-5	***0.001461***		***0.004628***		***0.000297***		***260***	***JNT***	6,200	T	--		***65***	***JT***
LW2-C401	LW2-C401-B	N	11/10/2004		Subsurface Sediment	30	69	cm	7629031	700153	-10	--		--		--		***23***	***JNT***	2,600	T	***51.6***	***JT***	--	
LW2-C401	LW2-C401-C	N	11/10/2004		Subsurface Sediment	69	188	cm	7629031	700153	-10	--		--		--		***9.14***	***ANJT***	2,200	T	***36***	***JT***	--	
LW2-C401	LW2-C401-D	N	11/10/2004		Subsurface Sediment	188	278	cm	7629031	700153	-10	--		--		--		***19***	***JNT***	2,100	T	***300***	***JT***	--	
LW2-C401	LW2-C401-E	N	11/10/2004		Subsurface Sediment	278	367	cm	7629031	700153	-10	--		--		--		***31.8***	***JNT***	1,900	T	--		***890***	***JT***
LW2-C401	LW2-C401-F	N	11/10/2004		Subsurface Sediment	367	504	cm	7629031	700153	-10	--		--		--		0.21	UJT	130	JT	1.6	UT	--	
LW2-C403	LW2-C403-B	N	11/10/2004		Subsurface Sediment	30	153	cm	7629841	700123	-6	0.000042	J	0.000066	U	0.000017	U	0.7	JNT	400	T	***11***	***JT***	--	
LW2-C403	LW2-C403-C	N	11/10/2004		Subsurface Sediment	153	242	cm	7629841	700123	-6	0.000011	U	0.000007	U	0.000016	U	0.45	NJT	21	JT	2.42	UT	--	
LW2-C403	LW2-C403-D	N	11/10/2004		Subsurface Sediment	242	361	cm	7629841	700123	-6	0.00001	U	0.00001	U	0.000026	U	0.066	UJT	7.5	JT	7.9	T	--	
LW2-C403	LW2-C403-F	N	11/10/2004		Subsurface Sediment	483	533	cm	7629841	700123	-6	--		--		--		--		--		--		--	
LW2-C413	LW2-C413-B1	N	10/26/2004		Subsurface Sediment	30	158	cm	7630354	699540	2	--		--		--		***25.7***	***JNT***	390	T	***132***	***JT***	--	
LW2-C413	LW2-C413-C1	N	10/26/2004		Subsurface Sediment	158	299	cm	7630354	699540	2	--		--		--		***32.8***	***JNT***	460	T	***96***	***JT***	--	
LW2-C413-2	LW2-C413-B2	FR	10/26/2004		Subsurface Sediment	30	152	cm	7630359	699540	1	--		--		--		***22.4***	***JNT***	650	T	***310***	***T***	--	
LW2-C413-2	LW2-C413-C2	FR	10/26/2004		Subsurface Sediment	152	274	cm	7630359	699540	1	--		--		--		***26***	***NJT***	290	T	***410***	***JT***	--	
LW2-C413-2	LW2-C413-D2	N	10/26/2004		Subsurface Sediment	274	349	cm	7630359	699540	1	--		--		--		***270***	***JNT***	940	T	***77***	***JT***	--	
LW2-C532	LW2-C532-B	N	10/18/2005		Subsurface Sediment	30	99	cm	7629257	700289	-21	--		--		--		***17***	***JT***	630	T	***180***	***JT***	--	
LW2-C532	LW2-C532-C	N	10/18/2005		Subsurface Sediment	99	198	cm	7629257	700289	-21	--		--		--		***17***	***JNT***	670	T	***120***	***JT***	--	
LW2-C532	LW2-C532-D	N	10/18/2005		Subsurface Sediment	198	293	cm	7629257	700289	-21	--		--		--		--		--		--		***380***	***JT***
LW2-C532	LW2-C532-E	N	10/18/2005		Subsurface Sediment	293	338	cm	7629257	700289	-21	--		--		--		***35***	***JNT***	2,500	T	***49***	***JT***	--	
LW2-C532	LW2-C532-F	N	10/18/2005		Subsurface Sediment	338	372	cm	7629257	700289	-21	--		--		--		0.44	JT	6.3	JT	1.5	UT	--	
LW2-NA3A	NA-3A-0508	N	10/22/2004		Subsurface Sediment	5	8	cm	7629429	700328	-21	--		--		--		--		--		--		--	
LW2-NA3A	NA-3A-0812	N	10/22/2004		Subsurface Sediment	8	12	cm	7629429	700328	-21	--		--		--		--		--		--		--	
LW2-NA3A	NA-3A-1216	N	10/22/2004		Subsurface Sediment	12	16	cm	7629429	700328	-21	--		--		--		--		--		--		--	
LW2-NA3A	NA-3A-1620	N	10/22/2004		Subsurface Sediment	16	20	cm	7629429	700328	-21	--		--		--		--		--		--		--	
LW2-NA3A	NA-3A-2024	N	10/22/2004		Subsurface Sediment	20	24	cm	7629429	700328	-21	--		--		--		--		--		--		--	
LW2-NA3A	NA-3A-2428	N	10/22/2004		Subsurface Sediment	24	28	cm	7629429	700328	-21	--		--		--		--		--		--		--	
LW2-NA3A	NA-3A-2832	N	10/22/2004		Subsurface Sediment	28	32	cm	7629429	700328	-21	--		--		--		--		--		--		--	
LW2-NA3A	NA-3A-3238	N	10/22/2004		Subsurface Sediment	32	38	cm	7629429	700328	-21	--		--		--		--		--		--		--	
LW2-NA3A	NA-3A-3844	N	10/22/2004		Subsurface Sediment	38	44	cm	7629429	700328	-21	--		--		--		--		--		--		--	
LW2-NA3A	NA-3A-4854	N	10/22/2004		Subsurface Sediment	48	54	cm	7629429	700328	-21	--		--		--		--		--		--		--	
LW2-NA3A	NA-3A-5864	N	10/22/2004		Subsurface Sediment	58	64	cm	7629429	700328	-21	--		--		--		--		--		--		--	
LW2-NA3A	NA-3A-6874	N	10/22/2004		Subsurface Sediment	68	74	cm	7629429	700328	-21	--		--		--		--		--		--		--	
LW2-NA3A	NA-3A-7884	N	10/22/2004		Subsurface Sediment	78	84	cm	7629429	700328	-21	--		--		--		--		--		--		--	
LW2-NA3A	NA-3A-8894	N	10/22/2004		Subsurface Sediment	88	94	cm	7629429	700328	-21	--		--		--		--		--		--		--	
LW2-NA3B	NA-3B-3464	N	10/22/2004		Subsurface Sediment	34	64	cm	7629433	700322	-20	--		--		--		***7.2***	***NJT***	730	JT	***26***	***JT***	--	
LW2-NA3B	NA-3B-6494	N	10/22/2004		Subsurface Sediment	64	94	cm	7629433	700322	-20	--		--		--		***17.2***	***JT***	1,300	JT	***74***	***JT***	--	
LW3-C688	LW3-C688-B	N	1/15/2008		Subsurface Sediment	30	114	cm	7628760	701367	-5	***0.000273***	***J***	***0.00035***	***J***	0.000155	U	***15***	***JNT***	250	JT	--		***13***	***JT***
LW3-C688	LW3-C688-C	N	1/15/2008		Subsurface Sediment	114	221	cm	7628760	701367	-5	***0.000211***	***J***	***0.000758***	***J***	0.0000754	U	***30***	***T***	250	JT	--		***71***	***JT***
LW3-C688	LW3-C688-D	N	1/15/2008		Subsurface Sediment	221	281	cm	7628760	701367	-5	***0.000576***	***J***	***0.00208***	***J***	***0.000355***	***J***	***1,300***	***JT***	540	T	--		***230***	***JT***
LW3-C688	LW3-C688-F	N	1/15/2008		Subsurface Sediment	404	457	cm	7628760	701367	-5	***0.00122***	***J***	***0.00572***		***0.000594***		***81***	***T***	2,700	JT	--		***54***	***JT***
LW3-C690	LW3-C690-B	N	1/15/2008		Subsurface Sediment	30	151	cm	7628470	701000	-11	0.0000146	U	***0.000647***	***J***	0.00000801	U	***18***	***JT***	540	T	--		***17***	***T***

Table 2-1. FS Results for Samples Collected Within WCPA - Focused COCs

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Location ID	Sample Name	Sample Type	Sample Date	Dredge Area	Depth Class	Start Depth	End Depth	Depth Units	X	Y	Elevation (NAVD88) ft NAVD88	1,2,3,7,8-PeCDD ug/kg		2,3,4,7,8-PeCDF ug/kg		2,3,7,8-TCDD ug/kg		DDx - 6 Isomers (Calculated U = 1/2) ug/kg		Total PAH (Calculated U = 1/2) ug/kg		Total PCB Aroclors (Calculated U = 1/2) ug/kg		Total PCB Congener (Calculated U = 1/2) ug/kg	
					Sediment/Bank Soil CUL							0.0002		0.0003		0.0002		6.1		23,000		9		9	
					Sitewide RAL							0.0008		0.2		0.0006		160		30,000		75		75	
					PTW Threshold							0.01		0.2		0.01		7,050		NA		200		200	
LW3-C690	LW3-C690-C	N	1/15/2008		Subsurface Sediment	151	274	cm	7628470	701000	-11	*0.000412*	J	*0.00134*	J	0.000174	J	*64*	*JT*	850	T	--		*48*	*T*
LW3-C690	LW3-C690-D	N	1/15/2008		Subsurface Sediment	274	346	cm	7628470	701000	-11	*0.000809*	J	*0.00961*		*0.000441*	J	*83*	*JNT*	2,000	JT	--		*220*	*T*
LW3-C690	LW3-C690-E	N	1/15/2008		Subsurface Sediment	346	471	cm	7628470	701000	-11	*0.00741*		*0.0128*		*0.00117*		*92*	*JNT*	6,600	T	--		*450*	*JT*
LW3-C695	LW3-C695-B	N	12/20/2007		Subsurface Sediment	30	95	cm	7629190	700056	-6	--		--		--		*27*	*JNT*	1,500	T	*73*	*JT*	--	
LW3-C695	LW3-C695-D	N	12/20/2007		Subsurface Sediment	221	343	cm	7629190	700056	-6	--		--		--		*57*	*NJT*	3,000	T	*750*	*JT*	--	
LW3-C695	LW3-C695-E	N	12/20/2007		Subsurface Sediment	343	384	cm	7629190	700056	-6	--		--		--		*62*	*NJT*	2,500	T	*930*	*T*	--	
LW3-C698	LW3-C698-B	N	12/20/2007		Subsurface Sediment	30	152	cm	7630300	699718	-16	--		--		--		*10*	*JNT*	330	T	*56*	*JT*	--	
TOSCO99DMMU1	TOSCO99DMMU1SDO1	N	1/21/1999		Subsurface Sediment	0	304	cm	7629016	700391	-24	--		--		--		*20*	*AJT*	3,300	T	*260*	*JT*	--	
TOSCO99DMMU1	TOSCO99DMMU1SDO3	FR	1/21/1999		Subsurface Sediment	0	304	cm	7629016	700391	-24	--		--		--		*33*	*AJT*	3,200	T	*350*	*JT*	--	
TOSCO99DMMU2	TOSCO99DMMU2SDO4	N	1/21/1999		Subsurface Sediment	0	164	cm	7629278	700566	-29	--		--		--		*52*	*AJT*	1,600	T	*99*	*JT*	--	
WLCCWI08DMMU1A	WLCCWI08DMMU1A4041	N	9/2/2008	2011 Chevron Dredge Area	Subsurface Sediment	91	121	cm	7628747	700605	-35	--		--		--		*13*	*AJT*	1,500	JT	*37.9*	*JT*	--	
WLCCWI08DMMU1A	WLCCWI08DMMU1A4142	N	9/2/2008	2011 Chevron Dredge Area	Subsurface Sediment	121	152	cm	7628747	700605	-35	--		--		--		*10.6*	*AJT*	1,600	JT	12.2	UT	--	
WLCCWI08DMMU1B	WLCCWI08DMMU1B4041	N	9/3/2008	2011 Chevron Dredge Area	Subsurface Sediment	121	152	cm	7628834	700677	-33	--		--		--		*11.3*	*AJT*	1,400	JT	13.5	UT	--	
WLCCWI08DMMU1B	WLCCWI08DMMU1B4142	N	9/3/2008	2011 Chevron Dredge Area	Subsurface Sediment	152	182	cm	7628834	700677	-33	--		--		--		*12.1*	*AJT*	1,740	JT	11.6	UT	--	
WLCCWI08DMMU1C	WLCCWI08DMMU1C4041	N	9/4/2008	2011 Chevron Dredge Area	Subsurface Sediment	91	121	cm	7628936	700759	-33	--		--		--		*13.9*	*AJT*	1,400	JT	*34.3*	*JT*	--	
WLCCWI08DMMU1C	WLCCWI08DMMU1C4142	N	9/4/2008	2011 Chevron Dredge Area	Subsurface Sediment	121	152	cm	7628936	700759	-33	--		--		--		*11.6*	*AJT*	996.4	JT	11.6	UT	--	
WLCCWI08DMMU2A	WLCCWI08DMMU2A4041	N	9/5/2008	2011 Chevron Dredge Area	Subsurface Sediment	152	182	cm	7628879	700461	-33	--		--		--		*7.83*	*AJT*	1,500	JT	13.1	UT	--	
WLCCWI08DMMU2A	WLCCWI08DMMU2A4142	N	9/5/2008	2011 Chevron Dredge Area	Subsurface Sediment	182	213	cm	7628879	700461	-33	--		--		--		*11*	*AT*	1,600	T	12.5	UT	--	
WLCCWI08DMMU2ABC	WLCCWI08DMMU2ABC	N	9/5/2008	2011 Chevron Dredge Area	Subsurface Sediment	60	213	cm	7628986	700528	-33	--		--		--		*8.24*	*AJT*	1,900	JT	14.3	UT	--	
WLCCWI08DMMU2B	WLCCWI08DMMU2B4041	N	9/5/2008	2011 Chevron Dredge Area	Subsurface Sediment	91	121	cm	7628979	700542	-33	--		--		--		*17.8*	*AJT*	2,700	JT	13	UT	--	
WLCCWI08DMMU2B	WLCCWI08DMMU2B4142	N	9/5/2008	2011 Chevron Dredge Area	Subsurface Sediment	121	152	cm	7628979	700542	-33	--		--		--		*18.2*	*AJT*	2,300	JT	11.8	UT	--	
WLCCWI08DMMU2C	WLCCWI08DMMU2C4041	N	9/5/2008	2011 Chevron Dredge Area	Subsurface Sediment	60	91	cm	7629098	700580	-33	--		--		--		*16.1*	*AJT*	2,800	JT	15.5	UT	--	
WLCCWI08DMMU2C	WLCCWI08DMMU2C4142	N	9/5/2008	2011 Chevron Dredge Area	Subsurface Sediment	91	121	cm	7629098	700580	-33	--		--		--		*12.4*	*AJT*	1,840	JT	11.7	UT	--	
WLCCWI08DMMU3A	WLCCWI08DMMU3A13	N	9/8/2008	2011 KMLT Dredge Area	Subsurface Sediment	30	91	cm	7628650	700651	-24	--		--		--		*11.3*	*AJT*	2,700	JT	12.6	UT	--	
WLCCWI08DMMU3B	WLCCWI08DMMU3B13	N	9/8/2008		Subsurface Sediment	30	91	cm	7628779	700408	-18	--		--		--		*704*	*AJT*	3,530	T	*261*	*JT*	--	
WLCCWI08DMMU3D	WLCCWI08DMMU3D13	N	9/8/2008		Subsurface Sediment	30	91	cm	7628873	700325	-13	--		--		--		*6.87*	*AJT*	8,600	JT	7.79	UT	--	
WLCCWI08DMMU3E	WLCCWI08DMMU3E13	N	9/5/2008		Subsurface Sediment	30	91	cm	7628981	700402	-24	--		--		--		*13*	*AT*	1,200	T	11.8	UT	--	
WLCPSK09DMMU1	WLCPSK091-A	N	11/12/2009		Subsurface Sediment	0	176	cm	7633135	700671	0	--		--		--		3.9	AT	*43,000*	*T*	*390*	*T*	--	
WLCPSK09DMMU1	WLCPSK091-Z	N	11/12/2009		Subsurface Sediment	207	327	cm	7633135	700671	0	--		--		--		*55*	*AT*	15,000	JT	*270*	*T*	--	
WLCPSK09DMMU2	WLCPSK092-A	N	11/11/2009		Subsurface Sediment	0	128	cm	7631557	700773	-50	--		--		--		2	AT	170	JT	6.7	UT	--	
WLCPSK09DMMU2	WLCPSK092-B	N	11/11/2009		Subsurface Sediment	128	280	cm	7631557	700773	-50	--		--		--		4.9	AT	310	JT	*19*	*T*	--	
WLCPSK09DMMU2	WLCPSK092-Z	N	11/11/2009		Subsurface Sediment	280	375	cm	7631557	700773	-50	--		--		--		5.6	AT	310	JT	*19*	*JT*	--	
WLCPWL09APS-1	WLCPWL09APS-1T	N	12/29/2009		Subsurface Sediment	0	60	cm	7628973	700319	-21	--		--		--		4.9	AJT	1,100	JT	*49*	*JT*	--	
WLCPWL09APS-1	WLCPWL09APS-1B	N	12/29/2009		Subsurface Sediment	60	121	cm	7628973	700319	-21	--		--		--		*9.9*	*AJT*	1,300	JT	*56*	*JT*	--	
WLCPWL09APS-2	WLCPWL09APS-2T	N	12/29/2009		Subsurface Sediment	0	60	cm	7629116	700296	-15	--		--		--		3.7	AJT	1,400	JT	*51*	*JT*	--	
WLCPWL09APS-2	WLCPWL09APS-2B	N	12/29/2009		Subsurface Sediment	60	121	cm	7629116	700296	-15	--		--		--		2.5	AJT	1,100	JT	*40*	*JT*	--	
WLCPWL09APS-3	WLCPWL09APS-3T	N	12/29/2009		Subsurface Sediment	0	60	cm	7629238	700264	-20	--		--		--		*11*	*AJT*	1,700	JT	*86*	*JT*	--	
WLCPWL09APS-3	WLCPWL09APS-3B	N	12/29/2009		Subsurface Sediment	60	121	cm	7629238	700264	-20	--		--		--		*14*	*AJT*	1,500	JT	*110*	*JT*	--	
WLCPWL09DRB-1	WLCPWL09DRB-1Z	N	12/29/2009		Subsurface Sediment	320	350	cm	7629031	700383	-22	--		--		--		*8.8*	*AJT*	1,400	JT	*79*	*JT*	--	
WLCPWL09DRB-2	WLCPWL09DRB-2Z	N	12/29/2009		Subsurface Sediment	307	338	cm	7629105	700447	-25	--		--		--		*7.4*	*AJT*	2,300	JT	*66*	*JT*	--	
WLCPWL09DRB-3	WLCPWL09DRB-3Z	N	12/29/2009		Subsurface Sediment	280	310	cm	7629193	700495	-25	--		--		--		*9.1*	*AJT*	4,100	JT	*82*	*JT*	--	
WLCPWL09DRB-D	WLCPWL09DRB-DP	N	12/29/2009		Subsurface Sediment	0	302	cm	7629110	700442	-24	--		--		--		*25*	*AJT*	1,700	JT	*54*	*JT*	--	
WLCPWL09URB-1	WLCPWL09URB-1Z	N	12/29/2009		Subsurface Sediment	295	326	cm	7629179	700364	-17	--		--		--		*12*	*AJT*	2,200	JT	*130*	*JT*	--	
WLCPWL09URB-2	WLCPWL09URB-2Z	N	12/29/2009		Subsurface Sediment	118	149	cm	7629295	700478	-23	--		--		--		*130*	*AJT*	9,900	JT	*200*	*JT*	--	
WLCPWL09URB-3	WLCPWL09URB-3Z	N	12/29/2009		Subsurface Sediment	167	198	cm	7629268	700417	-21	--		--		--		*8.2*	*AJT*	1,400	JT	*69*	*JT*	--	
WLCPWL09URB-D	WLCPWL09URB-DP	N	12/29/2009		Subsurface Sediment	0	194	cm	7629248	700420	-20	--		--		--		*9.6*	*AJT*	2,100	JT	*83*	*TJ*	--	
WLCWTI00N20	WLCWTI00N20N20	N	9/22/2000		Subsurface Sediment	15	30	cm	7628972	700109	9	--		--		--		--		--		--		--	
WLCWTI00N40	WLCWTI00N40N40	N	9/22/2000		Subsurface Sediment	15	30	cm	7628959	700126	8	--		--		--		--		--		--		--	
WLCWTI00S20	WLCWTI00S20S20	N	9/22/2000		Subsurface Sediment	15	30	cm	7628998	700078	8	--		--		--		--		--		--		--	
WLCWTI00S40	WLCWTI00S40S40	N	9/22/2000		Subsurface Sediment	15	30	cm	7629011	700062	9	--		--		--		--		--		--		--	
WLCWTI00TP11	WLCWTI00TP11TP11	N	9/21/2000		Subsurface Sediment	122	137	cm	7628955	700089	13	--		--		--		--		--		--		--	
WLR0499WRVC03	WLR0499WRVC03WRVC03	N	4/29/1999		Subsurface Sediment	0	320	cm	7637512	694910	-38	--		--		--		4.3	AUT	51	T	43	UT	--	
WLR0499WRVC04	WLR0499WRVC04WRVC04	N	4/29/1999		Subsurface Sediment	0	353	cm	7636312	695684	-38	--		--		--		3.2	AUT	140	T	32	UT	--	
WLR0499WRVC05	WLR0499WRVC05WRA	N	4/29/1999		Subsurface Sediment	0	350	cm	7633168	697274	-31	--		--		--		3.9	AUT	33	T	39	UT	--	
WLR0499WRVC05	WLR0499WRVC05WRVC05	N	4/29/1999		Subsurface Sediment	0	350	cm	7633168	697274	-31	0.0014	U	0.0018	U	0.00076	U	3.3	AUT	100	T	33	UT	--	
WLR0499WRVC06	WLR0499WRVC06WRVC06	N	4/29/1999		Subsurface Sediment	0	366	cm	7633356	697087	-32	--		--		--		3.4	AUT	120	T	34	UT	--	
WLR0499WRVC07	WLR0499WRVC07WRVC07	N	4/29/1999		Subsurface Sediment	0	366	cm	7633442	697348	-34	--		--		--		3.5	AUT	78	T	35	UT	--	
WLR0499WRVC08	WLR0499WRVC08WRVC08	N	4/29/1999		Subsurface Sediment	0	434	cm	7633118	697626	-32	--		--		--		3.5	AUT	120	T	35	UT	--	
WLR0499WRVC08	WLR0499WRVC08WRVC8C	N	4/29/1999		Subsurface Sediment	320	434	cm	7633118	697626	-32	--		--		--		*8.5*	*AT*	110	T	28	UT	--	
WLR0499WRVC09	WLR0499WRVC09WRVC09	N	4/29/1999		Subsurface Sediment	0	368	cm	7633972	697011	-31	--		--		--		*9.3*	*AT*	250	T	34	UT	--	
WLR1199WRVC03	WLR1199WRVC03WRVC03	N	11/29/1999		Subsurface Sediment	0	320	cm	7633124	697636	-27	--		--		--		*7.5*	*AJT*	120	T	--		--	
WLR1199WRVC04	WLR1199WRVC04WRVC04	N	11/29/1999		Subsurface Sediment	0	350	cm	7633470	697050	-27	--		--		--		4.8	AJT	130	T	--		--	
WLR1199WRVC05	WLR1199WRVC05WRVC05	N	11/29/1999		Subsurface Sediment	0	350	cm	7637517	694914	-28	--		--		--		*7*	*AJT*	120	T	--		--	
WLR1199WRVC06	WLR1199WRVC06C06ABC	N	11/29/1999		Subsurface Sediment	0	274	cm	7642826	689208	-17	--		--		--		6.1	AJT	250	T	32	UT	--	
WLR1199WRVC06	WLR1199WRVC06RVC06D	N	11/29/1999		Subsurface Sediment	274	365	cm	7642826	689208	-17	--		--		--		*8.2*	*AT*	400	T	29	UT	--	
WLR1199WRVC07	WLR1199WRVC07C07ABC	N	11/29/1999		Subsurface Sediment	0	228	cm	7643404	688852	-30	--		--		--		*10*	*AT*	860	T	29	UT	--	
WLR1199WRVC07	WLR1199WRVC07RVC07D	N	11/29/1999		Subsurface Sediment	228	304	cm	7643404	688852	-30	--		--		--		*17*	*AT*	460	T	26	UT	--	
WR-WSI98SD117	WR-WSI98SD1170000A	N	10/16/1997		Subsurface Sediment	0	90	cm	7629340	700260	-22	--		--		--		*47*	*T*	1,600	T	*160*	*JT*	--	

Table 2-1. FS Results for Samples Collected Within WCPA - Focused COCs

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Location ID	Sample Name	Sample Type	Sample Date	Dredge Area	Depth Class	Start Depth	End Depth	Depth Units	X	Y	Elevation (NAVD88)	1,2,3,7,8-PeCDD		2,3,4,7,8-PeCDF		2,3,7,8-TCDD		DDx - 6 Isomers (Calculated U = 1/2)		Total PAH (Calculated U = 1/2)		Total PCB Aroclors (Calculated U = 1/2)		Total PCB Congener (Calculated U = 1/2)	
											ft NAVD88	ug/kg		ug/kg		ug/kg		ug/kg		ug/kg		ug/kg		ug/kg	
											Sediment/Bank Soil CUL	**0.0002**		**0.0003**		**0.0002**		**6.1**		**23,000**		**9**		**9**	
											Sitewide RAL	**0.0008**		**0.2**		**0.0006**		**160**		**30,000**		**75**		**75**	
											PTW Threshold	**0.01**		**0.2**		**0.01**		**7,050**		**NA**		**200**		**200**	

Notes:

ug/kg = micrograms per kilogram

mg/kg = milligrams per kilogram

cm = centimeters

ft = feet

A = Summed value based on a limited number of analytes

N = normal/primary sample

J = estimated result

FR = field replicate

T = Result calculated or selected from >1 reported value

U = non detect result

Bold Italic = detected result exceeds Sediment/Bank Soil CUL

Yellow = detected result exceeds applicable RAL (Sitewide)

Underline = detected result exceeds PTW Threshold

Table 2-2. Pre-Remedial Design Results for Sample Collected Within WCPA - Focused COCs

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Location	Sample Name	Sample Date	Sample Type	Dredge Area	Depth Class	Start Depth	End Depth	Depth Unit	X Coordinate	Y Coordinate	Surface Elevation (ft NAVD88)	1,2,3,7,8-PeCDD ug/kg		2,3,4,7,8-PeCDF ug/kg		2,3,7,8-TCDD ug/kg		DDx – 6 Isomers ug/kg		Total PAHs ug/kg		Total PCB Aroclors ug/kg		Total PCB Congeners ug/kg	
											Sediment/Bank Soil CUL	**0.0002**		**0.0003**		**0.0002**		**6.1**		**23,000**		**9**		**9**	
											Sitewide RAL	**0.0008**		**0.2**		**0.0006**		**160**		**30,000**		**75**		**75**	
											PTW Threshold	**0.01**		**0.2**		**0.01**		**7,050**		**NA**		**200**		**200**	
B236	PDI-SG-B236-BL1	20-Apr-18	N		Surface Sediment	0	30	cm	7628694	701205	-11.7	***0.00036***	***JN***	***0.0012***	***J***	0.00018	JN	***6.17***		441.4		--		7.9	
B237	PDI-SG-B237-BL1	20-Apr-18	N		Surface Sediment	0	30	cm	7628595	700933	-19.6	***0.00035***	***JN***	***0.001***	***J***	0.00015	JN	***9.85***		647.4		--		***10.1***	
B239	PDI-SG-B239-BL1	21-Apr-18	N		Surface Sediment	0	30	cm	7628724	700674	-32.8	***0.00053***	***J***	***0.07***		***0.00035***	***JN***	***8.57***		910.1		--		***14.4***	
B241	PDI-SG-B241-BL1	21-Apr-18	N		Surface Sediment	0	30	cm	7629094	700309	-14.0	***0.0006***	***JN***	***0.00087***	***J***	***0.00031***	***JN***	***6.74***		1,378		--		***18.1***	
B245	PDI-SG-B245-BL1	21-Apr-18	N		Surface Sediment	0	22	cm	7629623	700262	-6.1	0.00012	J	0.00019	JN	0.000059	U	2.175		307.51		--		2.9	
B246	PDI-SG-B246-BL1	22-May-18	N		Surface Sediment	0	30	cm	7629842	700111	-3.3	***0.0004***	***J+***	***0.00052***	***JN***	0.00015	U	4.22		672.1		--		7.3	
B251	PDI-SG-B251-BL1	22-May-18	N		Surface Sediment	0	30	cm	7630185	699861	-18.5	***0.00049***	***J+***	***0.00064***	***J***	0.00023	U	***6.63***		734.3		--		***11.5***	
B253	PDI-SG-B253-BL1	23-May-18	N		Surface Sediment	0	24	cm	7630335	699548	3.5	***0.00024***	***J+***	0.0003	J+	0.000056	U	0.94	U	435.5		--		***15.7***	
B257	PDI-SG-B257-BL1	22-May-18	N		Surface Sediment	0	24	cm	7630634	699465	-1.7	0.000053	U	0.000074	U	0.000077	U	0.94	U	3.38		--		0.1	
S157	PDI-SG-S157	14-May-18	N	2011 Dredge Area	Surface Sediment	0	30	cm	7628996	700977	-35.4	***0.00051***	***J***	***0.0061***		***0.0005***	***J***	***13.275***		392.9		--		***23.9***	
S158	PDI-SG-S158	14-May-18	N	2011 Dredge Area	Surface Sediment	0	25	cm	7629028	701045	-35.1	***0.00056***	***J***	***0.0017***	***J***	***0.00064***	***J***	***11.75***		1,339		--		***42.8***	
S159	PDI-SG-S159	18-May-18	N	2011 Dredge Area	Surface Sediment	0	30	cm	7629035	700770	-33.9	***0.00068***	***J***	***0.0029***	***J***	***0.00033***	***JN***	***25.6***		3,402		--		***18.6***	
S160	PDI-SG-S160	18-May-18	N	2011 Dredge Area	Surface Sediment	0	30	cm	7628845	700432	-32.7	***0.00055***	***JN***	***0.0023***	***J***	***0.00038***	***JN***	***8.77***		1,286		--		***18.7***	
S161	PDI-SG-S161	14-May-18	N	Riverward end of Chevron Slip (not dredged in 2011)	Surface Sediment	0	29	cm	7629278	700784	-38.3	***0.00052***	***J***	***0.00076***	***J***	***0.00053***	***J***	***7.56***		771.9		--		***9.8***	
S162	PDI-SG-S162	18-May-18	N		Surface Sediment	0	30	cm	7629428	700572	-23.2	***0.00053***	***JN***	***0.0023***	***J***	0.0002	U	***7.05***		1,154		--		***11.4***	
S163	PDI-SG-S163	18-May-18	N		Surface Sediment	0	30	cm	7629262	700356	-19.7	***0.00077***	***J***	***0.0032***	***J***	***0.00052***	***J***	***8.61***		1,039		--		***15.3***	
S164	PDI-SG-S164	20-Jun-18	N		Surface Sediment	0	27	cm	7629973	700029	-9.5	***0.00069***	***J***	***0.00074***	***J***	***0.00037***	***JN***	5.22		2,898		--		***13.2***	
S165	PDI-SG-S165	16-May-18	N		Surface Sediment	0	27	cm	7630314	699724	-16.0	0.00029	U	***0.0013***	***J***	***0.00057***	***JN***	5.9		410.3		--		***14.3***	
SC-S155	PDI-SC-S155-0to2.1	05-Sep-18	N		Subsurface Sediment	0	2.1	ft	7628607	701255	-1.32	***0.00038***	***J***	***0.0012***	***J***	0.00018	JN	***18.379***		619.1		***12.1***		--	
SC-S155	PDI-SC-S155-2.1to4.2	05-Sep-18	N		Subsurface Sediment	2.1	4.2	ft	7628607	701255	-1.32	***0.001***	***J***	***0.01***		***0.00062***	***J***	***89.14***		562.8		***27.1***		--	
SC-S155	PDI-SC-S155-4.2to5.3	05-Sep-18	N		Subsurface Sediment	4.2	5.3	ft	7628607	701255	-1.32	0.00006	J	0.000087	J+	0.000016	U	2.577		176.5		2.6	U	--	
SC-S157	PDI-SC-S157-0to2	17-Aug-18	N	2011 Dredge Area	Subsurface Sediment	0	2	ft	7629021	700988	-37.3	0.00022	U	***0.0012***	***J***	***0.00023***	***JN***	***7.065***		777		***13.2***		--	
SC-S157	PDI-SC-S157-2to3.7	17-Aug-18	N	2011 Dredge Area	Subsurface Sediment	2	3.7	ft	7629021	700988	-37.3	***0.00023***	***J***	***0.0008***	***J***	0.00012	JN	***6.407***		1,764		***9.4***		--	
SC-S157	PDI-SC-S157-3.7to6	17-Aug-18	N	2011 Dredge Area	Subsurface Sediment	3.7	6	ft	7629021	700988	-37.3	0.00012	U	0.000075	U	0.0001	U	2.18605		28.86		2.7	U	--	
SC-S157	PDI-SC-S157-6to8	17-Aug-18	N	2011 Dredge Area	Subsurface Sediment	6	8	ft	7629021	700988	-37.3	0.00012	U	0.000074	U	0.0001	U	0.2464		45.19		2.7	U	--	
SC-S157	PDI-SC-S157-8to10	17-Aug-18	N	2011 Dredge Area	Subsurface Sediment	8	10	ft	7629021	700988	-37.3	0.00013	U	0.000074	U	0.000097	U	0.386		16.21		2.7	U	--	
SC-S157	PDI-SC-S157-10to12.4	17-Aug-18	N	2011 Dredge Area	Subsurface Sediment	10	12.4	ft	7629021	700988	-37.3	0.000091	U	0.000068	U	0.00008	U	0.25	U	15.08		2.6	U	--	
SC-S157	PDI-SC-S157-12.4to14	17-Aug-18	N	2011 Dredge Area	Subsurface Sediment	12.4	14	ft	7629021	700988	-37.3	0.000098	U	0.000062	U	0.000085	U	0.089		52.25		2.6	U	--	
SC-S157	PDI-SC-S157-14to15.9	17-Aug-18	N	2011 Dredge Area	Subsurface Sediment	14	15.9	ft	7629021	700988	-37.3	0.000077	U	0.00006	U	0.00008	U	0.035	U	15.02		2.6	U	--	
SC-S163	PDI-SC-S163-0to2	27-Jul-18	N		Subsurface Sediment	0	2	ft	7629284	700353	-20.8	***0.00086***	***J***	***0.00083***	***JN***	***0.00073***	***JN***	***15.069***		2,998		***27***		--	
SC-S163	PDI-SC-S163-10TO12.7	27-Jul-18	N		Subsurface Sediment	10	12.7	ft	7629284	700353	-20.8	***0.0025***	***J***	***0.027***		***0.0023***		***112.636***		14,264		***285***		--	
SC-S163	PDI-SC-S163-12.7to13	27-Jul-18	N		Subsurface Sediment	12.7	13	ft	7629284	700353	-20.8	0.000092	U	0.000052	U	0.000084	U	0.23215		2,722		2.7	U	--	
SC-S163	PDI-SC-S163-2to4	27-Jul-18	N		Subsurface Sediment	2	4	ft	7629284	700353	-20.8	***0.0012***	***J***	***0.0016***	***J***	***0.0013***		***25.7446***		3,373		***177***		--	
SC-S163	PDI-SC-S163-4to6	27-Jul-18	N		Subsurface Sediment	4	6	ft	7629284	700353	-20.8	***0.00098***	***J***	***0.0012***	***J***	***0.0013***		***17.1974***		3,842		***59.8***		--	
SC-S163	PDI-SC-S163-6to8	27-Jul-18	N		Subsurface Sediment	6	8	ft	7629284	700353	-20.8	***0.0016***	***J***	***0.0036***	***J***	***0.0017***		***28.434***		3,966		***88.2***		--	
SC-S163	PDI-SC-S163-8to10	27-Jul-18	N		Subsurface Sediment	8	10	ft	7629284	700353	-20.8	***0.0019***	***JN***	***0.0043***	***J***	***0.0011***		***17.6245***		5,981		***50.8***		--	

Notes:

ug/kg = micrograms per kilogram

mg/kg = milligrams per kilogram

cm = centimeters

ft = feet

J = estimated result

N = normal/primary sample

U = non detect result

Bold Italic = detected result exceeds Sediment/Bank Soil CUL

Yellow = detected result exceeds applicable RAL (Sitewide)

Underline = detected result exceeds PTW Threshold

Table 2-3. Summary of CULs and RALs for Selected Parameters in Sediment and River Bank Soil
Pre-Design Investigation Work Plan, Willbridge Cove Project Area

	From ROD Table 17	From ROD Table 21			
Contaminant	Sediment/Bank Soil CUL (µg/kg)	Site-wide RAL (µg/kg)	PTW Threshold (µg/kg)	Navigation Channel RALs (µg/kg)	Regional Soil Background 95% UPL (mg/kg)*
Table 21 Focused COCs					
PCBs	9	75	200	1,000	--
Total PAHs	23,000	*30,000*	NA	170,000	--
2,3,7,8-TCDD	0.0002	0.0006	0.01	0.002	--
1,2,3,7,8-PeCDD	0.0002	0.0008	0.01	0.003	--
2,3,4,7,8-PeCDF	0.0003	0.2	0.2	1	--
DDx	6.1	160	7,050	650	--
Table 21 Additional Contaminants					
2,3,7,8-TCDF	0.0004	--	0.6	--	--
1,2,3,4,7,8-HxCDF	0.0004	--	0.04	--	--
cPAHs (BaP Eq)	*85 (Beach areas - Direct contact)* *774 (nearshore sediment, excluding beach areas - direct contact CUL)* *1,076 (Navigation channel)*	--	*774,000*	--	--
Chlorobenzene	--	--	>320	--	--
Naphthalene	--	--	>140,000	--	--
Table 17 CUL River Bank Soils and Sediment					
Aldrin	2	--	--	--	--
Arsenic	3 mg/kg	--	--	--	8.791
BEHP	135	--	--	--	--
Cadmium	0.51 mg/kg	--	--	--	0.627
Chlordanes	1.4	--	--	--	--
Copper	359 mg/kg	--	--	--	33.75
DDD	114	--	--	--	--
DDE	50	--	--	--	--
DDT	246	--	--	--	--
Dieldrin	0.07	--	--	--	--
Lindane	5	--	--	--	--
Lead	196 mg/kg	--	--	--	28
Mercury	0.085 mg/kg	--	--	--	0.23
Dioxin/Furan (2,3,7,8-TCDD eq)	0.01				--
TPH Diesel	91 mg/kg	--	--	--	--
Tributyltin	3,080	--	--	--	--
Zinc	459 mg/kg	--	--	--	182.9

Notes:

Values updated in accordance with Explanation of Significant Differences (EPA, 2019a) and Errata #2 for Portland Harbor Superfund Site Record of Decision ROD Table 17 (EPA, 2020c)

Dioxin/furan (2,3,7,8-TCDD eq) value is calculated based on the 2005 *World Health Organization Re-evaluation of Human and Mammalian Toxic Equivalency Factors for Dioxins and Dioxin-like Compounds* (Van den Berg et al., 2006).

*Background values for all metals, except lead, are those listed for the Portland Basin in Table 3 of *Development of Oregon Background Metals Concentrations in Soil* (Oregon DEQ 2013). The background value for lead is taken from the South Willamette Valley portion of Table 3 because the listed value for the Portland Basin appears to include anthropogenic influences.

-- = not applicable
µg/kg = microgram(s) per kilogram
BEHP = bis(2-ethylhexyl)phthalate
COC = contaminant of concern
cPAH = carcinogenic polycyclic aromatic hydrocarbon
CUL = cleanup level
DDD = dichlorodiphenyldichloroethane
DDE = dichlorodiphenyldichloroethene
DDT = dichlorodiphenyltrichloroethane
DDx = DDD, DDE, and DDT
mg/kg = milligram(s) per kilogram
PAH = polycyclic aromatic hydrocarbon
PCB = polychlorinated biphenyl
PTW = principal threat waste
RAL = remedial action level
ROD = Record of Decision
TPH = total petroleum hydrocarbons
UPL = upper prediction limit

Table 3-1. Summary of Proposed Sampling Designs for WCPA Sediment and River Bank Soil

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Data Gap Area	Current and Anticipated Use	Sample Design for SMA Characterization and Refinement	Existing Sample Locations in Area to be incorporated into SMA refinement during PDI evaluation	Number and Type of PDI Sample Locations	Target Core Depth(s)
Preliminary SMA 1	Intermediate region between FMD area and shoreline	Resample RAL exceedance location (G407) with sufficient density for data replacement Step-out surface grabs and cores on 150-ft spacing along/adjacent to long axis of SMA	FS: C403; C698 Pre-RD: B246, S164, B251, S165, B253	Primary surface grab/cores – 1 Primary surface grabs – 2 Step-out surface grabs/cores – 5	10 ft
Preliminary SMA 2	Includes regularly-dredged FMD areas, not recently-dredged FMD areas, and areas under docks	Resample RAL exceedance locations (AN-CTD-03, AN-CTD-07) with sufficient density for data replacement Surface grabs and cores on 150-ft grid within remainder of preliminary SMA Surface grab beneath docks Step-out surface grabs and cores on 150-ft spacing around perimeter of preliminary SMA	Pre-RD: S157, SC-S157, S158, S159, S161, S162	Primary surface grab/cores – 6 Primary surface grabs – 3 Under-dock surface grabs – 4 Step-out surface grabs/cores – 7	10 ft
Preliminary SMA 3	Includes not-recently dredged FMD area, may extend into intermediate area and area under dock	Surface grabs and cores on 150-ft grid within preliminary SMA Surface grabs beneath dock Step-out surface grabs and cores on 150-ft spacing around perimeter of preliminary SMA	FS: C532 Pre-RD: S163, SC-S163, B241	Primary surface grabs/cores – 4 Under-dock surface grab – 1 Step-out surface grabs/cores – 6	15 ft 10 ft in deeper water area
Preliminary SMAs 4 and 5	Includes regularly dredged FMD areas, intermediate area, and area under dock	Resample RAL exceedance locations which were subsequently removed by dredging in 2011 (AN-CTPD-01 and AN-CTOD-08) to establish current conditions, include cores at resample locations Surface grabs and cores on 150-ft grid within preliminary SMA Surface grab sample beneath dock Step-out surface grabs on steep shoreline near historical PTW threshold exceedances Step-out surface grabs and cores on 150-ft spacing around perimeter of preliminary SMA	Pre-RD: S160, B239	Primary surface grabs/cores – 9 Under dock surface grab –1 Step-out surface grabs – 2 Step-out surface grabs/cores - 2	10 ft 15 ft in shoaled area
River Bank Adjacent Preliminary SMA 4	River bank	Surface samples and cores at 100 ft spacing along MLW and OHW elevation contours on river bank		Surface soil samples – 12 Subsurface soil cores – 12	5 ft

Notes:
FMD = future maintenance dredge
FS = Feasibility Study
ft = feet
MLW = mean low water
OHW = ordinary high water
Pre-RD = Pre-Remedial Design Investigation
PTW = principal threat waste
SMA = sediment management area

Table 3-2 - Proposed Sediment Sample Locations and Associated Analytical Schedule

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Location ID	Primary or Stepout	Sample Type (Grab/Core)	Target Core Depth (ft)	Target X	Target Y	Elevation Portland Harbor 2018 Bathymetry (NAVD88)	Within FMD Area	FMD Operational Elevation (NAVD88)	Immediate Analysis and Archiving Schedule
Preliminary SMA 1									
PD01	Stepout	Surface Grab & Core	10	7630034	699944	-6.2	No	NA	A
PD02	Primary	Surface Grab	NA	7630123	699852	-8.6	No	NA	X
PD03	Primary	Surface Grab & Core	10	7630139	699833	-9.5	No	NA	B
PD04	Primary	Surface Grab	NA	7630155	699813	-10.9	No	NA	X
PD05	Stepout	Surface Grab & Core	10	7630111	699799	0.0	No	NA	A
PD06	Stepout	Surface Grab & Core	10	7630218	699706	-0.2	No	NA	A
PD07	Stepout	Surface Grab & Core	10	7630326	699601	-1.8	No	NA	A
PD08	Stepout	Surface Grab & Core	10	7630454	699489	-4.5	No	NA	A
Preliminary SMA 2									
PD09	Stepout	Surface Grab & Core	10	7629110	701165	-32.5	No - edge of channel	-34.7	A
PD10	Stepout	**Surface Grab & Core**	10	7629417	700791	-40.4	No - edge of channel	-34.7	A
PD11	Stepout	Surface Grab & Core	10	7629606	700558	-28.2	No - edge of channel	-34.7	A
PD12	Primary - Under Dock	Surface Grab	NA	7629005	701116	-20.7	No	NA	X
PD13	Primary	Surface Grab	NA	7629121	700972	-37.2	Yes - recently dredged	-34.7	X
PD14	Primary	Surface Grab & Core	10	7629137	700953	-37.0	Yes - recently dredged	-34.7	C
PD15	Primary	Surface Grab	NA	7629153	700933	-36.7	Yes - recently dredged	-34.7	X
PD16	Primary - Under Dock	Surface Grab	NA	7629210	700872	-24.3	No	NA	X
PD17	Primary	Surface Grab	NA	7629240	700793	-37.5	Yes - recently dredged	-34.7	X
PD18	Primary	Surface Grab & Core	10	7629240	700768	-38.0	Yes - recently dredged	-34.7	B
PD19	Primary	Surface Grab & Core	10	7629356	700675	-34.9	Yes - not recently dredged	-19.7*	B
PD20	Primary - Under Dock	Surface Grab	NA	7629410	700638	-25.5	No	NA	X
PD21	Primary	Surface Grab & Core	10	7629478	700546	-23.9	Yes - not recently dredged	-14.7**	B
PD22	Stepout	Surface Grab & Core	10	7629579	700428	-20.6	No	NA	A
PD23	Primary	Surface Grab & Core	10	7629027	700859	-33.9	Yes - recently dredged	-34.7	C
PD24	Stepout	Surface Grab & Core	10	7629167	700689	-35.0	Yes - recently dredged	-34.7	A
PD25	Stepout	Surface Grab & Core	10	7628907	700907	-34.7	Yes - recently dredged	-33.2	A
PD26	Primary - Under Dock	Surface Grab	NA	7629037	700731	-24.0	No	NA	X
PD27	Stepout	Surface Grab & Core	10	7628906	700718	-33.2	Yes - recently dredged	-34.7	A
Preliminary SMA 3									
PD28	Primary	Surface Grab & Core	10	7629070	700632	-34.7	Yes - recently dredged	-34.7	C
PD29	Stepout	Surface Grab & Core	10	7629391	700519	-23.8	Yes - not recently dredged	-14.7	A
PD30	Stepout	Surface Grab & Core	10	7629483	700407	-22.4	No	NA	A
PD31	Primary	Surface Grab & Core	15	7629282	700433	-22.6	Yes - not recently dredged	-14.7	B
PD32	Primary	Surface Grab & Core	15	7629377	700309	-22.3	No	NA	B
PD33	Stepout	Surface Grab & Core	15	7629425	700192	-16.6	No	NA	A
PD34	Primary - Under Dock	Surface Grab	NA	7629126	700405	-20.1	No	NA	X
PD35	Primary	Surface Grab & Core	15	7629152	700330	-14.6	Yes - not recently dredged	-14.7	B
PD36	Primary	Surface Grab & Core	15	7629255	700209	-17.4	No	NA	B
PD37	Stepout	Surface Grab & Core	15	7629318	700113	0.0	No	NA	A
PD38	Stepout	Surface Grab & Core	15	7629053	700233	-13.1	Yes - not recently dredged	-14.7	A
PD39	Stepout	Surface Grab & Core	15	7629140	700120	-11.8	No	NA	A
Preliminary SMA 4									
PD40	Stepout	Surface Grab & Core	10	7628637	700704	-33.2	Yes - recently dredged	-33.2	A
PD42	Primary	Surface Grab & Core	10	7628755	700547	-32.2	Yes - recently dredged	-34.7	C
PD43	Primary - Under Dock	Surface Grab	NA	7628791	700526	-20.0	No	NA	X
PD44	Primary	Surface Grab & Core	10	7628854	700450	-33.1	Yes - recently dredged	-34.7	C
PD45	Stepout	Surface Grab & Core	10	7629003	700514	-34.0	Yes - recently dredged	-34.7	A
PD46	Primary	Surface Grab & Core	15	7628968	700363	-21.2	Yes - not recently dredged	-19.7*	B

Table 3-2 - Proposed Sediment Sample Locations and Associated Analytical Schedule

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Location ID	Primary or Stepout	Sample Type (Grab/Core)	Target Core Depth (ft)	Target X	Target Y	Elevation Portland Harbor 2018 Bathymetry (NAVD88)	Within FMD Area	FMD Operational Elevation (NAVD88)	Immediate Analysis and Archiving Schedule
PD47	Primary	Surface Grab & Core	10	7628607	700614	-14.5	No	NA	B
PD48	Primary	Surface Grab & Core	10	7628696	700502	-18.2	No	NA	B
PD49	Stepout	Surface Grab	NA	7628741	700426	-9.7	No	NA	A
PD50	Primary	Surface Grab & Core	10	7628773	700394	-14.2	No	NA	B
PD51	Stepout	Surface Grab	NA	7628799	700360	-10.5	No	NA	A
PD52	Primary	Surface Grab & Core	10	7628834	700355	-23.9	No	NA	B
PD53	Primary	Surface Grab & Core	10	7628912	700298	-17.4	No	NA	B
Preliminary SMA 5									
PD41	Primary	Surface Grab & Core	10	7628744	700632	-32.8	Yes - recently dredged	-34.7	C

Notes:

*Permitted elevation is -39 CRD or -33.7 NAVD88, but operational elevation is much shallower

**Permitted elevation is -32 CRD or -26.7 NAVD88, but operational elevation is much shallower

A = Archive all sampling intervals for all chemical analyses

B = Analyze surface grab and core intervals 1-2 ft, 2-3 ft, 3-4 ft, and 4-5 ft immediately. Archive core samples 0-1 foot and below 5 feet.

C = Analyze surface grab immediately and the four sediment core intervals at and below the the operational elevation immediately. Archive all other core sample intervals.

X = Analyze surface grab immediately.

DDD = dichlorodiphenyldichloroethane

DDE = dichlorodiphenyldichloroethene

DDT = dichlorodiphenyltrichloroethane

DDx = DDD, DDE, and DDT

PAH = polycyclic aromatic hydrocarbon

PCB = polychlorinated biphenyl

TOC = total organic carbon

Coordinates are in NAD 1983 StatePlane Oregon North FIPS 3601 Feet Int

Samples for the following analyses will be collected from each sampling interval.

PAHs by SW8270 C or D-SIM
PCB Aroclors by SW8082
DDX isomers by 1699M
Dioxins/Furans by E1613
Grainsize by D7928, D6913
TOC by 9060
Archive aliquot

Table 3-3 - Proposed River Bank Soil Sample Locations and Associated Analytical Schedule

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Location ID	Sample Description	Target X	Target Y	Target Boring Depth	Analysis and Archiving Scheme
WC-S01	Soil Boring	7628501	700669	5 ft	All surface soil samples will be analyzed immediately for Table 17 analyses listed below. All subsurface soil samples will be held frozen pending analysis of the surface interval. Subsurface soil intervals released for analysis will be analyzed for Table 21 analyses listed below.
WC-S02	Soil Boring	7628534	700624	5 ft	
WC-S03	Soil Boring	7628576	700549	5 ft	
WC-S04	Soil Boring	7628590	700560	5 ft	
WC-S05	Soil Boring	7628652	700470	5 ft	
WC-S06	Soil Boring	7628661	700480	5 ft	
WC-S07	Soil Boring	7628719	700401	5 ft	
WC-S08	Soil Boring	7628728	700407	5 ft	
WC-S09	Soil Boring	7628802	700273	5 ft	
WC-S10	Soil Boring	7628798	700303	5 ft	
WC-S11	Soil Boring	7628835	700236	5 ft	
WC-S12	Soil Boring	7628856	700256	5 ft	

Notes:

Surface soils will be analyzed for the following:

- Metals, including mercury by SW6010B or C and SW7471A or B
- TPH-DRO by NWTPH-Dx
- PAHs by SW8270 C or D-SIM
- Bis(2-ethylhexyl)phthalate (BEHP) by low level SW8270C or D-SIM
- Pesticides by E1699
- Dioxins/Furans by E1613
- PCB congeners by E1668

Analysis of subsurface soils, if needed, will be performed using the following methods:

- PAHs by SW8270 C or D-SIM
- PCB Aroclors by SW8082
- DDT isomers by 1699M
- Dioxins/Furans by E1613

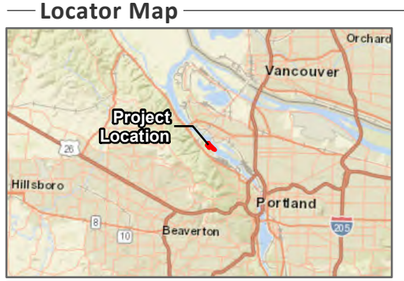
Coordinates are in NAD 1983 StatePlane Oregon North FIPS 3601 Feet Intl

Figures

Willbridge Cove Project Area

Forest Park
Smith and Bybee Lakes Park
Columbia River
Willamette River
Portland
Bethany
Oak Hills
Cedar Mill
Washington
Multnomah
Heron Lakes Golf Course
Portland International Raceway
Portland International Airport
Columbia Slough
Johnson Creek
McCarthy Creek
Miller Creek
Newberry
Skyline
Germantown
Rock Creek
Abbey Creek
Springville
Kaiser
Doane Creek
Saltzman Creek
Burgard
Columbia
Lombard
Saint Louis
Smith
Fessenden
Richmond
Oswego
Wall
Portsmouth
Willis
Chautauqua
Peninsular
Delaware
Kilpatrick
Denver
Marine
Vancouver
Whitaker
Schmeer
Victory 5 Ramp
Walker
6th
3rd
33rd
Columbia
Willamette
Pearson Field
Columbia House
Clark
Evergreen
Cornfoot
47th
Dekum
15th
Ainsworth
Albina
Jessup
Killingsworth
Alberta
Prescott
37th
42nd
Fremont
33rd
Cook
Williams
Russell
Knott
21st
36th
39th
41st
Tillamook
Broadway
24th
Halsey
Multnomah
16th
Irving
20th
12th
32nd
Glisan
Glisan Circle
Burnside
Sandy
Lovejoy
23rd
19th
18th
Everett
Broadway Bridge
Burnside Bridge
Vista
4th
Front
Saint Helens
Nicolai
Ward
Vaughn
Weidler
Going
Brandena Going
Gregory
Wilbridge
Cornell
Murray
Barnes
Cedar Hills
112th
158th
143rd
185th
Thompson
West Union
Willow Creek
Cedar Mill Creek
Bronson Creek
Evergreen
Walker
Bethany
Cornelius Pass
Hillsboro
Beaverton
Orchard
Vancouver
Project Location
26
30
99
5
405
84
14

Locator Map



Legend

- Limited Access
- Highway
- Major Road
- Railroads (Local)
- Rivers (Local)
- Stream/River
- City
- Hospital
- Willbridge Cove Project Area
- Local Park
- State Park or Forest
- Golf Course
- Stadium or Arena

Map Notes
* Base data from ESRI Basemaps

0 5 10 20 Miles

N

FIGURE 1-1
SITE LOCATION
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

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Willamette River

RM 7.5
RM 7.6
RM 7.7
RM 7.8
RM 7.9
RM 8
RM 8.1

-44
-42
-38
-36
-34
-32
-28
-26
-24
-22
-18
-16
-14
-12
-8
-6
-4
-2
2
4
6
8
12
14

Preliminary SMA 1
Preliminary SMA 2
Preliminary SMA 3
Preliminary SMA 4
Preliminary SMA 5

Authorized Dredge Areas/
Future Maintenance Dredge (FMD) Areas

McCall Dock
Kinder Morgan Dock
Chevron Dock
Phillips 66 Dock
Tidewater Barge

Atofina Chemicals - former Arkema Portland

Glacier Northwest, Inc.

McCall Oil and Chemical Corporation

GS Roofing

Kinder Morgan Liquids Terminals LLC

Chevron Willbridge Terminal

Phillips 66 Willbridge Terminal

Tube Forgings of America

NW Front Ave

Document Path: \\dc1vs011\GIS\Proj\PortlandHarbor\Group\p2_Willbridge\Cove\Mxd\Files\Report_PDI\40_WC_SMA_Remedy\Tech_wDredge.mxd

Locator Map

Legend

- Shallow/Intermediate Zone Boundary
- Top of Bank
- River Miles (Tenths)
- Bathymetry (2ft Contour) NAVD88
- Willbridge Cove Project Area
- Navigation Channel
- Tax Lots (Metro RLIS)
- Authorized Dredge Areas/ Future Maintenance Dredge (FMD) Areas

Bathymetry Zones

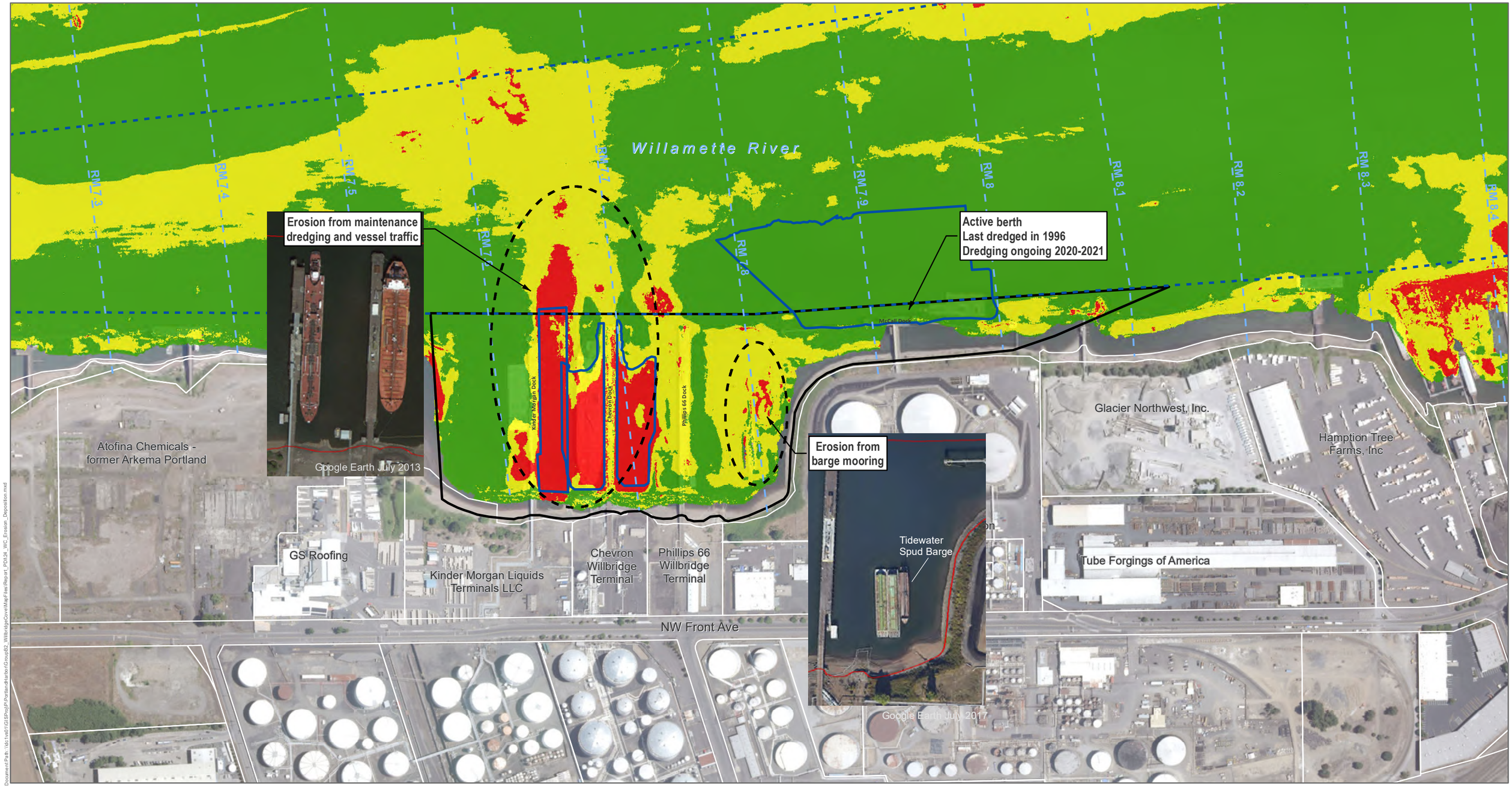
- Intermediate Area
- Shallow Area

Map Notes
* Metro RLIS Tax Lots (Tax Lots Identification TLID)
* Metro 2019 Aerial Imagery
* Base data from Portland Harbor Environmental Data Portal (http://ph-public-data.com/)

0 187.5 375 Feet

FIGURE 1-2
SITE FEATURES RELEVANT TO ANTICIPATED TECHNOLOGY ASSIGNMENT
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



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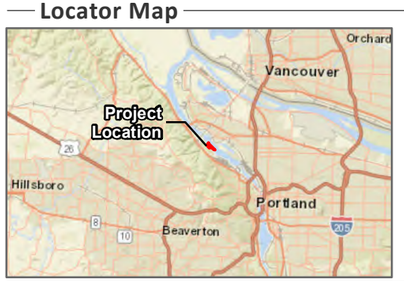
Locator Map



Legend

- River Miles (Tenths)
- Dredge Area
- Navigation Channel
- Willbridge Cove Project Area
- Tax Lots (Metro RLIS)

Deposition and Erosion Area Compared 2009 through 2018

- Erosional: -2.5 cm/yr or more
- Neutral: -2.5 to 2.5 cm/yr
- Depositional: 2.5 cm/yr or more

Map Notes
* Metro RLIS Tax Lots (Tax Lots Identification TLID)
* Metro 2018 Aerial Imagery
* Base data from Portland Harbor Environmental Data Portal (http://ph-public-data.com/)
* Deposition & erosion surface from the PDI Evaluation Report, June 2019

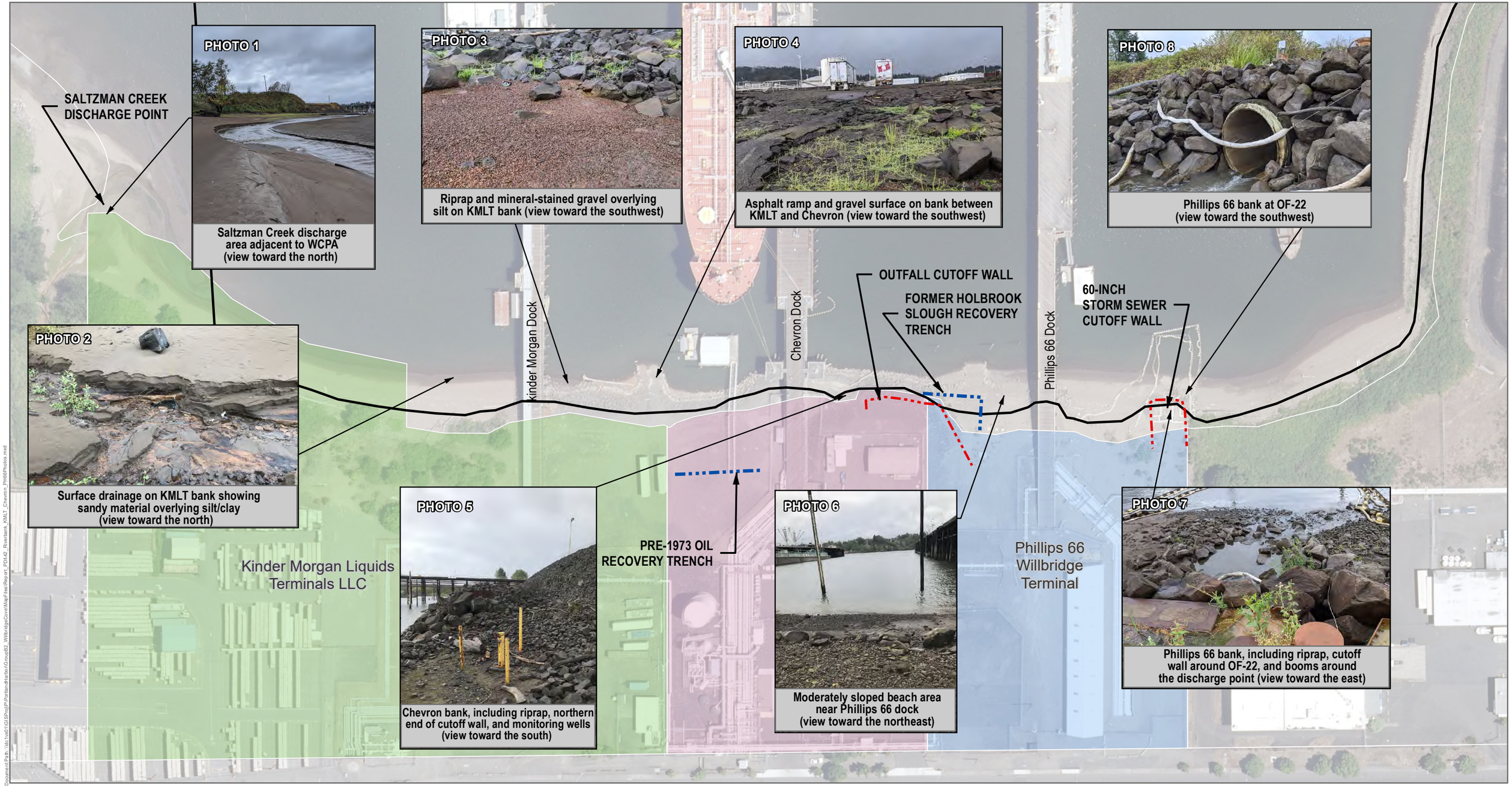
0 187.5 375 750 Feet

FIGURE 2-1

EROSION AND DEPOSITION RATES IN WILLBRIDGE COVE PROJECT AREA

Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



Locator Map

Legend

- Former Recovery Trench
- Cutoff Wall
- Tax Lots (Metro RLIS)
- Phillips 66 Willbridge Terminal
- Chevron Willbridge Terminal
- Kinder Morgan Liquids Terminal LLC

Map Notes
* Metro RLIS Tax Lots
* Metro 2018 Aerial Imagery

0 137.5 275 Feet

OUTFALL #1

FIGURE 2-2

RIVER BANK CONDITIONS ALONG KINDER MORGAN, CHEVRON, AND PHILLIPS 66

Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

McCALL DOCK

CHEVRON DOCK

PHILLIPS 66 DOCK

Tidewater Spud Barge

McCALL

FORMER CHEMAX

HIGH PURITY PRODUCTS

BRENNTAG PACIFIC

ASPHALT PLANT

NW Front Ave

PHOTO 3

McCall bank and Tidewater barges (view to the southeast)

PHOTO 2

OF-22 booms, Tidewater barges, and McCall bank (view toward the southeast)

PHOTO 1

McCall bank, moderately sloped sandy beach, and drainage from outfall WR-11 (view toward the south)

Locator Map

Legend

Tax Lots (Metro RLIS)

McCall Terminal

Map Notes
* Metro RLIS Tax Lots (Tax Lots Identification TLID)
* Metro 2018 Aerial Imagery

0 200 400 Feet

FIGURE 2-3
RIVER BANK CONDITIONS ALONG McCALL PROPERTY
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

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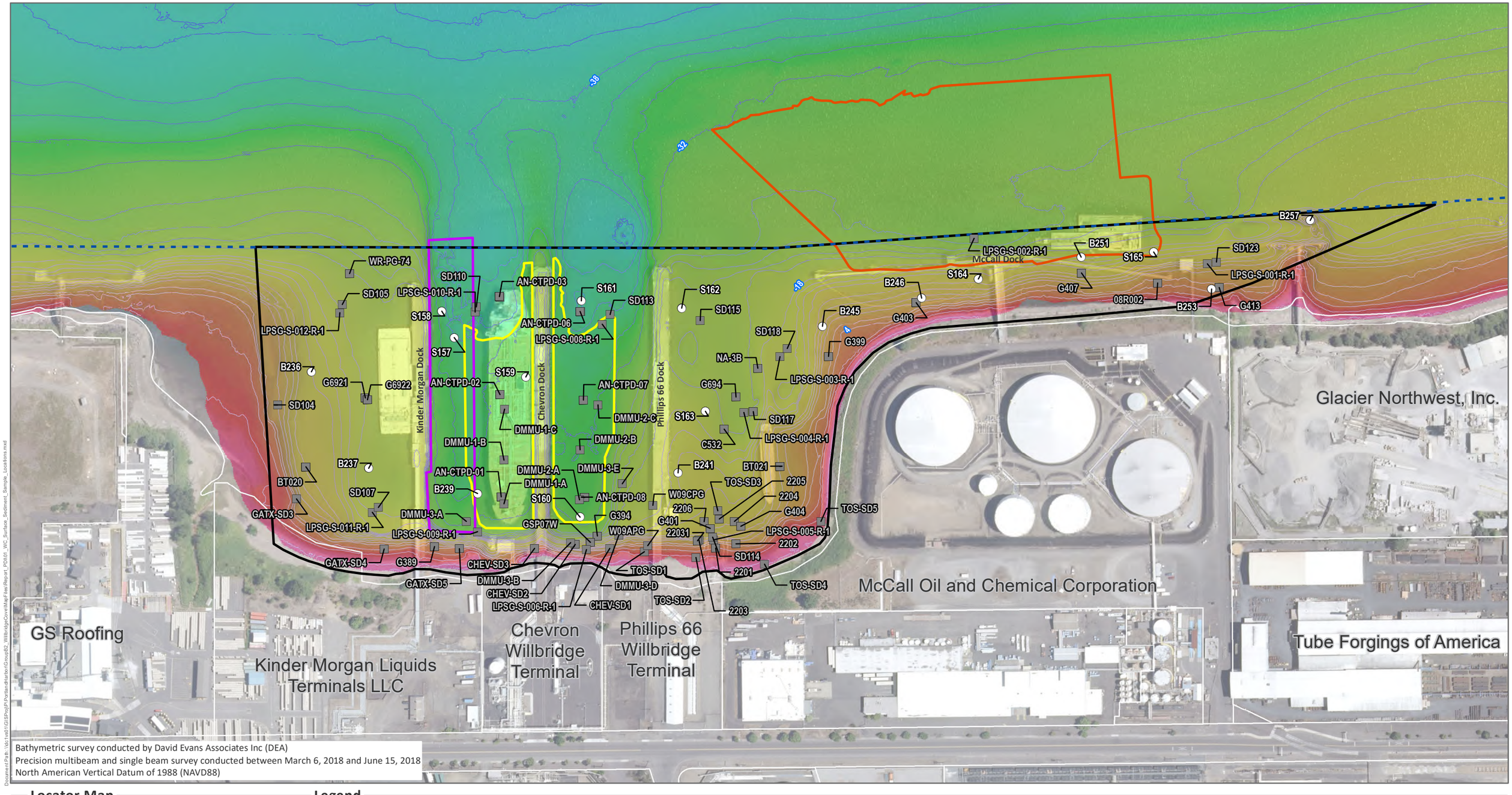

Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

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Locator Map

Legend

- PDI-BL (Pre-RD) Surface Sample
- FS Database Surface Sample
- Navigation Channel
- Willbridge Cove Project Area
- Kinder Morgan 2011 Dredge Prism
- Chevron 2011 Dredge Prisms
- McCall 2020-2021 Dredge Prism
- Tax Lots (Metro RLIS)

Bathymetric Survey (ft) (NAVD88)

High : 14.56 (Shallow)

Low : -76.48 (Deep)

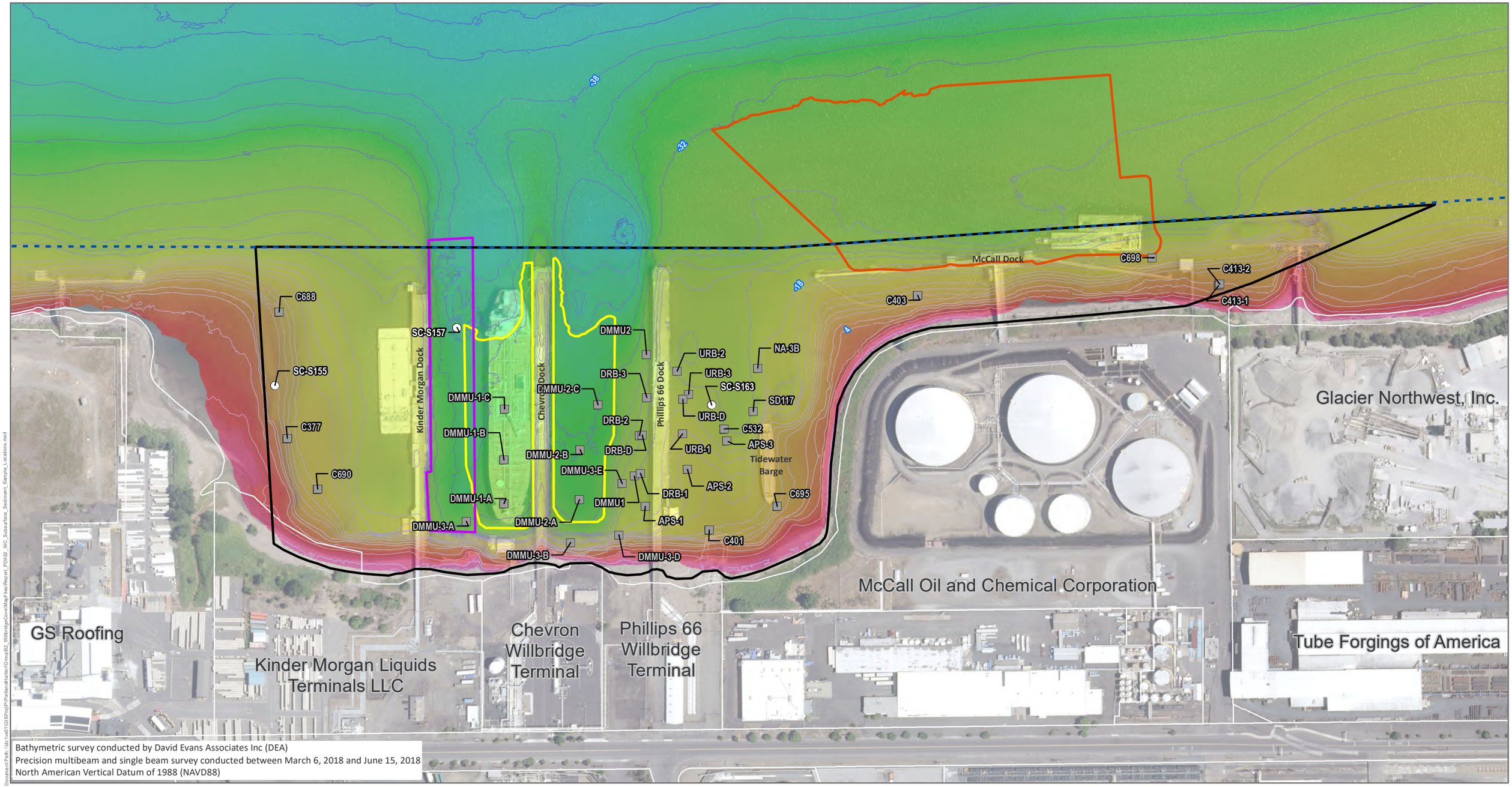
Bathymetry (2ft) Contours

Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

0 200 400 Feet

FIGURE 2-4
SURFACE SEDIMENT SAMPLE LOCATIONS
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

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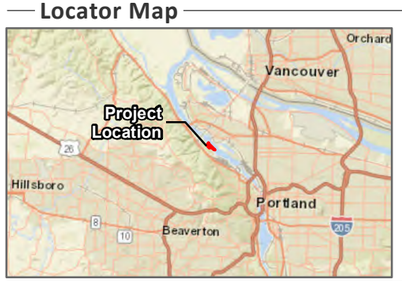
Locator Map



Legend

- PDI-BL (Pre-RD) Subsurface Sample
- FS Database Subsurface Sample
- Navigation Channel
- Willbridge Cove Project Area
- Kinder Morgan 2011 Dredge Prism
- Chevron 2011 Dredge Prisms
- McCall 2020-2021 Dredge Prism
- Tax Lots (Metro RLIS)

Bathymetric Survey (ft) (NAVD88)

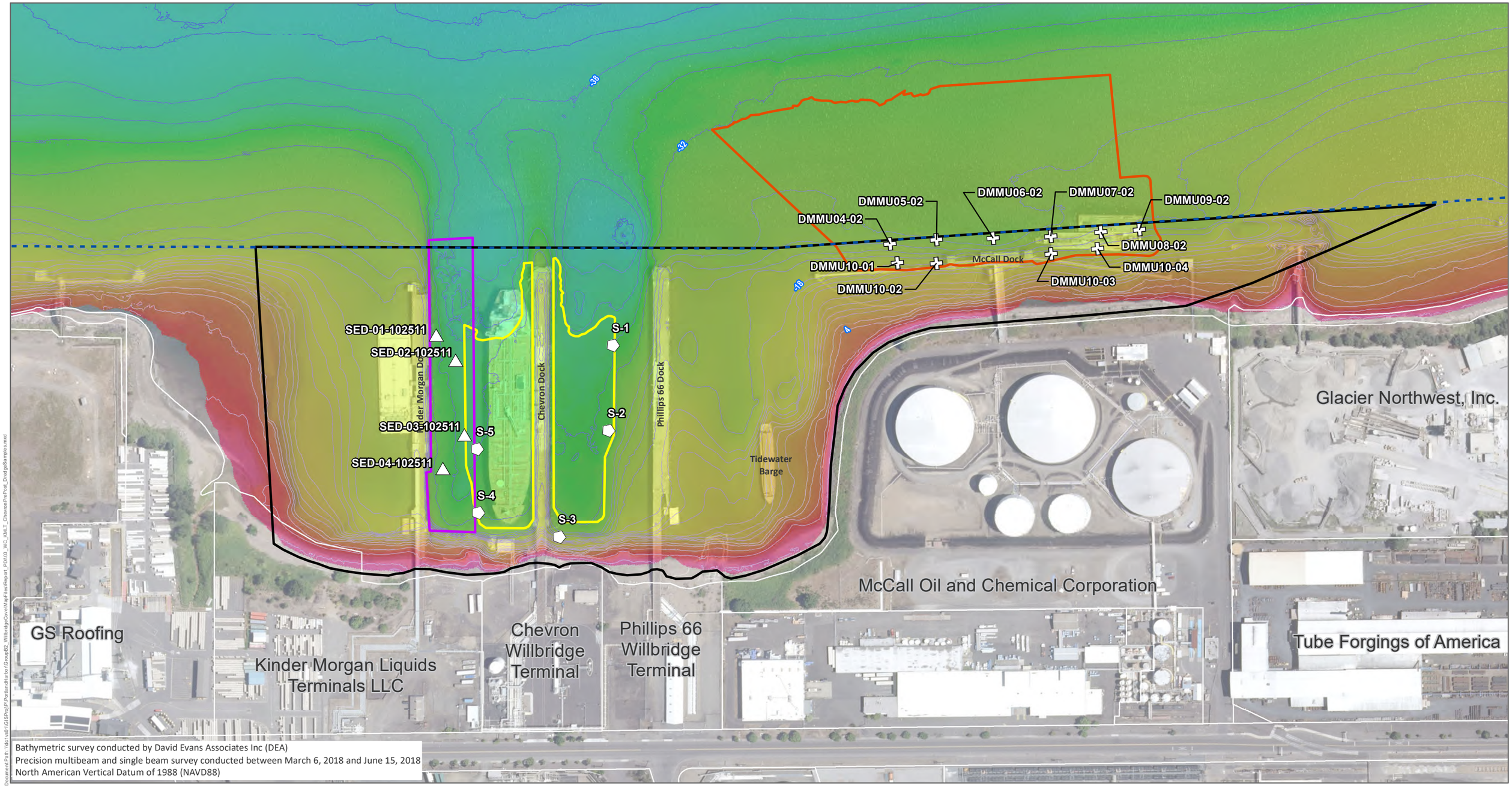
- High : 14.56 (Shallow)
- Low : -76.48 (Deep)
- Bathymetry (2ft) Contours

Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

0 200 400 Feet

FIGURE 2-5
SUBSURFACE SEDIMENT SAMPLE LOCATIONS
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

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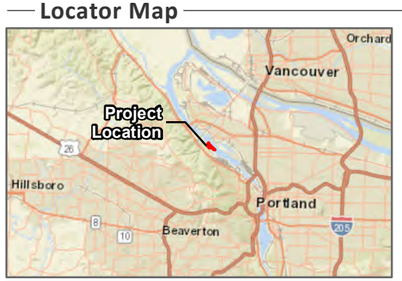
Locator Map



Legend

- Navigation Channel
- Willbridge Cove Project Area
- Tax Lots (Metro RLIS)
- 2011 KMLT Sample Location
- 2011 Chevron Sample Location
- 2019 McCall Sample Location
- Kinder Morgan 2011 Dredge Prism
- Chevron 2011 Dredge Prisms
- McCall 2020-2021 Dredge Prism

Bathymetric Survey (ft) (NAVD88)

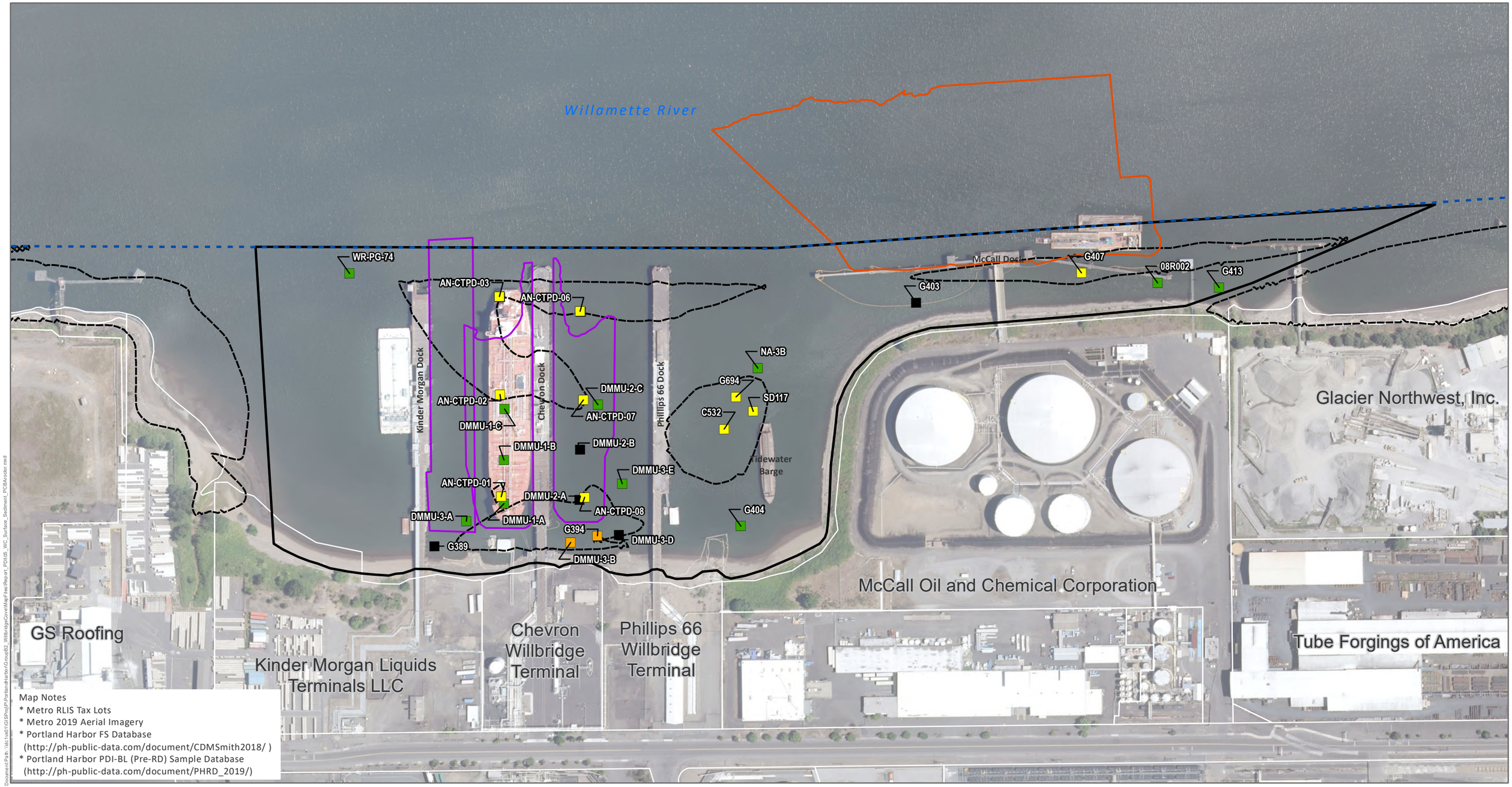
- High : 14.56 (Shallow)
- Low : -76.48 (Deep)
- Bathymetry (2ft) Contours

Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery

0 200 400 Feet

FIGURE 2-6
KMLT, CHEVRON, AND MCCALL SAMPLES AT AUTHORIZED DREDGE DEPTH
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

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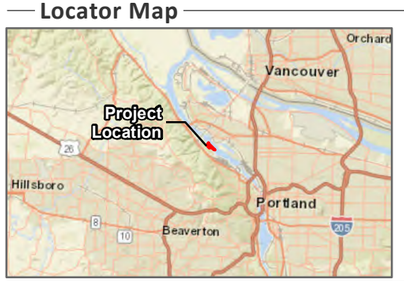
Locator Map



Legend

PCB Aroclors µg/kg
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

- Navigation Channel
- Willbridge Cove Project Area
- 2011 Dredge Prism
- 2020-2021 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

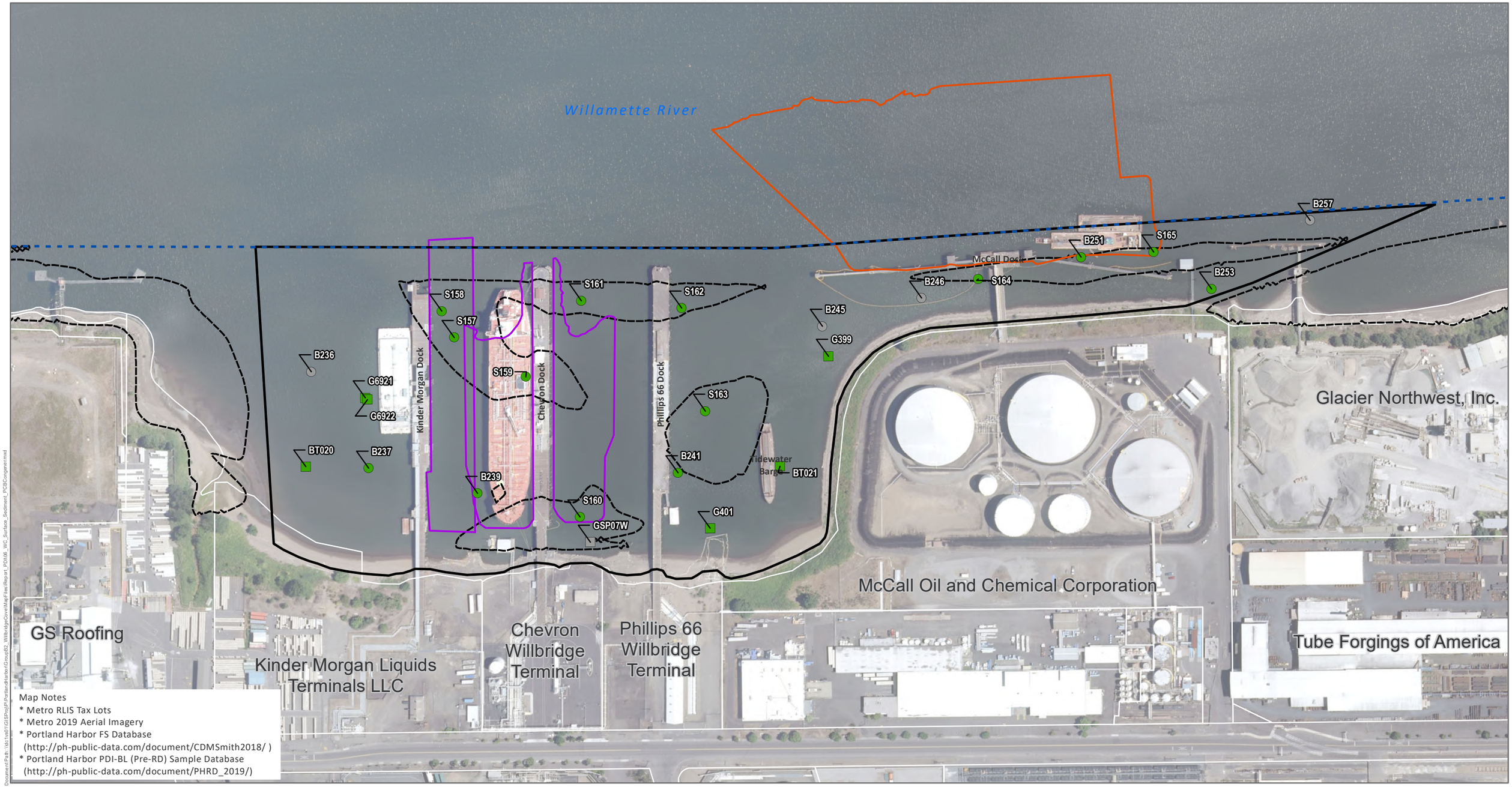
- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 200 400 Feet

FIGURE 2-7
SURFACE SEDIMENT TOTAL PCB AROCLOR CONCENTRATIONS
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

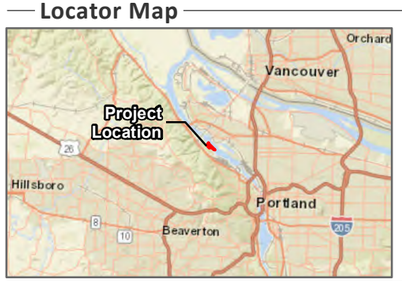
Jacobs



Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

Document Path: \\dc1\vol1\GIS\Proj\PHPortlandHarbor\Group82_WillbridgeCove\MapFiles\Report_PDI\09_WC_Surface_Sediment_PCBCongener.mxd

Locator Map



Legend

PCB Congener µg/kg
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

- Navigation Channel
- Willbridge Cove Project Area
- 2011 Dredge Prism
- 2020-2021 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

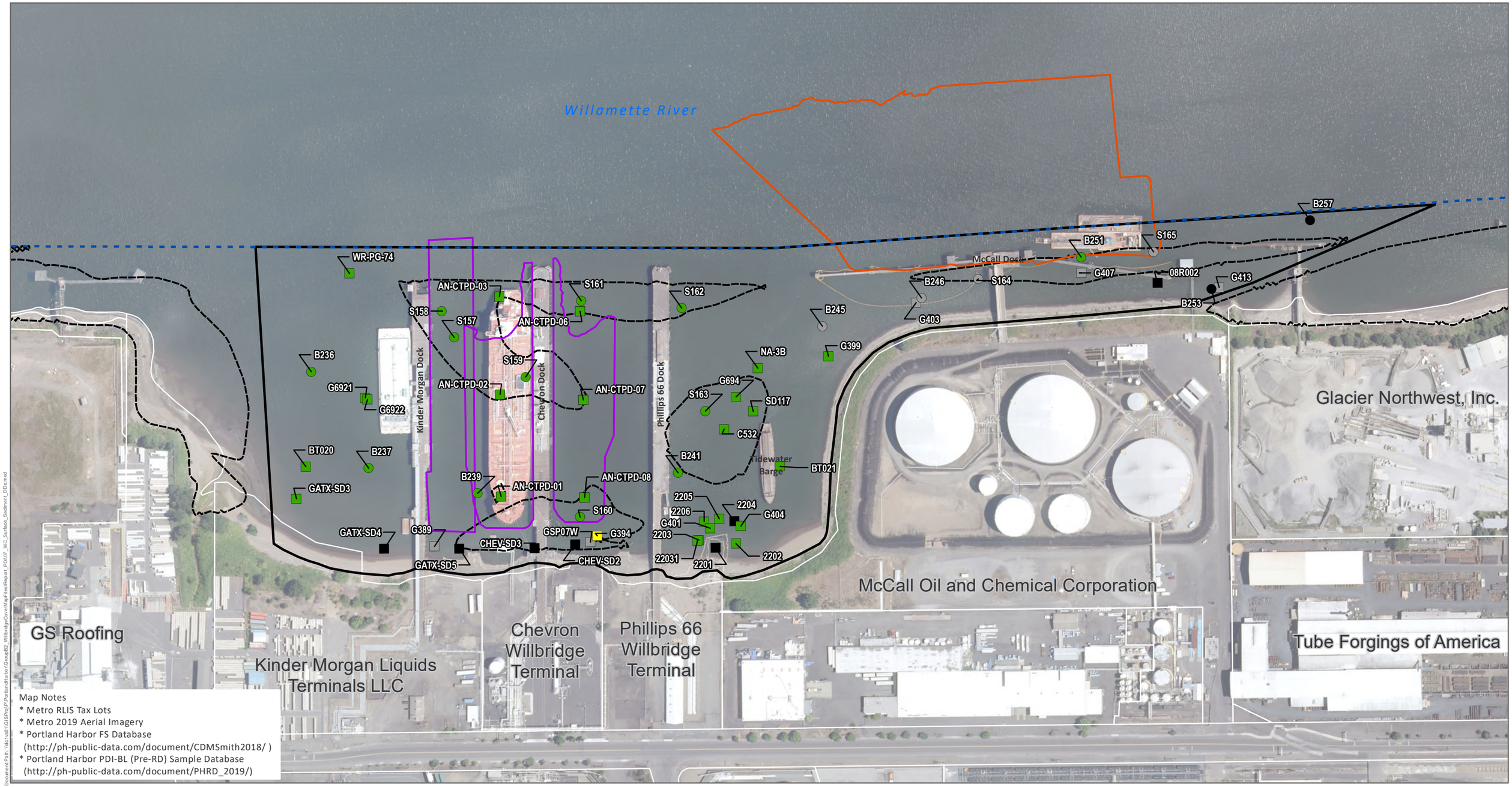
- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 200 400 Feet

FIGURE 2-8
SURFACE SEDIMENT TOTAL PCB CONGENER CONCENTRATIONS
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

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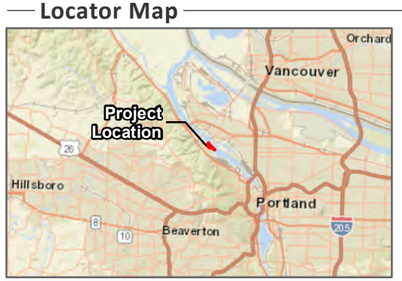
Locator Map



Legend

Total DDx µg/kg
- <6.1 (less than CUL)
- 6.1 to 160 (CUL to Sitewide RAL)
- 160 - 650 (Sitewide RAL to NAV RAL)
- Non-Detect

- Navigation Channel
- Willbridge Cove Project Area
- 2011 Dredge Prism
- 2020-2021 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 200 400 Feet

FIGURE 2-9
SURFACE SEDIMENT DDX CONCENTRATIONS
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

Willamette River

WR-PG-74
SD110
AN-CTPD-03
S161
SD105
LPSG-S-010-R-1
S162
SD113
SD115
S158
AN-CTPD-06
LPSG-S-012-R-1
S157
LPSG-S-008-R-1
B236
S159
G6921
G6922
AN-CTPD-02
AN-CTPD-07
SD104
Kinder Morgan Dock
Chevron Dock
Phillips 66 Dock
B237
BT020
AN-CTPD-01
SD107
B239
S160
AN-CTPD-08
W09CPG
GATX-SD3
G394
G401
LPSG-S-011-R-1
GSP07W
W09APG
22031
LPSG-S-009-R-1
LPSG-S-005-R-1
GATX-SD4
G389
CHEV-SD3
TOS-SD1
SD114
GATX-SD5
CHEV-SD2
LPSG-S-006-R-1
CHEV-SD1
TOS-SD2
2203
2201
TOS-SD4
B241
BT021
TOS-SD3
2205
2204
2206
TOS-SD5
G404
2202
SD118
B245
G399
NA-3B
LPSG-S-003-R-1
G694
S163
SD117
LPSG-S-004-R-1
C532
B257
S165
B251
LPSG-S-002-R-1
McCall Dock
SD123
LPSG-S-001-R-1
B246
S164
G407
08R002
B253
G413
G403

Glacier Northwest, Inc.

GS Roofing

Kinder Morgan Liquids Terminals LLC

Chevron Willbridge Terminal

Phillips 66 Willbridge Terminal

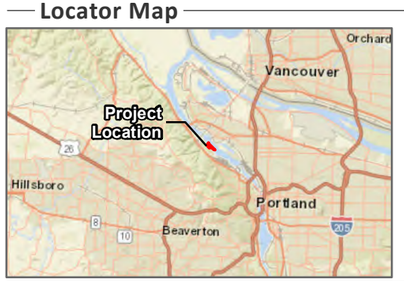
McCall Oil and Chemical Corporation

Tube Forgings of America

Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

Document Path: \\dc1vdi11\GIS\Proj\PH\PortlandHarbor\Group\B2_WillbridgeCove\MapFiles\Report_PDI\08_WC_Surface_Sediment_PAH.mxd

Locator Map



Legend

Total PAH µg/kg
- <23,000 (less than CUL)
- 23,000 to 30,000 (CUL to Sitewide RAL)
- Non-Detect

- Navigation Channel
- Willbridge Cove Project Area
- 2011 Dredge Prism
- 2020-2021 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

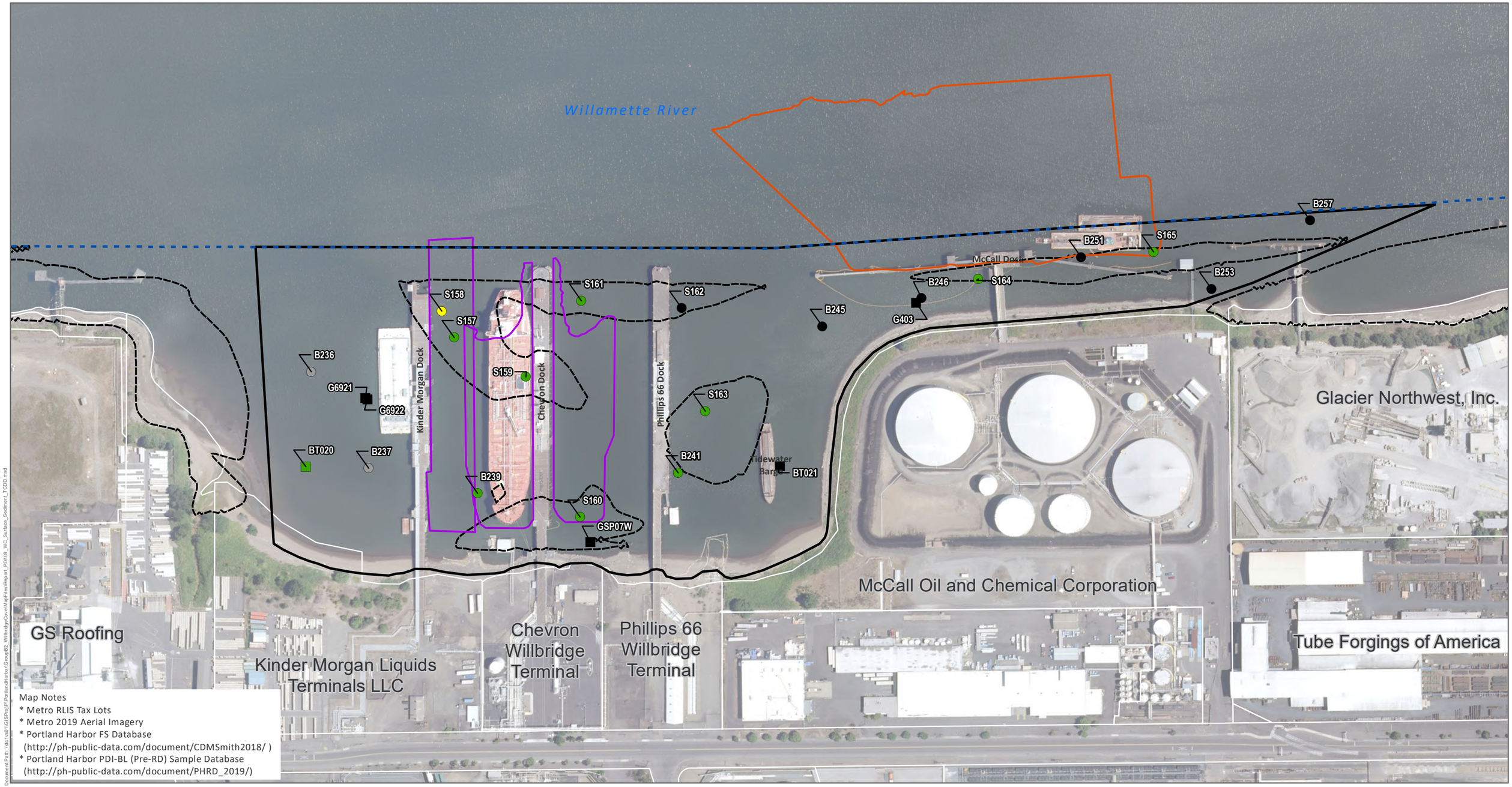
- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 200 400 Feet

FIGURE 2-10
SURFACE SEDIMENT TOTAL PAH CONCENTRATIONS
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

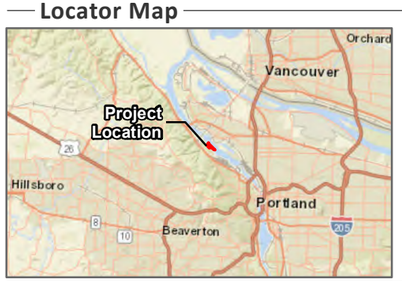
Jacobs



Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

Document Path: \\dc1\vol1\GIS\Proj\PHPortlandHarbor\Group82_WillbridgeCove\MapFiles\Report_PDI\09_WC_Surface_Sediment_TCDD.mxd

Locator Map



Legend

2,3,7,8-TCDD µg/kg
- <0.0002 (less than CUL)
- 0.0002 to 0.0006 (CUL to Sitewide RAL)
- 0.0006 to 0.002 (Sitewide RAL to NAV RAL)
- Non-Detect

- Navigation Channel
- Willbridge Cove Project Area
- 2011 Dredge Prism
- 2020-2021 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

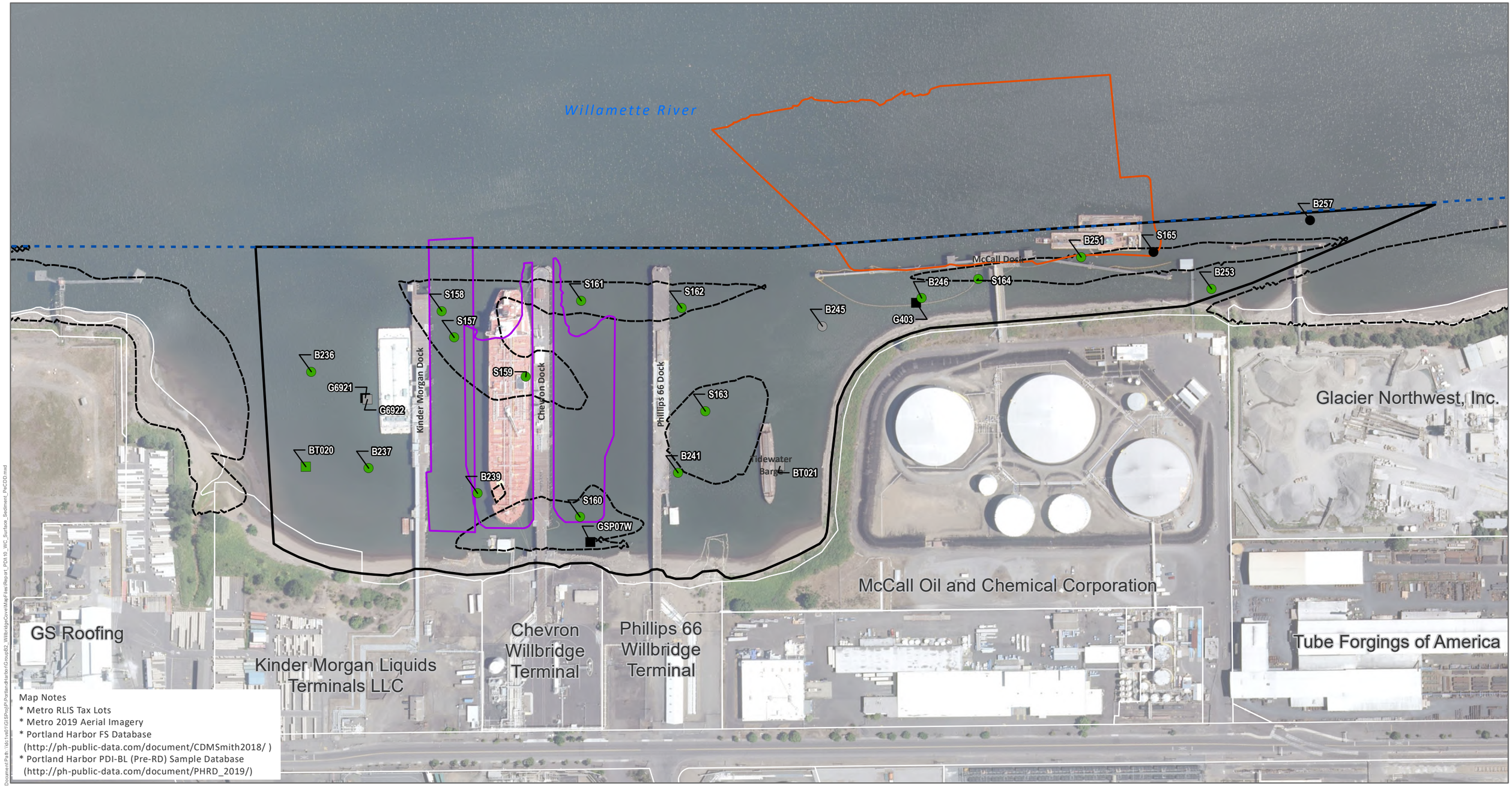
- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 200 400 Feet

FIGURE 2-11
SURFACE SEDIMENT 2,3,7,8-TCDD CONCENTRATIONS
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

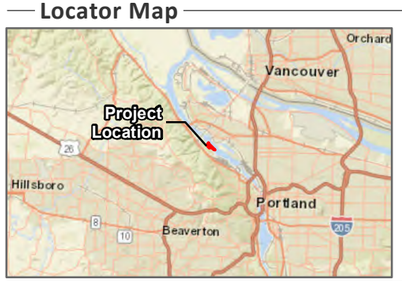
Jacobs



Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

Document Path: \\dc1\vol1\GIS\Proj\PortlandHarbor\Group82_WillbridgeCove\MapFiles\Report_PDI-10_WC_Surface_Sediment_PeCDD.mxd

Locator Map



Legend

1,2,3,7,8-PECD µg/kg
- <0.0002 (less than CUL)
- 0.0002 to 0.0008 (CUL to Sitewide RAL)
- Non-Detect

- Navigation Channel
- Willbridge Cove Project Area
- 2011 Dredge Prism
- 2020-2021 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

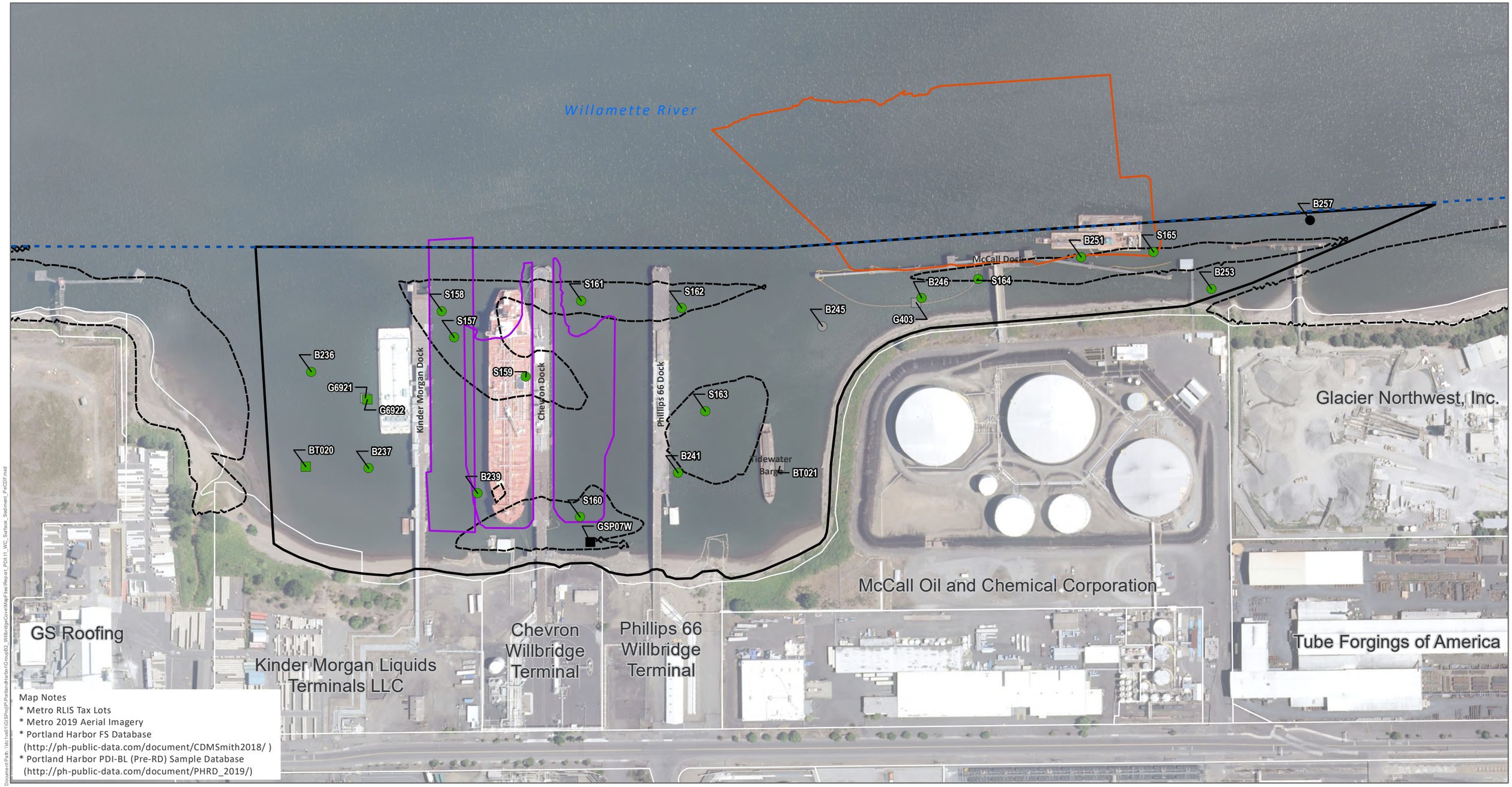
CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 200 400 Feet

FIGURE 2-12
SURFACE SEDIMENT 1,2,3,7,8-PECDD CONCENTRATIONS

Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

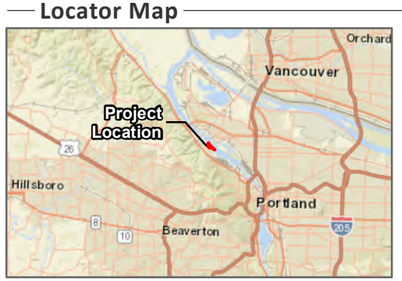
Jacobs



Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

Document Path: \\dc1\vol1\GIS\Proj\PHPortlandHarbor\Group82_WillbridgeCove\MapFiles\Report_P0611_WC_Surface_Sediment_PreCDF.mxd

Locator Map



Legend

2,3,4,7,8-PECDF µg/kg
- < 0.0003 (less than CUL)
- 0.0003 to 0.2 (CUL to Sitewide RAL/PTW)
- Non-Detect

- Navigation Channel
- Willbridge Cove Project Area
- 2011 Dredge Prism
- 2020-2021 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

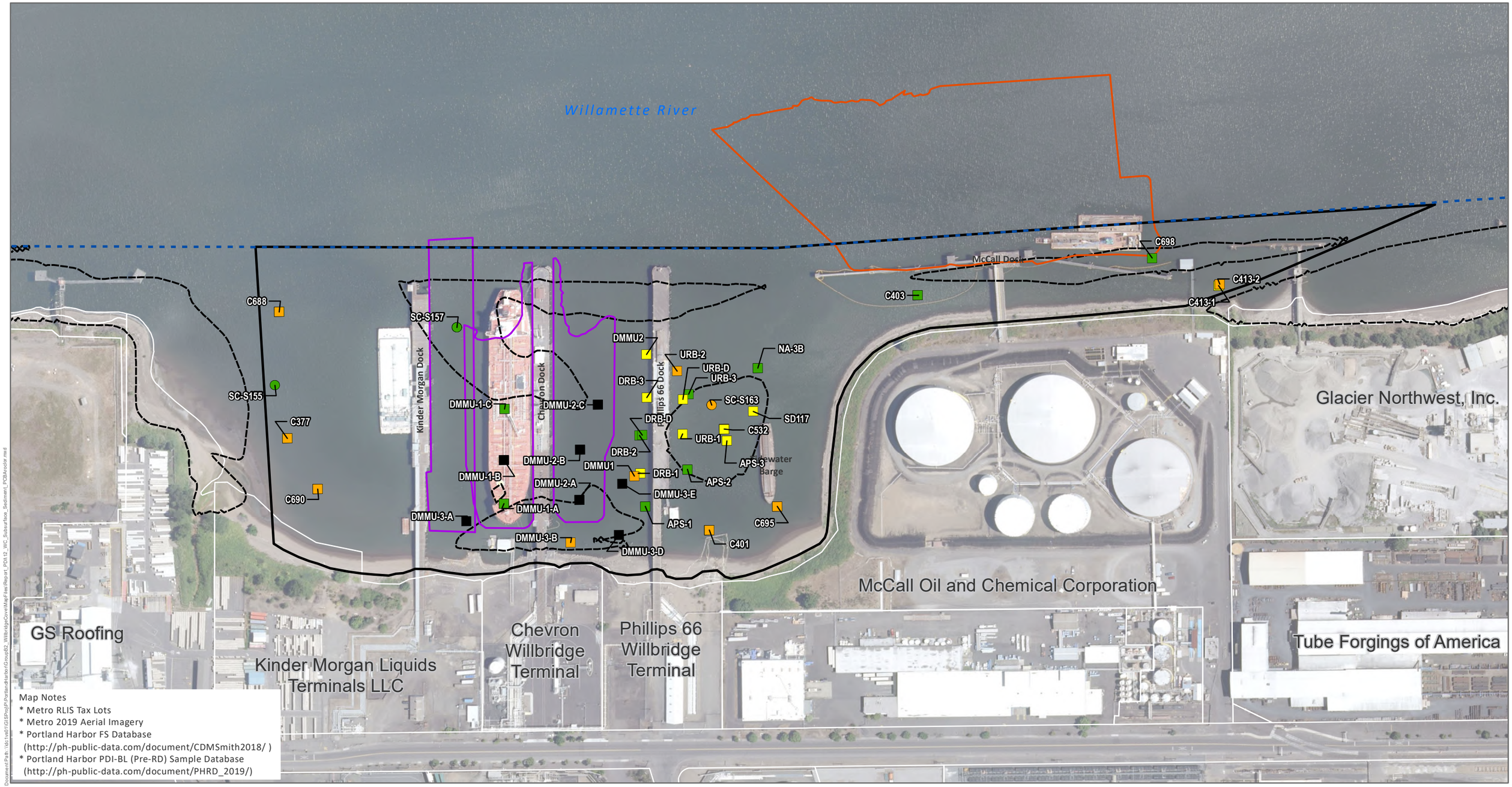
- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 200 400 Feet

FIGURE 2-13
SURFACE SEDIMENT 2,3,4,7,8-PECDF CONCENTRATIONS
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

Document Path: \\dc1vs01\GISProj\P\PortlandHarbor\Group82_WillbridgeCove\MapFiles\Report_PDI12_WC_Subsurface_Sediment_PCBAroclor.mxd

Locator Map

Legend

Total PCBs µg/kg

- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

- Navigation Channel
- Willbridge Cove Project Area
- 2011 Dredge Prism
- 2020-2021 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

Where aroclor and congener data avaiable, congener results presented.
Congener data presented for locations C377, C401, C532, C688, and C690.

0 200 400 Feet

FIGURE 2-14
SUBSURFACE SEDIMENT TOTAL PCBs
MAXIMUM CONCENTRATIONS IN CORES
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

C688
SC-S155
C377
C690
SC-S157
Kinder Morgan Dock
DMMU-1-C
DMMU-2-C
DMMU-2-B
DMMU-1-B
DMMU-1-A
DMMU-3-A
DMMU-2-A
DMMU-3-B
Chevron Dock
DMMU2
DMMU1
DMMU-3-E
DMMU-3-D
URB-2
URB-D
URB-3
DRB-3
DRB-2
DRB-D
DRB-1
Phillips 66 Dock
NA-3B
SD117
SC-S163
C532
APS-3
URB-1
APS-2
APS-1
C401
C695
Tidewater Barge
C403
McCall Dock
C698
C413-2
C413-1
Glacier Northwest, Inc.
GS Roofing
Kinder Morgan Liquids Terminals LLC
Chevron Willbridge Terminal
Phillips 66 Willbridge Terminal
McCall Oil and Chemical Corporation
Tube Forgings of America

Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

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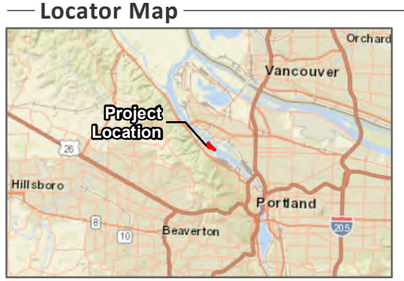
Locator Map



Legend

DDx µg/kg
- <6.1 (less than CUL)
- 6.1 to 160 (CUL to Sitewide RAL)
- 160 - 650 (Sitewide RAL to NAV RAL)
- 650 to 7050 (NAV RAL to PTW)
- Non-Detect

- Navigation Channel
- Willbridge Cove Project Area
- 2011 Dredge Prism
- 2020-2021 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

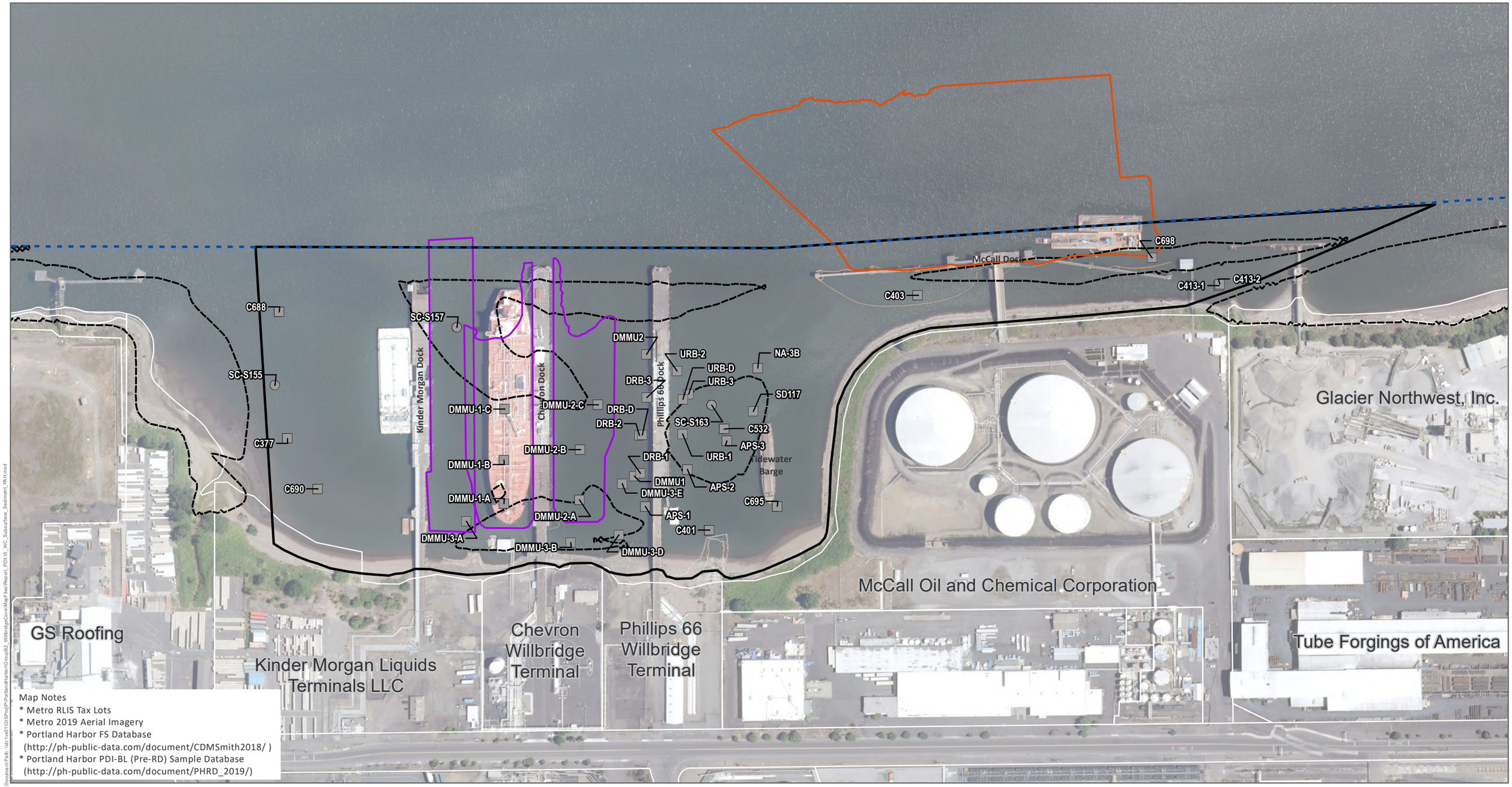
- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 200 400 Feet

FIGURE 2-15
SUBSURFACE SEDIMENT DDX
MAXIMUM CONCENTRATIONS IN CORES
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

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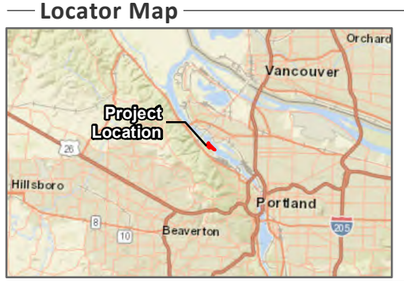
Locator Map



Legend

PAH µg/kg

- <23,000 (less than CUL)

- Navigation Channel
- Willbridge Cove Project Area
- 2011 Dredge Prism
- 2020-2021 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

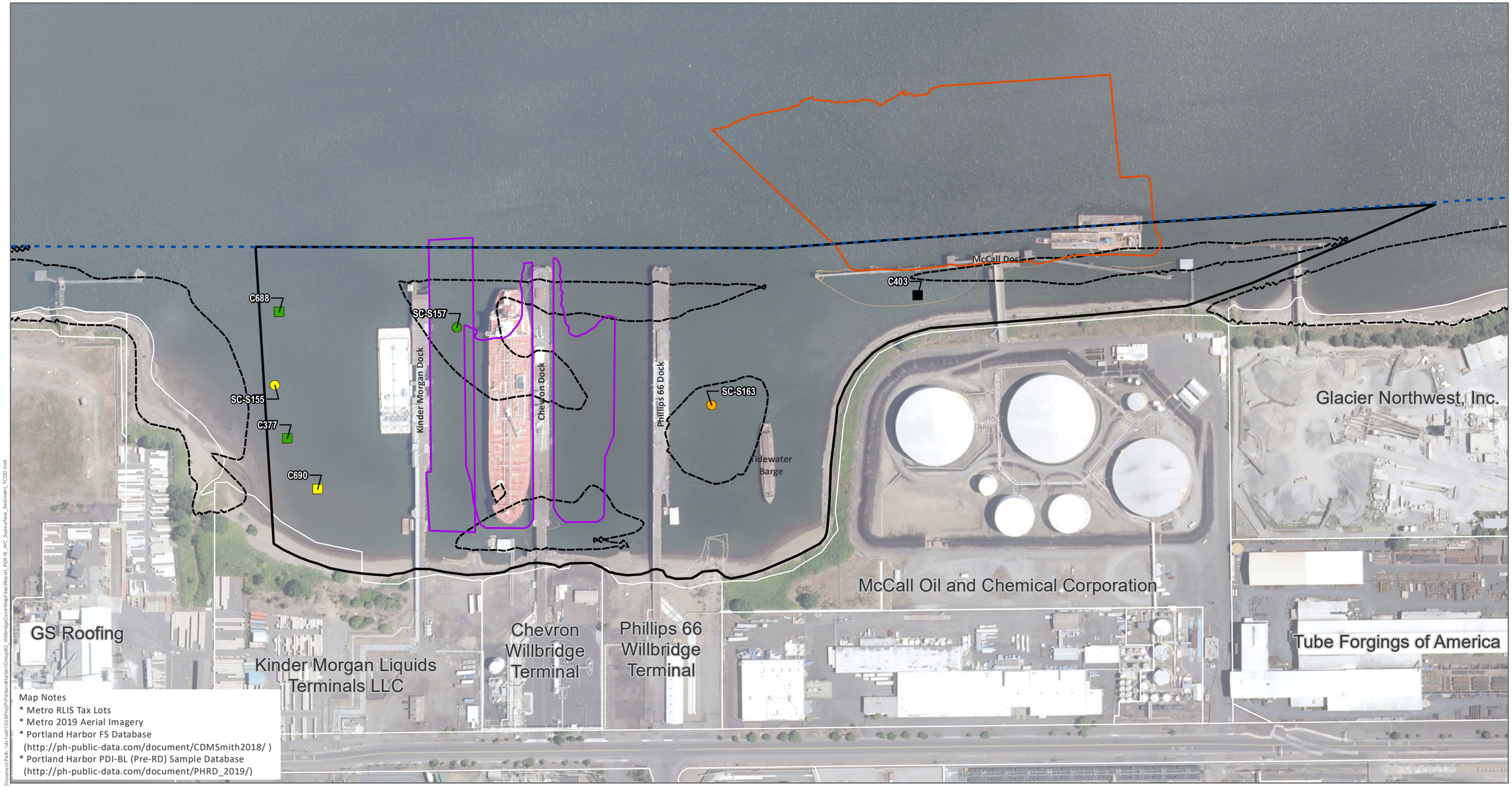
- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 200 400 Feet

FIGURE 2-16
SUBSURFACE SEDIMENT TOTAL PAH MAXIMUM CONCENTRATIONS IN CORES
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

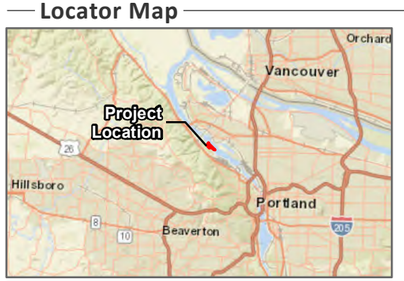
Jacobs



Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

Document Path: \\dc1\vol1\GIS\Proj\PortlandHarbor\Group82_WillbridgeCove\MapFiles\Report_PDI-18_WC_Subsurface_Sediment_TCDD.mxd

Locator Map



Legend

2,3,7,8-TCDD µg/kg
- <0.0002 (less than CUL)
- 0.0002 to 0.0006 (CUL to Sitewide RAL)
- 0.0006 to 0.002 (Sitewide RAL to NAV RAL)
- 0.002 to 0.01 (NAV RAL to PTW)
- Non-Detect

- Navigation Channel
- Willbridge Cove Project Area
- 2011 Dredge Prism
- 2020-2021 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

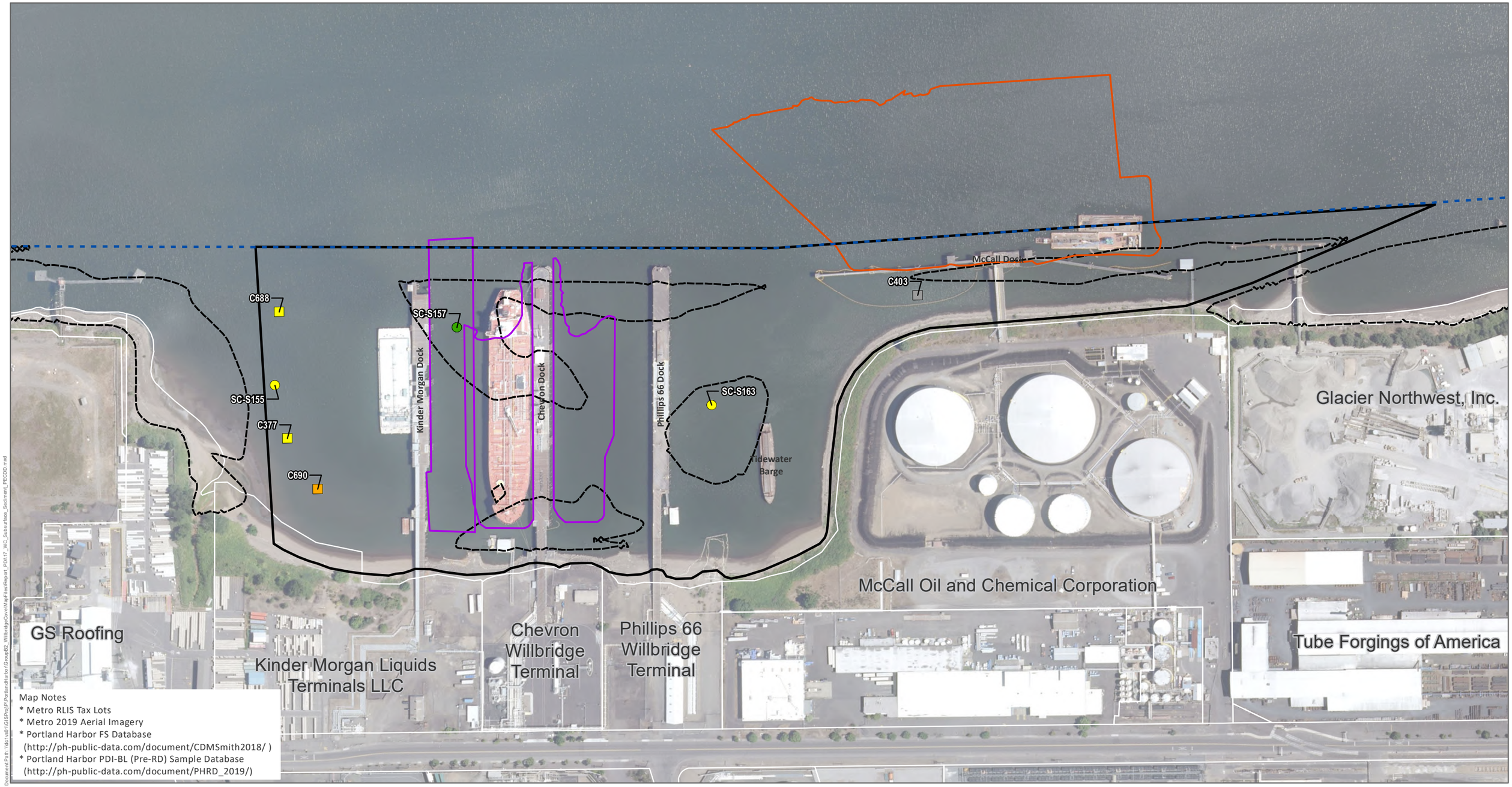
- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 200 400 Feet

FIGURE 2-17
SUBSURFACE SEDIMENT 2,3,7,8-TCDD MAXIMUM CONCENTRATIONS IN CORES
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

Document Path: \\dc1vsh11\GIS\Proj\PH\PortlandHarbor\Group82_WillbridgeCove\MapFiles\Report_PDI\17_WC_Subsurface_Sediment_PECDD.mxd

Locator Map

Legend

1,2,3,7,8-PECD µg/kg
- <0.0002 (less than CUL)
- 0.0002 to 0.0008 (CUL to Sitewide RAL)
- 0.0008 to 0.003 (Sitewide RAL to NAV RAL)
- 0.003 to 0.01 (NAV RAL to PTW)
- Non-Detect

- Navigation Channel
- Willbridge Cove Project Area
- 2011 Dredge Prism
- 2020-2021 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 200 400 Feet

FIGURE 2-18
SUBSURFACE SEDIMENT 1,2,3,7,8-PECDD MAXIMUM CONCENTRATIONS IN CORES
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

C688
SC-S157
SC-S155
C377
C690
Kinder Morgan Dock
Chevron Dock
Phillips 66 Dock
SC-S163
Tidewater Barge
C403
McCall Dock
Glacier Northwest, Inc.
GS Roofing
Kinder Morgan Liquids Terminals LLC
Chevron Willbridge Terminal
Phillips 66 Willbridge Terminal
McCall Oil and Chemical Corporation
Tube Forgings of America

Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

Document Path: \\dc1vdi11\GIS\Proj\PH\PortlandHarbor\Group82_WillbridgeCove\MapFiles\Report_PDI-19_WC_Subsurface_Sediment_PECDF.mxd

Locator Map

Legend

2,3,4,7,8-PECDF µg/kg
- < 0.0003 (less than CUL)
- 0.0003 to 0.2 (CUL to Sitewide RAL/PTW)
- Non-Detect

- Navigation Channel
- Willbridge Cove Project Area
- 2011 Dredge Prism
- 2020-2021 Dredge Pris
- Sediment Management Area (ROD ESD 2018)

- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 200 400 Feet

FIGURE 2-19
SUBSURFACE SEDIMENT 2,3,4,7,8-PECDF MAXIMUM CONCENTRATIONS IN CORES
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

SALTZMAN CREEK
DISCHARGE POINT

kinder Morgan Dock

Chevron Dock

Phillips 66 Dock

Tidewater
Spud Barge

KM-S-4
KM-S-3
S-2
S-1
SB-S1
KM-S-5
S-3
S-4
S-5
S-6
S-7
S-8
S-9
S-10
SB-N1
SB-N6
SB-S4
SB-N5
SB-N4

Kinder Morgan Liquids
Terminals LLC

PIPELINE

Chevron
Willbridge
Terminal

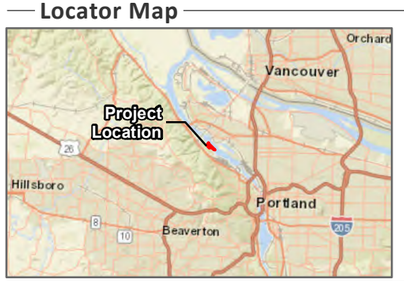
Phillips 66
Willbridge
Terminal

NW Front Ave

OUTFALL #1

Document Path: \\dc1vs01\GIS\proj\PortlandHarbor\Group\B2_WillbridgeCove\MapFiles\Report_PDI\45_WC_BankSampleLocations.mxd

Locator Map



Legend

- Surface Soil Sample (2008)
- KMLT Surface Sample Locations (2012)
- KMLT Soil Boring/LIF Locations (2018)
- Top of Bank
- Tax Lots (Metro RLIS)
- Chevron Willbridge Terminal
- Kinder Morgan Liquids Terminal LLC
- Phillips 66 Willbridge Terminal

Map Notes
* Metro RLIS Tax Lots
* Metro 2018 Aerial Imagery

0 137.5 275 Feet

FIGURE 2-20

KINDER MORGAN, CHEVRON, AND PHILLPS 66
BANK SOIL SAMPLING LOCATIONS

Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

McCALL DOCK

Riverbank Composite 1

Riverbank Composite 2

Riverbank Composite 3

GP-17

GP-18

GP-19

GP-16

GP-15

GP-14

CHEVRON DOCK

PHILLIPS 66 DOCK

Tidewater Spud Barge

McCALL

HIGH PURITY PRODUCTS

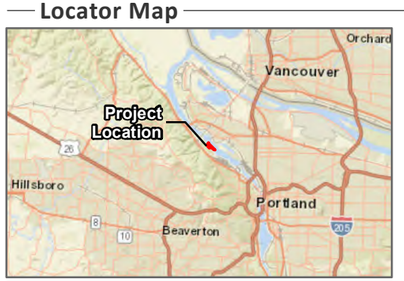
BRENNTAG PACIFIC

ASPHALT PLANT

NW Front Ave

Document Path: \\dc1vs01\GIS\Proj\Portland Harbor\Group\B2_Willbridge Cove\MapFiles\Report_PDI\44_WC_McCallBankSampleLocations.mxd

Locator Map



Legend

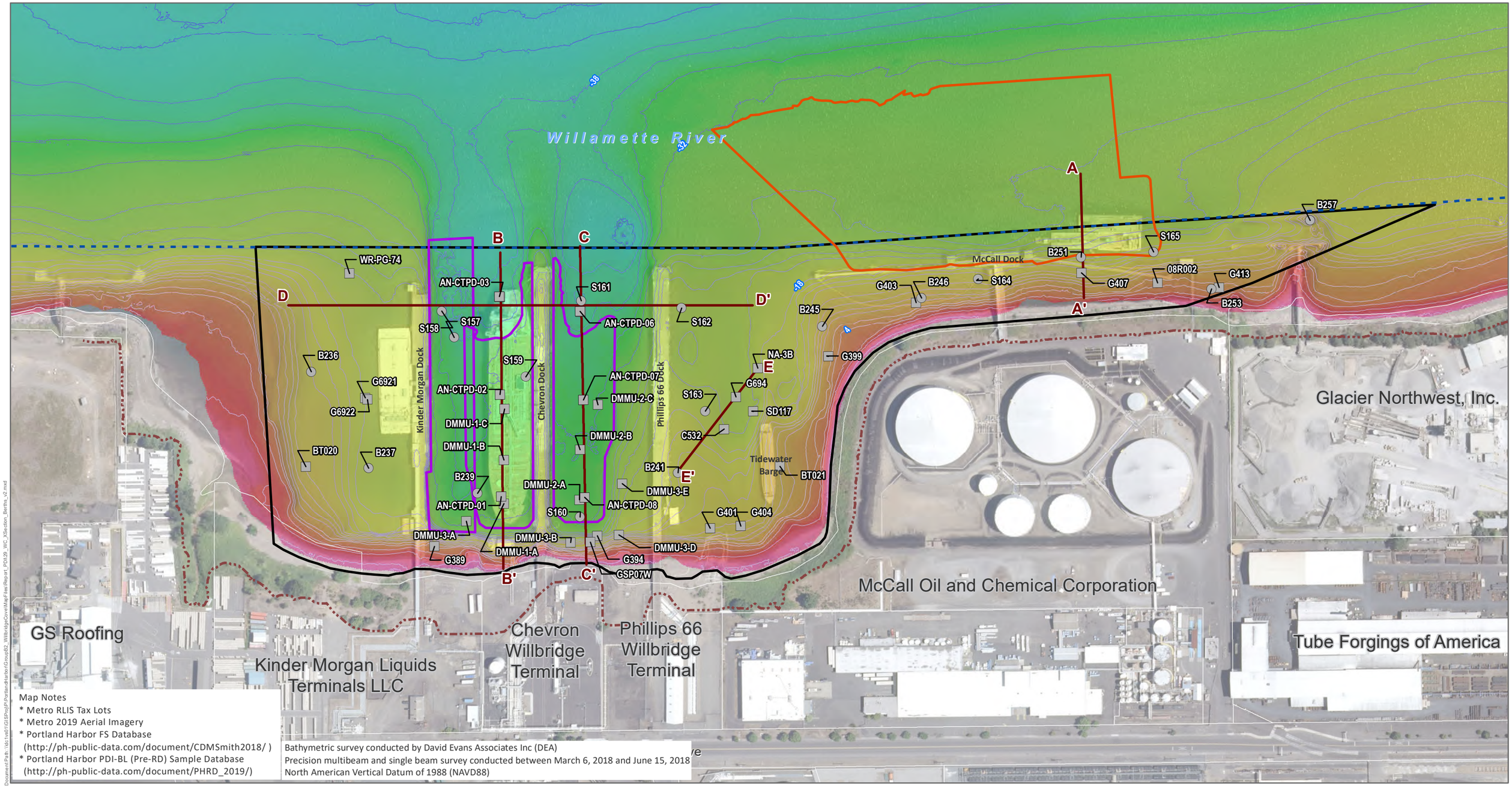
- McCall Oil Riverbank Samples (2000)
- Top of Bank
- Composite Soil Sample Location (2010)
- Tax Lots (Metro RLIS)
- McCall Terminal

Map Notes
* Metro RLIS Tax Lots (Tax Lots Identification TLID)
* Metro 2018 Aerial Imagery

0 200 400 Feet

FIGURE 2-21
McCALL PROPERTY
BANK SOIL SAMPLING LOCATIONS
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

Document Path: \\dc1vs01\GISProj\PortlandHarbor\Group\B2_WillbridgeCove\MapFiles\Report_PDI\29_WC_XSection_Berths_v2.mxd

Locator Map

Legend

- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)
- Cross Section Line
- Top of Bank
- Bathymetry (2ft) Contours
- Navigation Channel
- Willbridge Cove Project Area
- Dredge Prism
- Proposed Dredge Prism

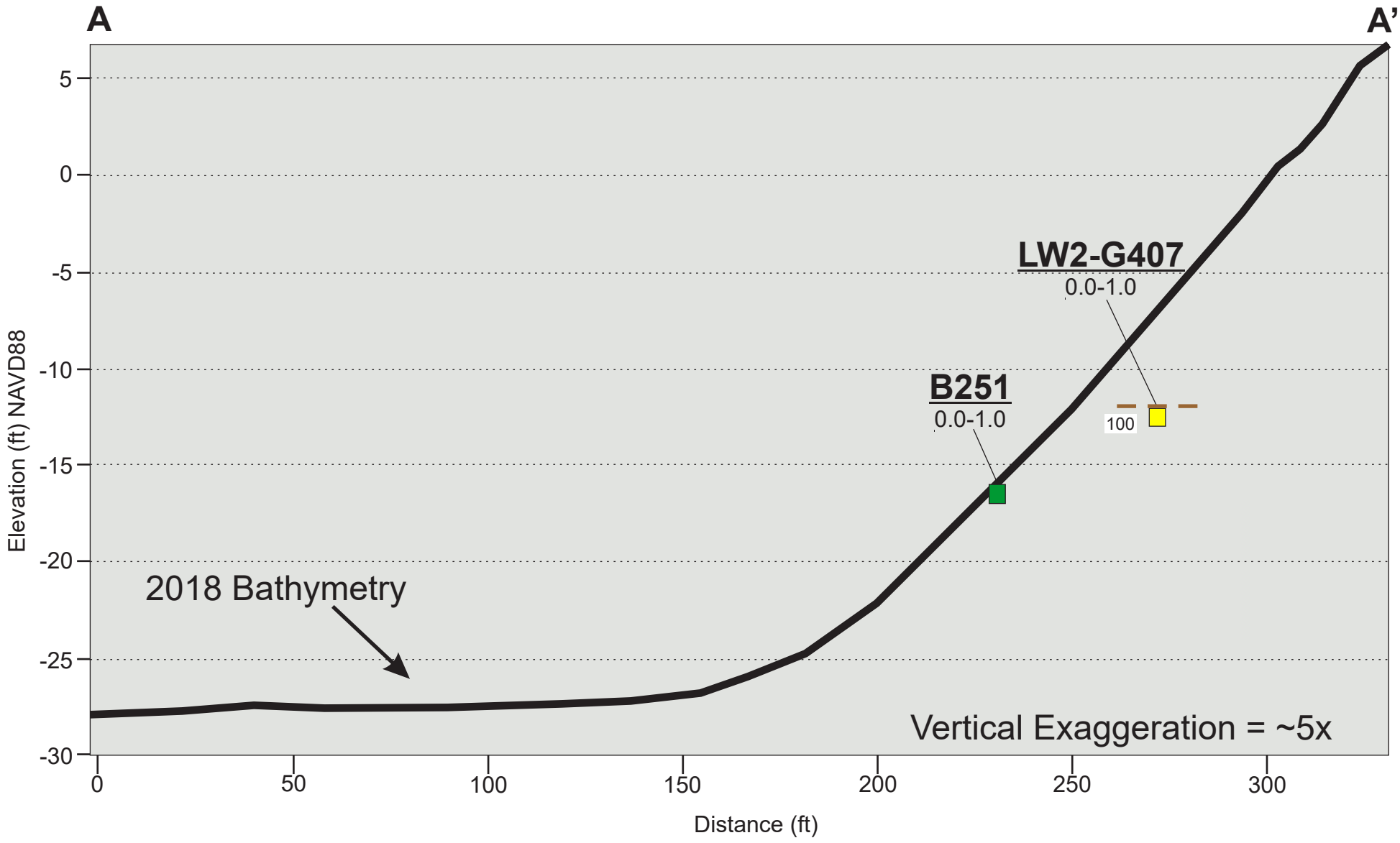
Bathymetric Survey (ft) (NAVD88)
High : 14.56 (Shallow)
Low : -76.48 (Deep)

Map Notes
* Metro RLIS Tax Lots (Tax Lots Identification TLID)
* Metro 2018 Aerial Imagery
* Base data from Portland Harbor Environmental Data Portal (http://ph-public-data.com/)

0 187.5 375 Feet

FIGURE 2-22A
CROSS-SECTION LOCATION MAP *Portland Harbor Superfund Site Willbridge Cove Project Area Portland, Oregon*

Jacobs



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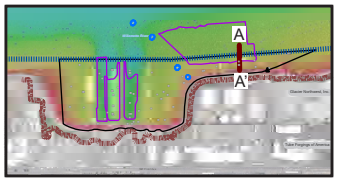


Sediment Sample - PCB µg/kg

- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)

- ~2004 Surface Elevation

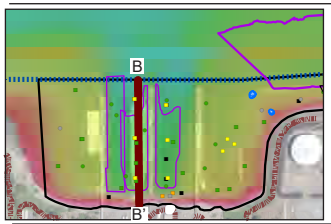
CUL - Cleanup Level
RAL - Remedial Action Level
PTW - Principal Threat Waste

Notes:

-Sample depths are listed below sample ID in ft bss.
-Sample with RAL exceedances have results listed next to samples in µg/kg.
-Inferred 2004 elevation is based on sample elevations in FS Database.

Figure 2-22B
CROSS-SECTION SHOWING PCB RESULTS ALONG A-A'
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



Legend

Sediment Sample - PCB µg/kg

- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- Non-Detect (ND)

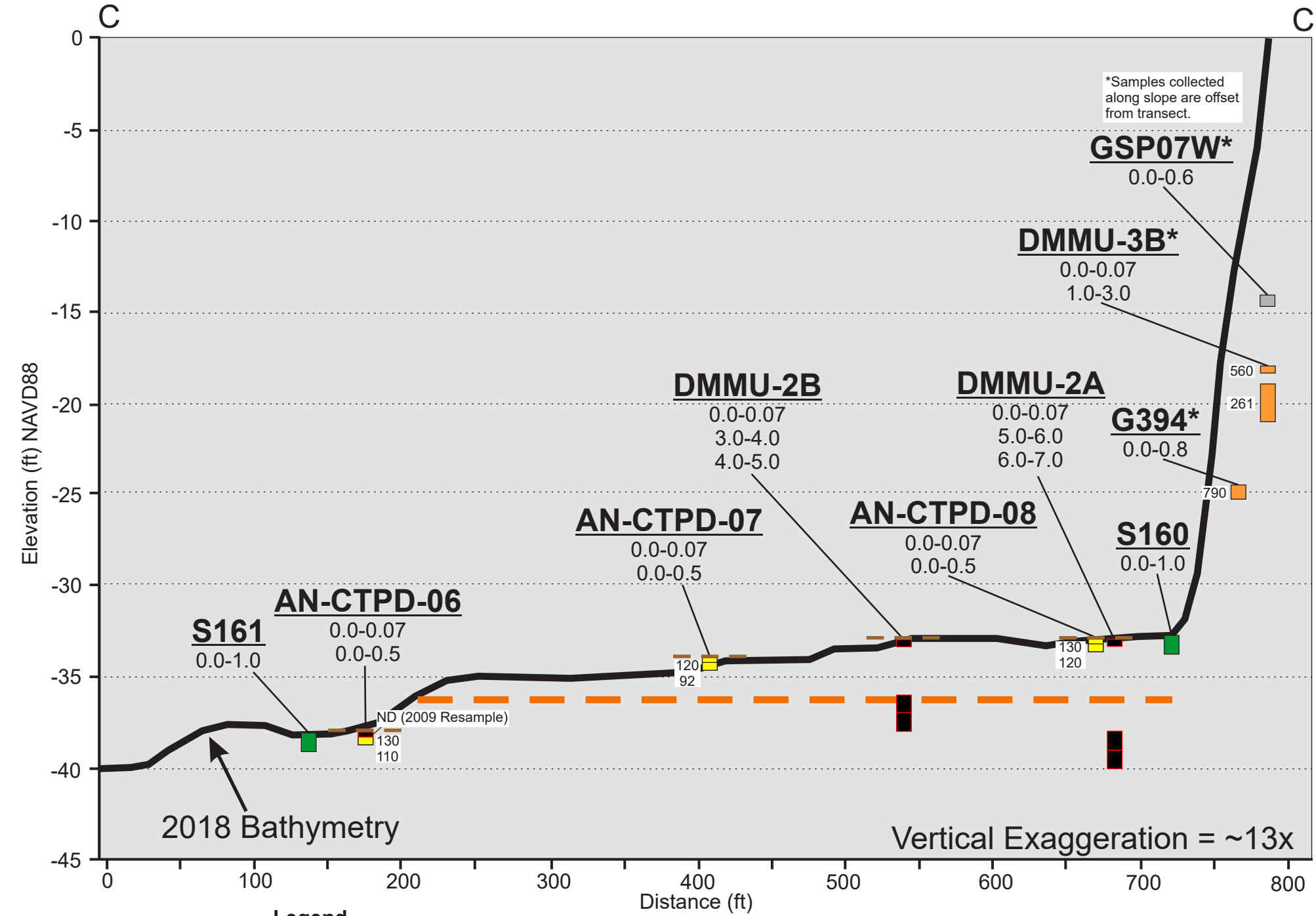
- Estimated Average Achieved Dredge Elevation (-36.2 ft bgs NAVD88)
- ~2002/2009 Surface Elevation

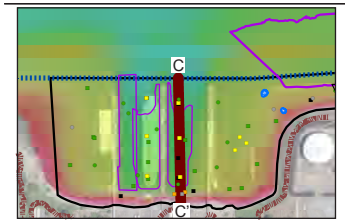
CUL - Cleanup Level
RAL - Remedial Action Level
PTW - Principal Threat Waste

Notes:
-Sample depths are listed below sample ID in ft bss.
-Sample with RAL exceedances have results listed next to samples in µg/kg.
-Estimated average provided by the Chevron Bathymetric Survey, 2012.
-Inferred 2002/2009 elevation is based on sample elevations in FS Database.

Figure 2-22C
CROSS-SECTION SHOWING PCB RESULTS ALONG B-B'
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs





Legend

Sediment Sample - PCB µg/kg

- <9 (Less than CSL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect (ND)

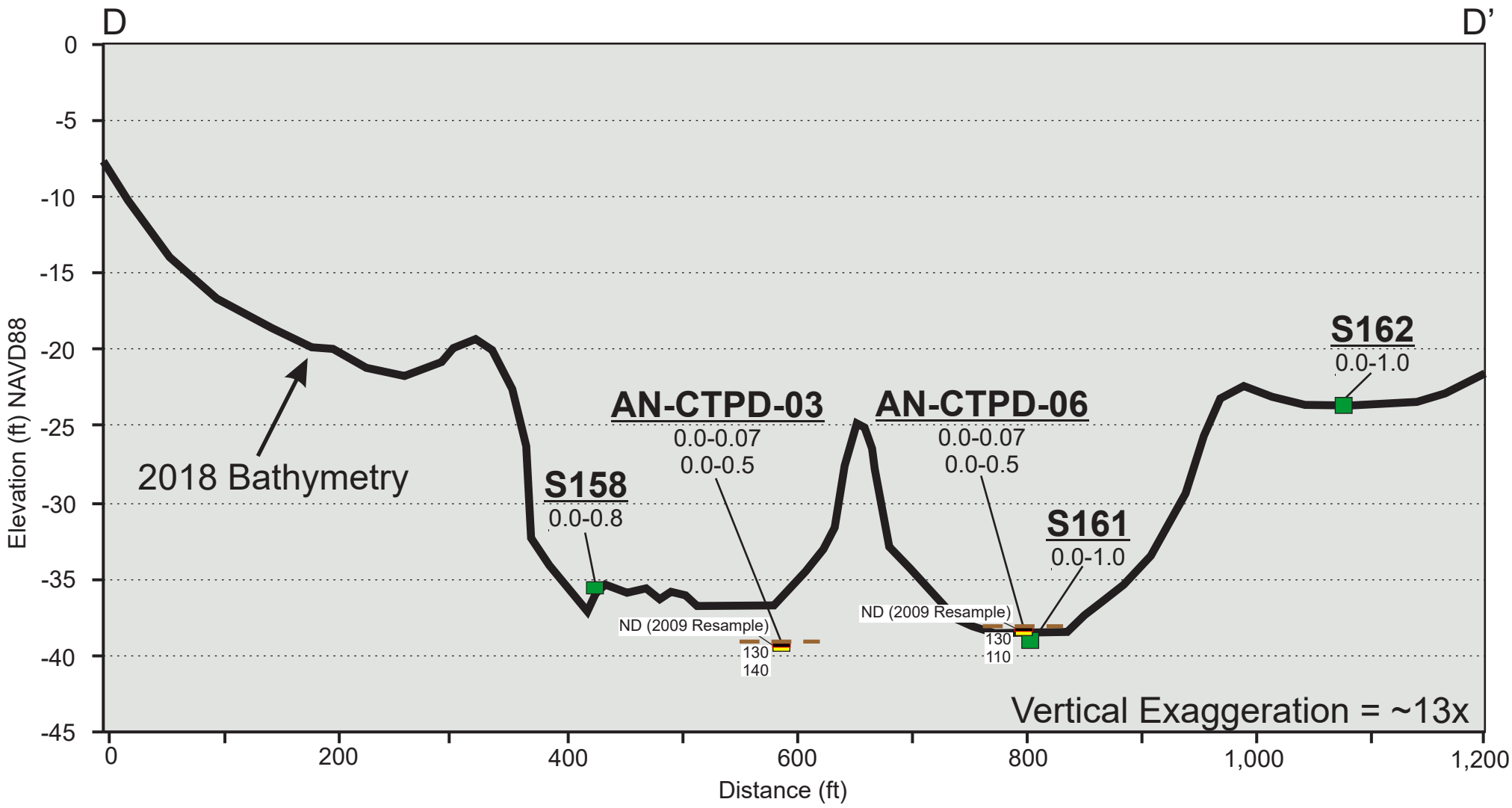
- Estimated Average Achieved Dredge Elevation (-36.2 ft bgs NAVD88)
- ~2002/2009 Surface Elevation

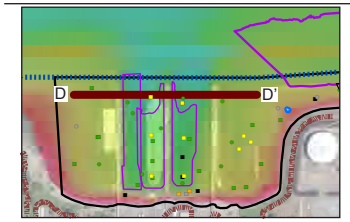
CUL - Cleanup Level
RAL- Remedial Action Level
NAV RAL - Navigation Channel RAL
PTW - Principal Threat Waste

Notes:
-Sample depths are listed below sample ID in ft bss.
-Estimated average provided by the Chevron Bathymetric Survey, 2012.
-Sample with RAL exceedances have results listed next to samples in µg/kg.
-Inferred 2002/2009 elevation is based on sample elevations in FS Database.

Figure 2-22D
CROSS-SECTION SHOWING PCB RESULTS ALONG C-C'
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs





Legend

Sediment Sample - PCB µg/kg

- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- Non-Detect (ND)

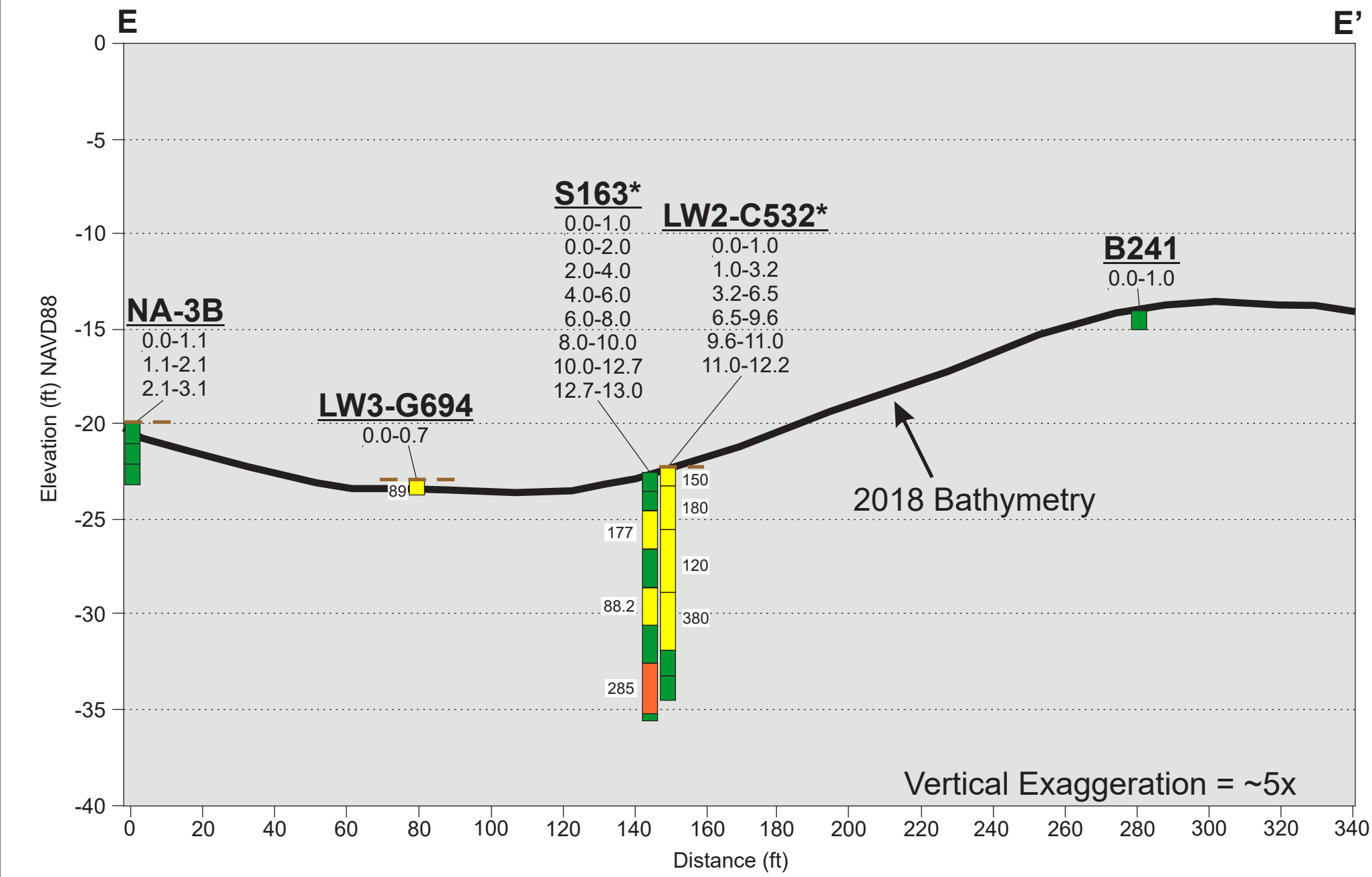
~2002/2009 Surface Elevation

CUL- Cleanup Level
RAL - Remedial Action Level
PTW - Principal Threat Waste

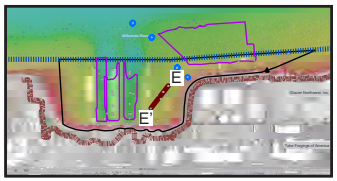
Notes:
-Sample depths are listed below sample ID in ft bss.
-Sample with RAL exceedances have results listed next to samples in µg/kg.
-Inferred 2002/2009 elevation is based on sample elevations in FS Database.

Figure 2-22E
CROSS-SECTION SHOWING PCB RESULTS ALONG D-D'
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



Legend



Sediment Sample - PCB µg/kg

- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)

~2004/2005/2007 Surface Elevation

CUL - Cleanup Level
RAL- Remedial Action Level
NAV RAL - Navigation Channel RAL
PTW - Principal Threat Waste

Notes:
*Sample offset ~25' from transect.
-Sample depths are listed below sample ID in ft bss.
-Sample with RAL exceedances have results listed next to samples in µg/kg.
-Inferred 2004/05/07 elevation is based on sample elevations in FS Database.

Figure 2-22F
CROSS-SECTION SHOWING PCB RESULTS ALONG E-E'
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

B257

Preliminary SMA 1

S165

B251

McCall Dock

C698

G407

S164

08R002

C413-1

C413-2

-18

B253

G413

C403

B246

G403

Shallow/Intermediate Boundary

-4

Location/ID	Interval (ft)	Total PCBs (µg/kg)
LW2-G407	0 - 0.95	100

Location/ID	Interval (ft)	Total PCBs (µg/kg)
G413	0 - 0.89	55.5
C413-B1	1.0 - 5.2	132
C413-C1	5.2 - 9.8	96

Location/ID	Interval (ft)	Total PCBs (µg/kg)
C413-B2 (rep)	1.0 - 5.0	310
C413-C2 (rep)	5.0 - 9.0	410
C413-D2	9.0 - 11.1	77

McCall Oil and Chemical Corporation

Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

Document Path: \\tc1vd01\GIS\Proj\P\Portland\Harbor\Group82_WillbridgeCove\MapFiles\Report_PDI\19_WC_DataGaps_Evaluation_Area1.mxd

Locator Map

Legend

PCB µg/kg - Surface Sediment
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

PCB µg/kg - Subsurface Sediment
Maximum detected concentration in core
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

- Bathymetry (2ft) Contours
- Shallow/Intermediate Boundary
- Willbridge Cove Project Area
- 2020-2021 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 100 200 Feet

FIGURE 2-23
PRELIMINARY SMA 1 EVALUATION RESULTS
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

Location/ID	Sample Interval (ft) 2002	Sample Interval (ft) 2009	Total PCBs (µg/kg)	Adjusted Sample Interval due to Deposition (ft)
AN-CTPD-03	0 - 0.07	0 - 0.07	2002: 130 2009: ND	3 - 3.07
AN-CTPD-03	0-0.5		2002: 140	3 - 3.5

Location/ID	Sample Interval (ft) 2002	Sample Interval (ft) 2009	Total PCBs (µg/kg)
AN-CTPD-06	0 - 0.07	0 - 0.07	2002: 130 2009: ND
AN-CTPD-06	0-0.5		2002: 110

Location/ID	Interval (ft)	Total PCBs (µg/kg)
AN-CTPD-02	0 - 0.07	110
AN-CTPD-02	0-0.5	91

Location/ID	Interval (ft)	Total PCBs (µg/kg)
DMMU2SDO4	0 - 5.4	99

Location/ID	Interval (ft)	Total PCBs (µg/kg)
AN-CTPD-07	0 - 0.07	120
AN-CTPD-07	0-0.5	92

AN-CTPD-03
(2018 bathymetry data indicate mudline elevation is shallower than when the 2002 sample was collected)

S161
(2018 PCB congener results for surface sample at same interval and elevation as TPD-06 0-0.5 ft sample)

S158

S162

AN-CTPD-06

Preliminary SMA 2

SC-S157

S157

DMMU2

S159

AN-CTPD-02
(Removed during 2011 dredging)

Kinder Morgan Dock

Chevron Dock

Phillips 66 Dock

DMMU-1-C
(Surface interval removed during 2011 dredging. Subsurface result shown represents leave surface)

AN-CTPD-07
(Removed during 2011 dredging)

DMMU-2-C
(Surface interval removed during 2011 dredging. Subsurface result shown represents leave surface

Tidewater Barge

Shallow/Intermediate Boundary

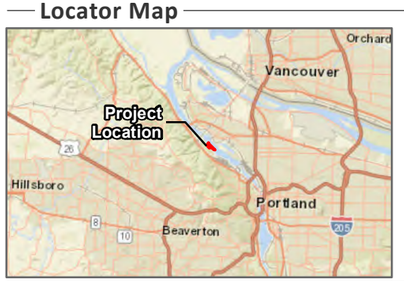
-44 -42 -32 -38 -36 -28 -34 -26 -24 -18 -16 -14 -12 -8 -6 -4 -2 -22 2

Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

Document Path: \\dc1vd01\GIS\Proj\P\Portland Harbor\Group\B2_Willbridge Cove\Map Files\Report_PDI\20_WC_DataGaps_Evaluation_Area2.mxd

Locator Map



Legend

PCB µg/kg - Surface Sediment
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect
- FS sample location removed during 2011 dredging

PCB µg/kg - Subsurface Sediment
Maximum detected concentration in core
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

- Bathymetry (2ft) Contours
- Shallow/Intermediate Boundary
- Willbridge Cove Project Area
- 2011 Dredge Prism
- Sediment Management Area (ROD ESD 2018)
- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

0 75 150 Feet

FIGURE 2-24
PRELIMINARY SMA 2 EVALUATION RESULTS
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

Location/ID	Interval (ft)	Total PCBs (µg/kg)
URB-2Z	3.9 - 4.9	200

Location/ID	Interval (ft)	Total PCBs (µg/kg)
SC-S163	0 - 1.0	15.3
	0 - 2.0	27
	2.0 - 4.0	177
	4.0 - 6.0	59.8
	6.0 - 8.0	88.2
	8.0 - 10.0	50.8
	10.0 - 12.7	285
	12.7 - 13.0	2.7

Location/ID	Interval (ft)	Total PCBs (µg/kg)
G694	0 - 0.7	89

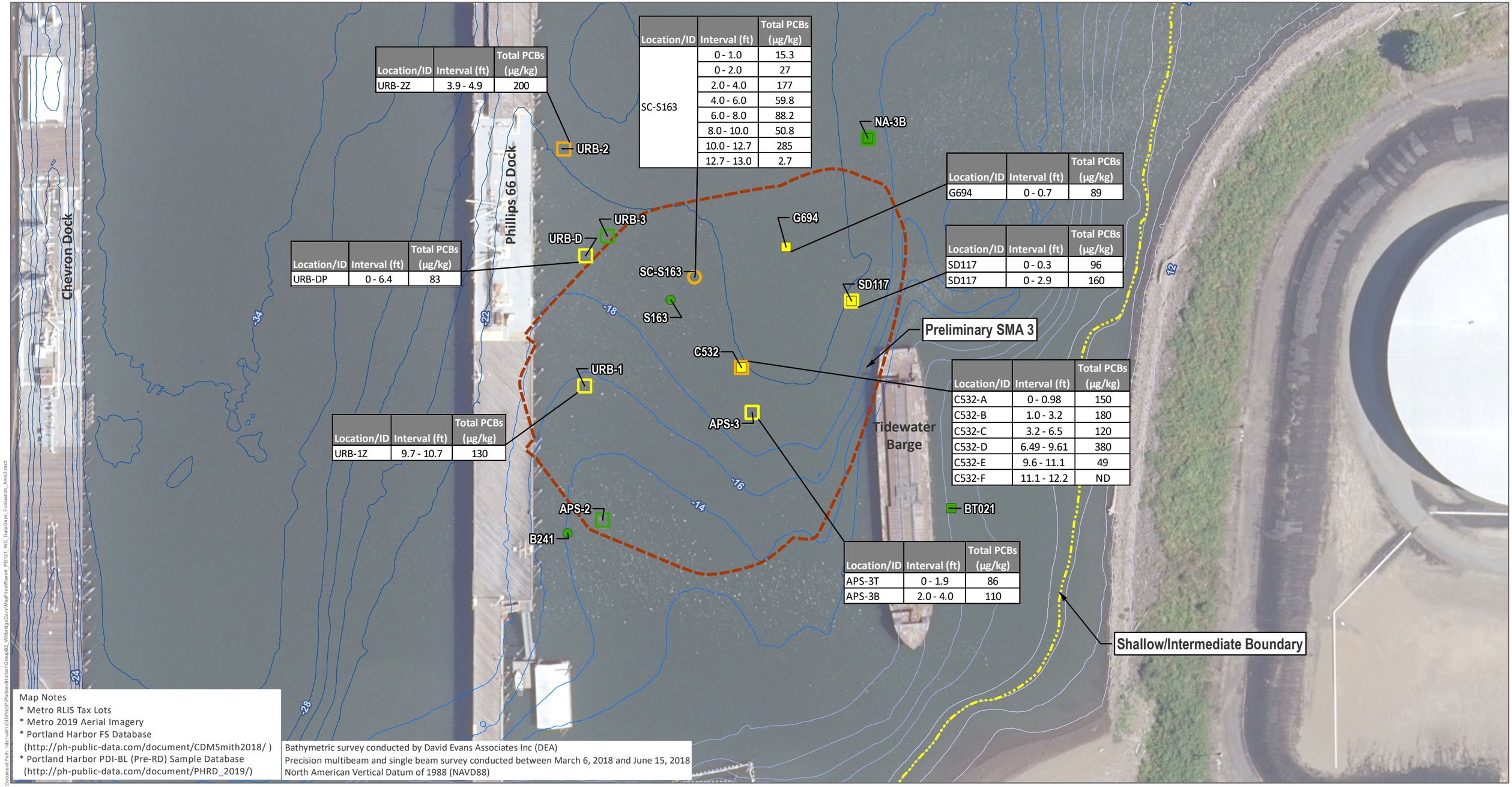
Location/ID	Interval (ft)	Total PCBs (µg/kg)
URB-DP	0 - 6.4	83

Location/ID	Interval (ft)	Total PCBs (µg/kg)
SD117	0 - 0.3	96
SD117	0 - 2.9	160

Location/ID	Interval (ft)	Total PCBs (µg/kg)
C532-A	0 - 0.98	150
C532-B	1.0 - 3.2	180
C532-C	3.2 - 6.5	120
C532-D	6.49 - 9.61	380
C532-E	9.6 - 11.1	49
C532-F	11.1 - 12.2	ND

Location/ID	Interval (ft)	Total PCBs (µg/kg)
URB-1Z	9.7 - 10.7	130

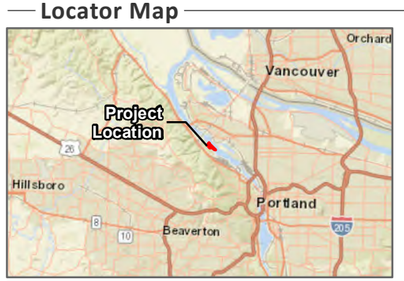
Location/ID	Interval (ft)	Total PCBs (µg/kg)
APS-3T	0 - 1.9	86
APS-3B	2.0 - 4.0	110



Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

Locator Map



Legend

PCB µg/kg - Surface Sediment
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

PCB µg/kg - Subsurface Sediment
Maximum detected concentration in core
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

- Bathymetry (2ft) Contours
- Shallow/Intermediate Boundary
- Sediment Management Area (ROD ESD 2018)
- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

0 75 150 Feet

FIGURE 2-25
PRELIMINARY SMA 3 EVALUATION RESULTS
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

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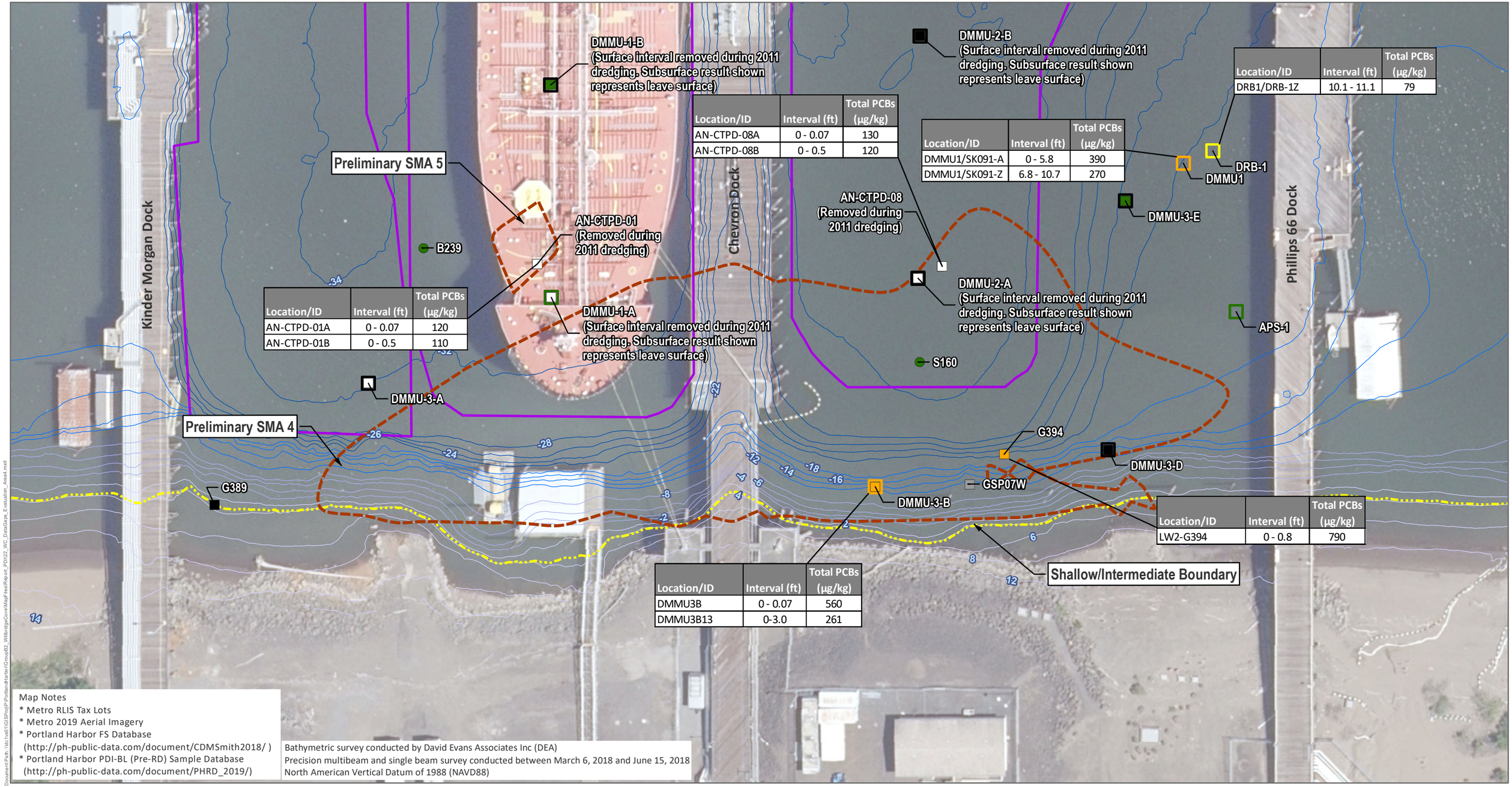

Location/ID	Interval (ft)	Total PCBs (µg/kg)
AN-CTPD-01A	0 - 0.07	120
AN-CTPD-01B	0 - 0.5	110

Location/ID	Interval (ft)	Total PCBs (µg/kg)
AN-CTPD-08A	0 - 0.07	130
AN-CTPD-08B	0 - 0.5	120

Location/ID	Interval (ft)	Total PCBs (µg/kg)
DMMU1/SK091-A	0 - 5.8	390
DMMU1/SK091-Z	6.8 - 10.7	270

Location/ID	Interval (ft)	Total PCBs (µg/kg)
DRB1/DRB-1Z	10.1 - 11.1	79

Location/ID	Interval (ft)	Total PCBs (µg/kg)
LW2-G394	0 - 0.8	790

Location/ID	Interval (ft)	Total PCBs (µg/kg)
DMMU3B	0 - 0.07	560
DMMU3B13	0-3.0	261

Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery
* Portland Harbor FS Database (http://ph-public-data.com/document/CDMSmith2018/)
* Portland Harbor PDI-BL (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)

Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

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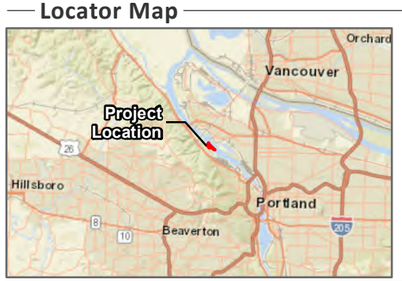
Locator Map



Legend

PCB µg/kg - Surface Sediment
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect
- FS sample location removed during 2011 dredging

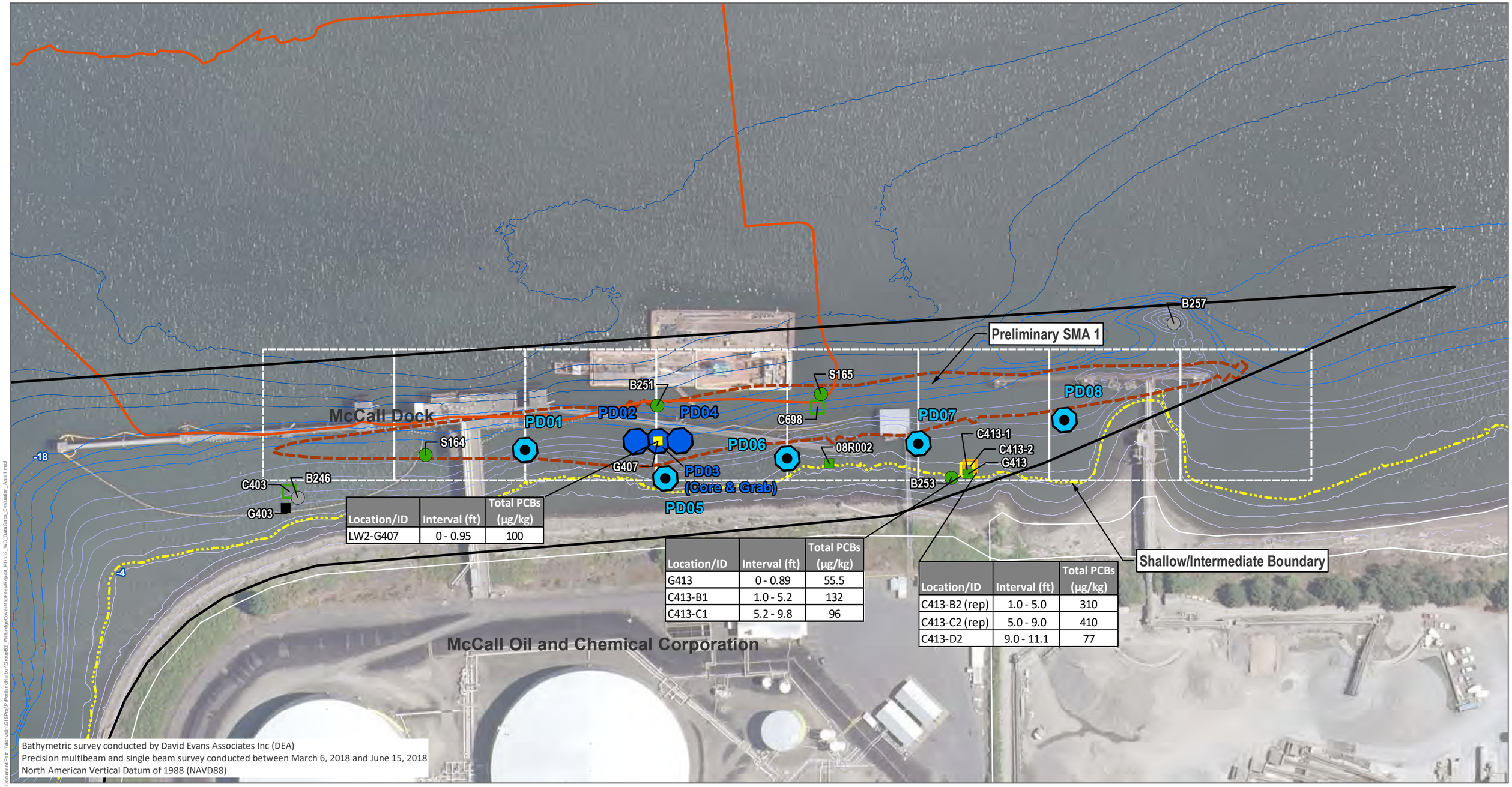
PCB µg/kg - Subsurface Sediment
Maximum detected concentration in core
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

- Bathymetry (2ft) Contours
- Shallow/Intermediate Boundary
- 2011 Dredge Prism
- Sediment Management Area (ROD ESD 2018)
- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

0 62.5 125 Feet

FIGURE 2-26
PRELIMINARY SMA 4 AND SMA 5 EVALUATION RESULTS
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



Location/ID	Interval (ft)	Total PCBs (µg/kg)
LW2-G407	0 - 0.95	100

Location/ID	Interval (ft)	Total PCBs (µg/kg)
G413	0 - 0.89	55.5
C413-B1	1.0 - 5.2	132
C413-C1	5.2 - 9.8	96

Location/ID	Interval (ft)	Total PCBs (µg/kg)
C413-B2 (rep)	1.0 - 5.0	310
C413-C2 (rep)	5.0 - 9.0	410
C413-D2	9.0 - 11.1	77

Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

Document Path: \\dc1vs01\GIS\Proj\P\Portland Harbor\Group\B2_Willbridge Cove\Mxd Files\Report_PDI\02_WC_DataGaps_Evaluation_Area1.mxd

Legend

Proposed Samples
- Primary Core/Grab (10ft)
- Primary Surface Grab
- Stepout Core/Grab (10ft)

PCB µg/kg - Surface Sediment
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

PCB µg/kg - Subsurface Sediment
Maximum detected concentration in core
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

- Bathymetry (2ft) Contours
- Shallow/Intermediate Boundary
- Willbridge Cove Project Area
- 2020-2021 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

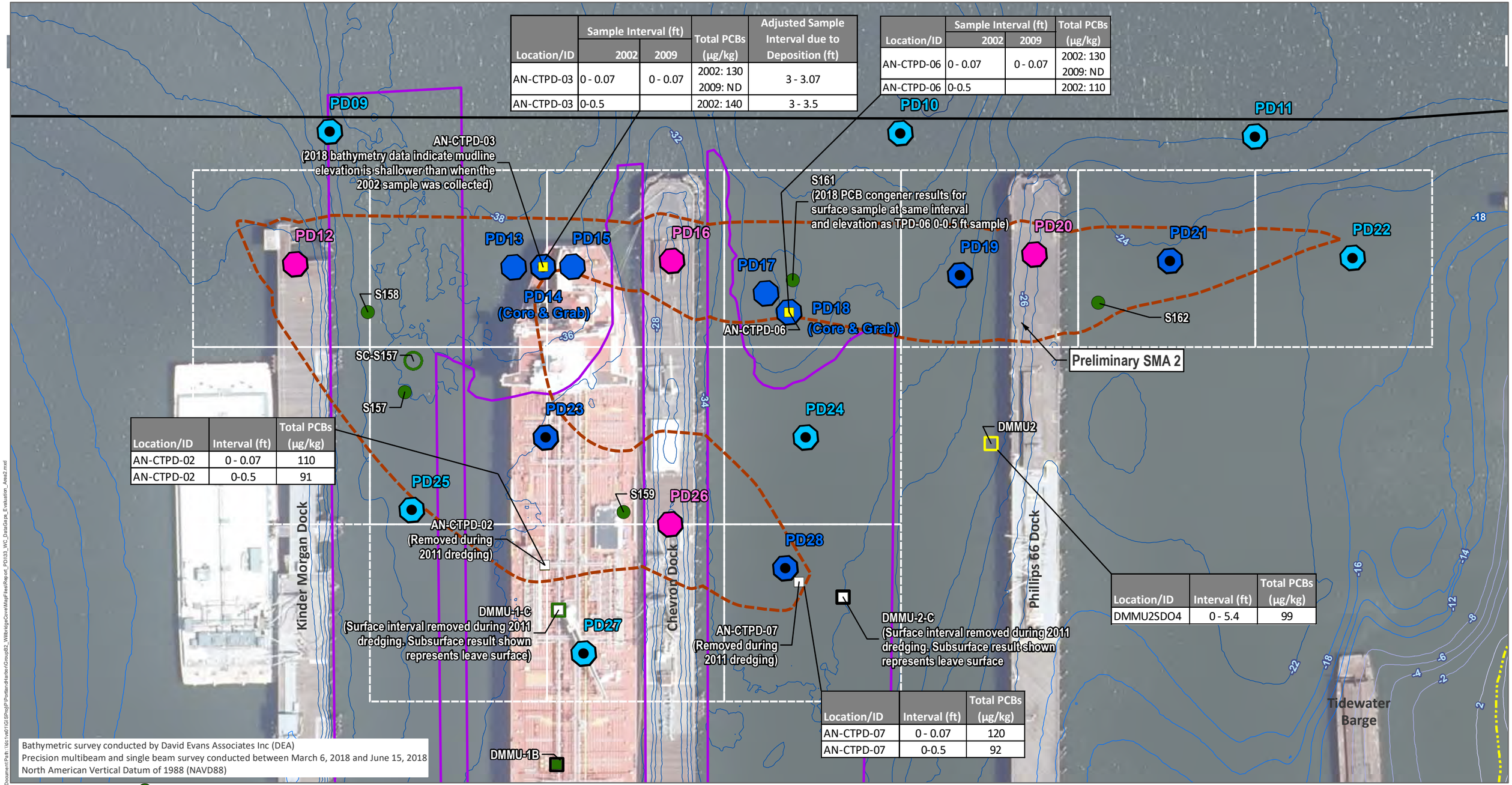
CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 100 200 Feet

FIGURE 3-1

PROPOSED SAMPLE LOCATIONS
PRELIMINARY SMA 1 EVALUATION
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



Location/ID	Sample Interval (ft) 2002	Sample Interval (ft) 2009	Total PCBs (µg/kg)	Adjusted Sample Interval due to Deposition (ft)
AN-CTPD-03	0 - 0.07	0 - 0.07	2002: 130 2009: ND	3 - 3.07
AN-CTPD-03	0-0.5		2002: 140	3 - 3.5

Location/ID	Sample Interval (ft) 2002	Sample Interval (ft) 2009	Total PCBs (µg/kg)
AN-CTPD-06	0 - 0.07	0 - 0.07	2002: 130 2009: ND
AN-CTPD-06	0-0.5		2002: 110

Location/ID	Interval (ft)	Total PCBs (µg/kg)
AN-CTPD-02	0 - 0.07	110
AN-CTPD-02	0-0.5	91

Location/ID	Interval (ft)	Total PCBs (µg/kg)
DMMU2SDO4	0 - 5.4	99

Location/ID	Interval (ft)	Total PCBs (µg/kg)
AN-CTPD-07	0 - 0.07	120
AN-CTPD-07	0-0.5	92

Document Path: \\dc1vs01\GISProj\P\PortlandHarbor\Group82_WillbridgeCove\MapFiles\Report_PDI\33_WC_DataGaps_Evaluation_Area2.mxd

Legend

Proposed Samples

- Primary Core/Grab (10ft)
- Stepout Core/Grab (10ft)
- Primary Surface Grab
- Stepout Grab
- Under Dock Grab

PCB µg/kg - Surface Sediment

- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

PCB µg/kg - Subsurface Sediment
Maximum detected concentration in core

- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

- Bathymetry (2ft) Contours
- Shallow/Intermediate Boundary
- 2011 Dredge Prism
- Willbridge Cove Project Area
- Sediment Management Area (ROD ESD 2018)

- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 75 150 Feet

FIGURE 3-2

PROPOSED SAMPLE LOCATIONS

PRELIMINARY SMA 2 EVALUATION

Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Location/ID	Interval (ft)	Total PCBs (µg/kg)
URB-2Z	3.9 - 4.9	200

Location/ID	Interval (ft)	Total PCBs (µg/kg)
URB-DP	0 - 6.4	83

Location/ID	Interval (ft)	Total PCBs (µg/kg)
URB-1Z	9.7 - 10.7	130

Location/ID	Interval (ft)	Total PCBs (µg/kg)
C532-A	0 - 0.98	150
C532-B	1.0 - 3.2	180
C532-C	3.2 - 6.5	120
C532-D	6.49 - 9.61	380
C532-E	9.6 - 11.1	49
C532-F	11.1 - 12.2	ND

Location/ID	Interval (ft)	Total PCBs (µg/kg)
SC-S163	0 - 1.0	15.3
	0 - 2.0	27
	2.0 - 4.0	177
	4.0 - 6.0	59.8
	6.0 - 8.0	88.2
	8.0 - 10.0	50.8
	10.0 - 12.7	285
	12.7 - 13.0	2.7

Location/ID	Interval (ft)	Total PCBs (µg/kg)
G694	0 - 0.7	89

Location/ID	Interval (ft)	Total PCBs (µg/kg)
SD117	0 - 0.3	96
SD117	0 - 2.9	160

Location/ID	Interval (ft)	Total PCBs (µg/kg)
APS-3T	0 - 1.9	86
APS-3B	2.0 - 4.0	110

S162, PD29, PD30, URB-2, PD31, NA-3B, PD32, URB-3, URB-D, G694, PD33, SC-S163, S163, SD117, PD34, C532, URB-1, PD36, Preliminary SMA 3, APS-3, Tidewater Barge, PD37, PD35, APS-2, BT021, B241, PD38, PD39, C401, Phillips 66 Dock, Shallow/Intermediate Boundary

Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

Legend

Proposed Samples
- Primary Core/Grab (10ft)
- Primary Core/Grab (15ft)
- Under Dock Grab
- Stepout Core/Grab (10ft)
- Stepout Core/Grab (15ft)
- Stepout Grab

PCB µg/kg - Surface Sediment
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

PCB µg/kg - Subsurface Sediment
Maximum detected concentration in core
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

- Bathymetry (2ft) Contours
- Shallow/Intermediate Boundary
- Sediment Management Area (ROD ESD 2018)
- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 75 150 Feet

FIGURE 3-3
PROPOSED SAMPLE LOCATIONS
PRELIMINARY SMA 3 EVALUATION
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Document Path: \\dc-tvs01\GIS\Proj\PortlandHarbor\GroupB2_Willbridge\CoveMapFiles\Report_PDI\34_WC_DataGaps_Evaluation_Area3.mxd

Kinder Morgan Dock

Chevron Dock

Phillips 66 Dock

Preliminary SMA 5

Preliminary SMA 4

Location/ID	Interval (ft)	Total PCBs (µg/kg)
AN-CTPD-01A	0 - 0.07	120
AN-CTPD-01B	0 - 0.5	110

Location/ID	Interval (ft)	Total PCBs (µg/kg)
AN-CTPD-08A	0 - 0.07	130
AN-CTPD-08B	0 - 0.5	120

Location/ID	Interval (ft)	Total PCBs (µg/kg)
DMMU1/SK091-A	0 - 5.8	390
DMMU1/SK091-Z	6.8 - 10.7	270

Location/ID	Interval (ft)	Total PCBs (µg/kg)
DRB1/DRB-1Z	10.1 - 11.1	79

Location/ID	Interval (ft)	Total PCBs (µg/kg)
DMMU3B	0 - 0.07	560
DMMU3B13	0-3.0	261

Location/ID	Interval (ft)	Total PCBs (µg/kg)
LW2-G394	0 - 0.8	790

PD40 PD41 PD42 PD43 PD44 PD45 PD46 PD47 PD48 PD49 PD50 PD51 PD52 PD53

DMMU-1-A (Surface interval removed during 2011 dredging. Subsurface result shown represents leave surface)

DMMU-1-B (Surface interval removed during 2011 dredging. Subsurface result shown represents leave surface)

DMMU-2-A (Surface interval removed during 2011 dredging. Subsurface result shown represents leave surface)

DMMU-2-B (Surface interval removed during 2011 dredging. Subsurface result shown represents leave surface)

AN-CTPD-01 (Removed during 2011 dredging)

AN-CTPD-08 (Removed during 2011 dredging)

DMMU-3-A DMMU-3-B DMMU-3-D DMMU-3-E DMMU1 DRB-1 APS-1 B239 S160 G389 G394 GSP07W

Shallow/Intermediate Boundary

Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

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Legend

Proposed Samples
- Primary Core/Grab (10ft)
- Primary Core/Grab (15ft)
- Under Dock Grab
- Stepout Core/Grab (10ft)
- Stepout Core/Grab (15ft)
- Stepout Grab

PCB µg/kg - Surface Sediment
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

PCB µg/kg - Subsurface Sediment
Maximum detected concentration in core
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

- Bathymetry (2ft) Contours
- Shallow/Intermediate Boundary
- 2011 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

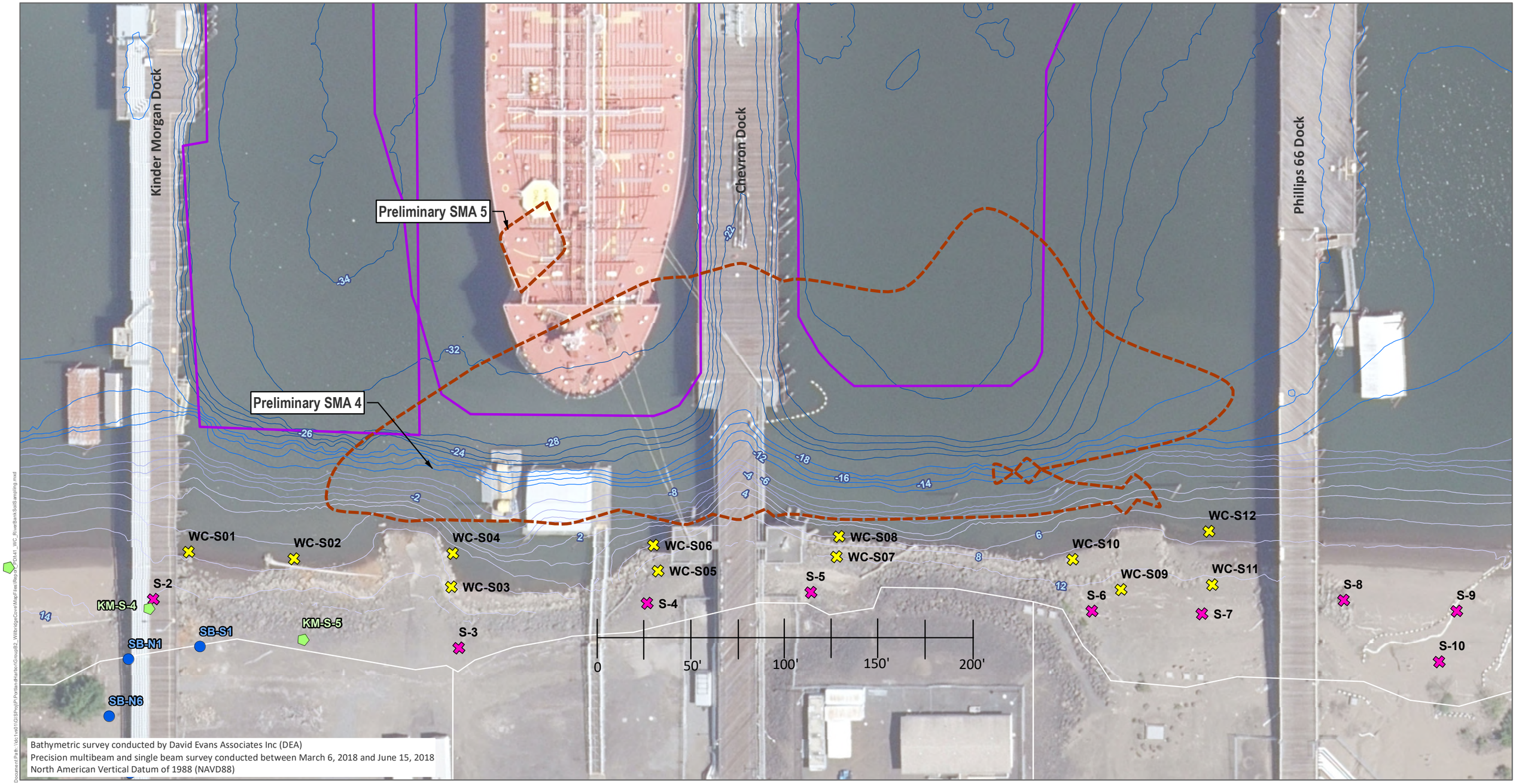
- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

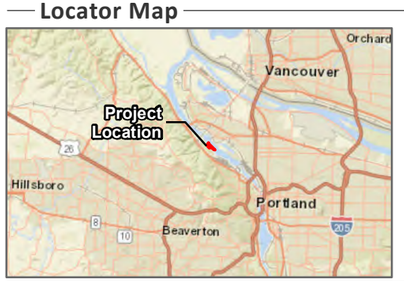
0 62.5 125 Feet

FIGURE 3-4
PROPOSED SAMPLE LOCATIONS
PRELIMINARY SMA 4 AND SMA 5 EVALUATION
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



Locator Map



Legend

- Proposed River Bank Soil Sample
- Surface Soils Sample (2008)
- KMLT Surface Sample Locations (2012)
- KMLT Soil Boring/LIF Locations (2018)
- Preliminary Sediment Management Area
- Dredge Area
- Bathymetry (2ft) Contours

Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery

0 62.5 125 Feet

FIGURE 3-5

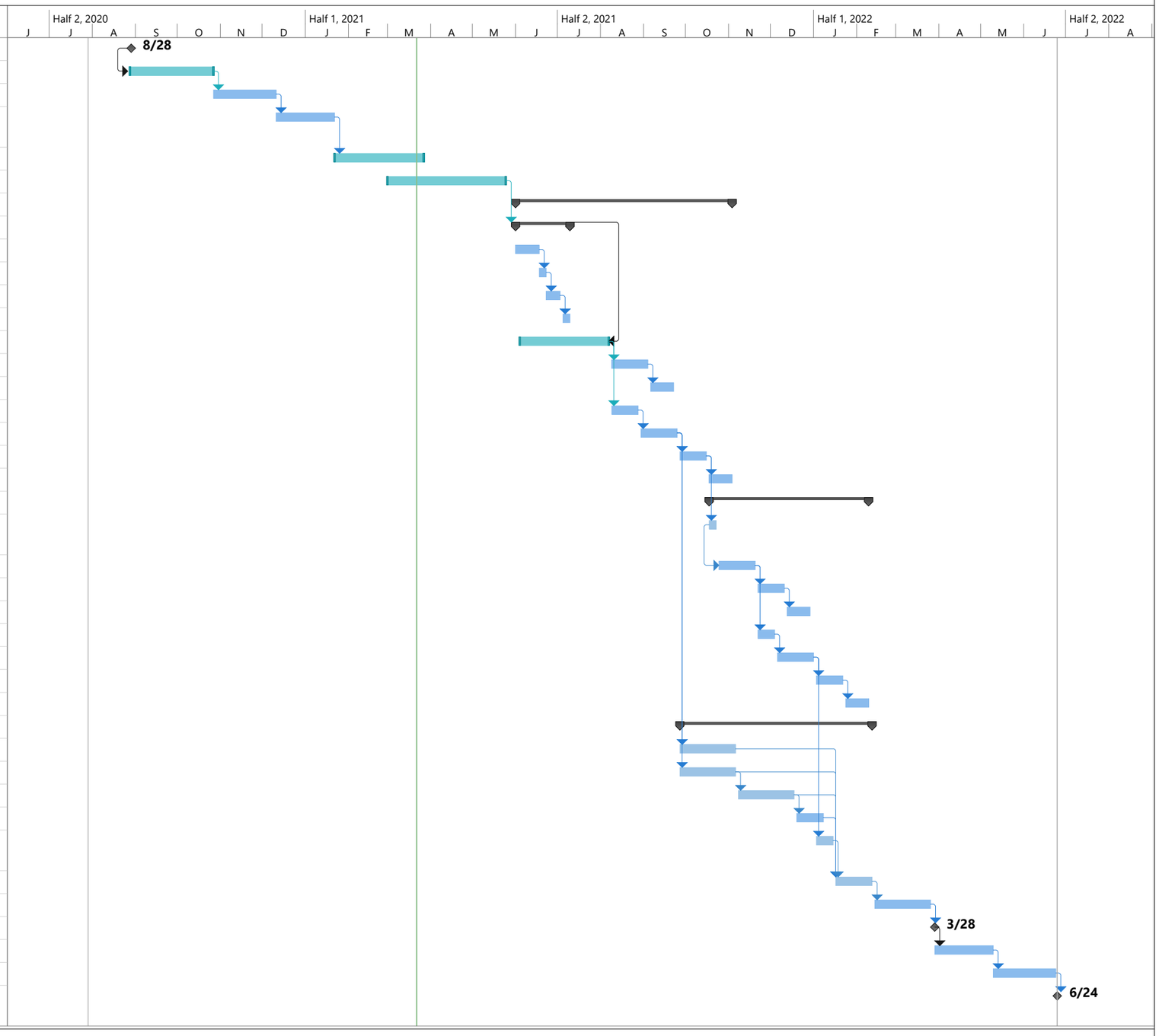
PROPOSED RIVER BANK SOIL SAMPLE LOCATIONS

Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

Figure 7-1 Schedule Willbridge Cove Project Area PDI

ID	Task Mode	Task Name	Duration	Start	Finish
1		Draft PDI Work Plan Submittal	1 day	Fri 8/28/20	Fri 8/28/20
2		EPA comments on Draft PDI WP	8.4 wks	Fri 8/28/20	Mon 10/26/20
3		Revised PDI Work Plan Submittal	45 edays	Mon 10/26/20	Thu 12/10/20
4		EPA Conditional Approval of Revised Draft PDI Work Plan	6 wks	Fri 12/11/20	Thu 1/21/21
5		PDI Work Plan Addendum	9.2 wks	Fri 1/22/21	Fri 3/26/21
6		Field Planning and Procurement	12.2 wks	Mon 3/1/21	Mon 5/24/21
7		**In River Work**	**22.3 wks**	**Tue 6/1/21**	**Wed 11/3/21**
8		**Sediment Sampling**	**5.8 wks**	**Tue 6/1/21**	**Fri 7/9/21**
9		Coring	13 days	Tue 6/1/21	Thu 6/17/21
10		Dive Grabs	3 days	Fri 6/18/21	Tue 6/22/21
11		Surface Grabs	8 days	Wed 6/23/21	Fri 7/2/21
12		Contingency	5 days	Mon 7/5/21	Fri 7/9/21
13		Laboratory Analysis - Sediment	9.2 wks	Fri 6/4/21	Fri 8/6/21
14		Data QA/QC; Validation	4 wks	Mon 8/9/21	Fri 9/3/21
15		Database for Sediment	2.5 wks	Mon 9/6/21	Wed 9/22/21
16		Assess Archive In River Samples	3 wks	Mon 8/9/21	Fri 8/27/21
17		Laboratory Analysis - Archive Samples	4 wks	Mon 8/30/21	Fri 9/24/21
18		Data QA/QC; Validation	3 wks	Mon 9/27/21	Fri 10/15/21
19		Database for Sediment	2.5 wks	Mon 10/18/21	Wed 11/3/21
20		**River Bank Work**	**16.5 wks**	**Mon 10/18/21**	**Wed 2/9/22**
21		River Bank Sampling (Low River Stage Required)	1 wk	Mon 10/18/21	Fri 10/22/21
22		Laboratory Analysis - River Banks	4 wks	Mon 10/25/21	Fri 11/19/21
23		Data QA/QC; Validation	3 wks	Mon 11/22/21	Fri 12/10/21
24		Database for River Bank Soil	2.5 wks	Mon 12/13/21	Wed 12/29/21
25		Assess Archive River Bank Samples	2 wks	Mon 11/22/21	Fri 12/3/21
26		Laboratory Analysis - Archive Samples	4 wks	Mon 12/6/21	Fri 12/31/21
27		Data QA/QC; Validation	3 wks	Mon 1/3/22	Fri 1/21/22
28		Database for River Bank Soil	2.5 wks	Mon 1/24/22	Wed 2/9/22
29		**Prepare Draft PDI Report**	**20 wks**	**Mon 9/27/21**	**Fri 2/11/22**
30		Prepare outline and initial text	6 wks	Mon 9/27/21	Fri 11/5/21
31		Compile Sediment Data into GIS maps/ tab	6 wks	Mon 9/27/21	Fri 11/5/21
32		Evaluate Preliminary SMA Boundaries	6 wks	Mon 11/8/21	Fri 12/17/21
33		Physical and Chemical Stability Evaluation	3 wks	Mon 12/20/21	Fri 1/7/22
34		Compile River Bank Data into GIS maps/ tables	2 wks	Mon 1/3/22	Fri 1/14/22
35		Draft remaining text	4 wks	Mon 1/17/22	Fri 2/11/22
36		Client review / editing Period	6 wks	Mon 2/14/22	Fri 3/25/22
37		Submit Draft PDI Evaluation to EPA	1 day	Mon 3/28/22	Mon 3/28/22
38		EPA review of Draft PDI Evaluation Report	6 wks	Tue 3/29/22	Mon 5/9/22
39		Comment Resolution/ Finalize Text	45 edays	Mon 5/9/22	Thu 6/23/22
40		Finalize PDI Evaluation Report - Submittal to EPA	1 day	Fri 6/24/22	Fri 6/24/22



Project: WCPA
Date: Mon 3/22/21

Task
Split
Milestone
Summary
Project Summary
Inactive Task
Inactive Milestone
Inactive Summary
Manual Task
Duration-only
Manual Summary Rollup
Manual Summary
Start-only
Finish-only
External Tasks
External Milestone
Deadline
Progress
Manual Progress

Attachment 1
Responses to EPA Comments on Revised Draft PDI Work Plan

Responses to Comment in Conditional Approval Letter for Revised Draft PDI Work Plan

Comment ID	Comment & Response History/**Comment**	**Response**
PDI WP Overview	Unless otherwise noted, Willbridge Cove Group's responses to EPA's comments on the Draft PDI WP and the Revised Draft PDI WP are acceptable. EPA submits the following clarifications, supplemental information and/or correction requests for General Comments 1, 2, and 3; Specific Comments 7, 12b, and 13.	N/A – including for context.
PDI WP General Comment 1	**EPA General Comment 1 (October 26, 2020)** **Contaminant Delineation:** The proposed sampling plan is insufficient to refine sediment management areas (SMAs) and the riverbanks pending characterization according to EPA's *Remedial Design Guidelines and Considerations* (RDGC). EPA requests the following modifications. a) Additional sampling locations should be positioned in and around the preliminary SMAs to horizontally delineate contamination. Per Section 5.1.2 of EPA's RDGC, contaminated sediment shall be delineated on a nominal 150- by 150-foot (ft) grid with step-out samples in a 150-ft buffer zone to confirm the extent of contamination (EPA 2020). The PDI WP argues that there are cases of older data which are no longer representative of site conditions for various reasons. Quantitative rationale must be provided to substantiate that claim after SMA refinement sampling conducted as part of the PDI. c) Include at least two additional samples on the north portion of the riverbank beyond S01 and S02 to horizontally delineate contamination on the bank adjacent to preliminary SMA 4 as well as at least two additional riverbank samples near SB-N6 (DDT removal area). Additional step-out samples should be located at both ends of the ordinary high water and mean low water transects as well as riverward of the samples near SB-N6 to confirm the extent of contamination. Furthermore, the Willbridge Cove Group should use a sampling technology, such as limited access direct push or angled borings, which can achieve greater than 5-ft depth. If DOC is not fully delineated within the 5-ft depth, EPA will require additional sampling to delineate DOC in the riverbank. d) The quantity and quality of sediment beneath dock structures within SMA footprints is not discussed. Because these areas have not been dredged, as evidenced by the residual "sand bars" in cross-section D-D', they could conceivably reflect higher historical sediment concentration. The sediment quality beneath the docks also is a factor in the recontamination assessment. The lack of sediment quality data beneath the docks is a data gap that should be considered in the PDIWP. *Willbridge Cove Group's Response (December 10, 2020)* a) Sampling designs have been revised to collect additional data needed to confirm and delineate SMAs. The location and types of samples are based on preliminary SMA characteristics (e.g., bathymetry and authorized dredge depths), anticipated remedial technology assignments based on the ROD Technology Decision Tree (e.g., dredging or capping), and the location, depths, and chemical results for existing FS and Pre-RD samples within and adjacent to the preliminary SMAs. Where appropriate, the sampling designs use the recommended 150-ft grid spacing. Sections 2.2 and 2.3 of the revised PDI Work Plan describes the locations, intervals, and analytical data for historical samples that were dredged in 2011 and are no longer representative of site conditions. This information was considered in development of the PDI sampling designs and will be reiterated as applicable in the PDI Evaluation Report. c) The river bank sampling design has been updated, but does not include the additional samples requested in the comment. The objective of river bank sampling is to determine whether the adjacent preliminary SMA is contiguous with river bank soil contamination. The DDT removal area in the vicinity of SB-N6 is not adjacent to Preliminary SMA 4 and elevated PCBs (the focused COC with RAL exceedances at Preliminary SMA 4) are not the driver for the DDT removal action under the KMLT dock. The under-dock removal action will include collection of post-removal confirmation samples. These post-removal results will be considered in the remedial design for the river bank if one is necessary. With regard to sample depths along the river bank adjacent to Preliminary SMA 4, sampling deeper than 5 feet to determine depth of contamination is not necessary for remedial design purposes in this area based on the expected technology assignment for the area and depth of feasible dredging or excavation. d) The sampling designs for preliminary SMAs that extend beneath dock structures have been modified to include surface sediment sample locations beneath the docks. *EPA Response (January 21, 2021)* a) The Revised Draft PDI WP does include additional samples, but it still fails to delineate SMAs based on the 150- by 150-foot grid specified in the RDGC. Samples at the following locations should be added to the PDI WP. • **SMA 1**: To achieve the required spatial resolution for SMA delineation, add one sample each in the downstream and upstream 150- by 150-foot grid squares (downstream of PD01 and upstream of PD07). • **SMA 2**: To achieve the required spatial resolution for SMA delineation, add one sample in the grid square north of PD24 along the boundary of the preliminary SMA and adjacent to the upstream side of the Kinder Morgan dock. Note that if PDI step-out samples PD08, PD09, and PD10 exceed RALs and/or PTW thresholds, the lateral and vertical extent of navigation channel RAL and/or PTW threshold exceedances will need to be delineated to no more than half the distance across the navigation channel, as per the Willbridge Cove Group's Statement of Work Section 5.6(c). • **SMA 4**: Locations PD40, PD44, and PD50 should be primary sampling locations to achieve the required spatial resolution for SMA delineation. Location PD43 should be a primary sample because of nearby subsurface PCB exceedances. c) EPA notes that the requested additional riverbank samples adjacent to preliminary SMA 4 and riverward of SB-N6 were not included in the PDI WP. Additionally, the requested riverbank soil sampling depth increase was not incorporated into the PDI WP. If the lateral extent of riverbank soil contamination exceeding RALs and/or PTW thresholds is not delineated adjacent to and potentially extending beyond preliminary SMA 4, or if adequate subsurface soil data are not generated for design of a riverbank remedial action in the WCPA, EPA will consider this a data gap requiring additional data collection in a supplemental PDI. d) The planned approach to include surface sediment sample locations beneath the docks is acceptable; however, if RAL or PTW threshold exceedances are present in surface sediment, additional sampling to delineate subsurface sediment exceedances will be required. Diver-assisted sediment cores up to 4 feet in length have been collected under structures in other RD project areas at Portland Harbor.	a. Request for additional delineation cores and grabs: i SMA 1: Additional cores were not added to the upstream and downstream ends of this preliminary SMA because there are already step-out sample/core locations upstream and downstream of the primary sample/core location at preliminary SMA 1. A step-out core and grab sample has been added between the shoreline and primary locations PD02, PD03, and PD04 based on comment Specific Comment 13. ii SMA 2: Additional step-out samples were not added around SMA 2. This area has been routinely dredged and the additional suggested samples are unlikely to substantively change the interpretation or application of the ROD decision tree. The Willbridge Cove Group acknowledges that additional sampling may be needed in the navigation channel pending the results from the row of step-out samples on the eastern edge of the project boundary. iii SMA 4: The suggested sampling locations were changed to primary sampling locations and two additional step-out sampling locations have been added in this area. c. River bank sampling i Two additional riverbank sampling locations have been added on the northern end of this area. The need for additional samples to characterize the river bank adjacent to SMA 4 will be determined after the PDI data are evaluated. d. Under dock sample cores i Comment is acknowledged, the need for additional samples to characterize the under-dock areas will be determined after the PDI data are evaluated.

Comment ID	Comment & Response History/**Comment**	**Response**
PDI WP General Comment 2	**EPA General Comment 2 (October 26, 2020)** Analysis of all ROD Table 21 contaminants in PDI sediment samples is expected as part of remedial design (RD). Existing sediment data should be discussed for all ROD Table 21 contaminants compared to the applicable RALs and/or PTW thresholds. References to and discussion of the focused contaminants of concern (COCs) need to be replaced with ROD Table 21 contaminants and an expanded discussion on the additional contaminants should be included in the PDI WP. Riverbank soil samples must be analyzed for contaminants with ROD Table 17 riverbank soil/sediment cleanup levels (CULs) and Table 21 contaminants per RDGC Appendix D Section 2.2 unless a technical rationale for excluding a specific analyte based on the conceptual site model (CSM) can be provided. *Willbridge Cove Group's Response (December 10, 2020)* The text has been clarified to note that the sediment samples will be analyzed for Table 21 contaminants except for chlorobenzene. Narrative has been added to discuss PTW thresholds that are exceeded for any of the other Table 21 COCs that do not have RALs. With regard to river bank soil, the proposed sampling design targets areas where bank impacts are not unexpected. Since these sample results will be used to establish the footprints for potential remedial action, the Table 21 parameters are appropriate. However, the soil samples will be analyzed using analytical methods with detection limits that are consistent with Table 17 CULs for possible future use in a recontamination evaluation if soil concentrations are below RALs. *EPA Response (January 21, 2021)* The riverbank sampling plan is inconsistent with RDGC Appendix D Sections 2.2.2 and 2.2.3. Development of the analytical suite for riverbank soil sampling should start with analysis of all ROD Table 17 and 21 contaminants and should consider the CSM, the nature of the COCs in the adjacent or nearby SMA, the history of activity in the WCPA, past releases, and any other site-specific considerations that might be relevant to developing the analyte list. The Willbridge Cove Group should modify the riverbank soil sampling analyte list to include the ROD Table 21 contaminants (minus chlorobenzene) and the Table 17 riverbank soil/sediment COCs or include a detailed rationale in the PDI WP for excluding certain Table 17 COCs.	The surface riverbank soil samples will be analyzed for Table 21 and Table 17 parameters except for chlorobenzene and tributyltin. Subsurface samples will be analyzed, where needed, to refine any remediation footprint. The need for additional data collection will be further evaluated with respect to evaluating potential recontamination pathways. This approach is consistent with EPA's Guidance for River Bank Characterizations and Evaluations at the Portland Harbor Superfund Site (December 2019) which is included as Appendix D to the *Remedial Design Guidelines and Considerations – Portland Harbor Superfund Site* (dated February 28, 2020). The River Bank Guidance details the following expectations in Section 2.2.2: *"The expected steps for delineating contamination within a river bank are as follows:* • *Characterize the **lateral** extent of contaminant concentrations exceeding the criteria listed in Table 1 (from ROD Tables 17 and 21) over the entire river bank. Spatial distribution and density of sample locations are to be designed in a SAP/QAPP to meet the investigation DQOs and collect data to support evaluations relative to RAO 9. When applicable, characterization data should be used to inform remedial design. Characterization should also provide data to confirm or deny the presence of nonaqueous phase liquid (NAPL) found in soil borings or wells at the top of the bank.* • *If surface sample results exceed PTW threshold values, which are NAPL, highly toxic PTW, and not reliably contained (NRC) PTW thresholds listed in Table 1, subsurface sampling is necessary to vertically bound contamination exceeding PTW threshold values to the depth appropriate to support remedial action design.* • *If results exceed CULs and/or RALs, vertical delineation of the extent of contamination would proceed to the depth appropriate to meet the investigation's DQOs developed in a preapproved SAP/QAPP. The depth of sampling must be sufficient to support evaluations relative to the protectiveness objectives of RAO 9 and, when needed, to support remedial design….."* The rationale for excluding chlorobenzene is already provided in the PDI Work Plan. A PDI Work Plan Addendum will be provided that documents that tributyltin is not a contaminant of concern within the WCPA. Briefly, The RI (EPA 2016) documented that complete or likely complete pathways for tributyltin were identified at Cascade General and Gunderson, which are outside of the WCPA. Further, tributyltin was introduced as anti-fouling additive to paint in the late 1960s and the cove was created through the placement of dredge fill between 1957 and 1966. RI and Pre-RD sampling data from the WCPA area indicate that sediment concentrations are well below the 3,080 µg/kg CUL. An evaluation of the available tributyltin results within the FS and Pre-RD data sets indicates that of the 9 Pre-RD surface sediment samples collected within the cove that were analyzed for tributyltin was detected in seven of the nine samples, at concentrations of 19 µg/kg or less; tributyltin was analyzed in 51 FS samples and detected in 24 at concentrations of 83 µg /kg or less. The analytical testing suite for this PDI work will not include tributyltin as it is not a constituent of concern within the WCPA based on the conceptual site model and existing sediment sampling results.

Comment ID	Comment & Response History/**Comment**	**Response**
PDI WP General Comment 3	**EPA General Comment 3 (October 26, 2020)** There are numerous RAL and/or PTW threshold exceedances in subsurface sediments outside of the SMA footprint for total PCBs; DDx; 2,3,7,8-TCDD; 1,2,3,7,8-PeCDD; and 1,2,3,4,7,8-HxCDF. These locations are shown on PDI WP Figures 2-12 through 2-17 for the focused COCs and tabulated for all ROD Table 21 contaminants in PDI WP Tables A-1 and A-2. Refinement of SMAs for RD needs to consider RAL and PTW threshold exceedances in subsurface sediment as well as at the surface as stated in RD principle #1 in EPA's RDGC. Whether or not areas with subsurface RAL exceedances will be included in an SMA is dependent on the chemical and physical stability of the buried material and reasonably anticipated future site uses (see RD principle #2 in EPA's RDGC). Additional subsurface sediment sampling should be performed to delineate these subsurface RAL and/or PTW threshold exceedances and EPA expects that a buried contamination evaluation will be performed as part of a future RD deliverable. *Willbridge Cove Group's Response (December 10, 2020)* The PDI Work Plan has been revised to include a physical/chemistry stability DQO (DQO 2) and describes the data requirements and evaluation process in Section 1.4.2 and the results of the related data gaps evaluations in Section 2.4. The data gap evaluations conclude that the existing data and planned PDI sampling data will be sufficient to characterize the chemical and physical stability of at-depth contamination in areas within and outside of the preliminary SMAs. No additional sampling specific to stability evaluations is needed. *EPA Response (January 21, 2021)* EPA does not agree that the planned PDI sampling data will provide adequate spatial coverage of the WCPA (see General Comment 1) or that no additional data is needed to evaluate the physical and chemical stability of buried contamination in the WCPA. EPA has the following comment on DQO 1 – SMA Refinement, and the PDI WP should be revised accordingly: a) The lateral extent of an SMA is dependent on both surface and subsurface contamination, and the extent of RAL and/or PTW threshold exceedances must be characterized to delineate SMAs. Additionally, the buried contamination evaluation must demonstrate physical and chemical stability of subsurface RAL exceedances before they are considered to be excluded from an SMA. Subsurface sediment exceeding PTW thresholds must be actively remediated in accordance with the ROD. Step 5 of Table 1-2 should be revised accordingly. EPA has the following comments on DQO 2 – Physical and Chemical Sediment Stability and the PDI WP should be revised accordingly: a) EPA disagrees with Willbridge Cove Group that no additional sampling specific to the stability evaluations is needed. There are multiple subsurface RAL and/or PTW threshold exceedances outside of the preliminary SMAs near which additional PDI samples should be collected. If Willbridge Cove Group elects to not collect additional samples outside of the preliminary SMAs, there may be insufficient data for SMA refinement (DQO 1) and/or the physical and chemical stability evaluations (DQO 2) that would require additional data collection as part of a supplemental PDI. b) Complete chemical stability modeling requires additional data which are not currently available or planned to be collected. It is acceptable to use porewater upwelling information from Appendix C of the RI; however, EPA recommends collecting current site-specific porewater contaminant concentrations for inputs to chemical fate and transport modeling. If the Willbridge Cove Group chooses to use literature-based partition coefficients to determine porewater concentrations for modeling, EPA should be consulted in the selection of appropriately conservative partition coefficients and resulting porewater concentrations should be compared to the site-specific porewater concentrations provided in Appendix C of the RI. c) Depth profiles of Cesium-137 and ROD Table 21 contaminants should be considered as an additional line of evidence. d) Hydrodynamic modeling and shear stress evaluations should use the 100-year flood event and include an analysis of the effects of climate change and seismic impacts, consistent with ROD requirements for caps. e) EPA expects a buried contamination long-term monitoring plan to be developed in future RD deliverables. f) Temporal trends in surface sediment contaminant concentrations, potential in-water construction, and reasonably anticipated future use should all be considered as additional lines of evidence to inform the stability of buried deposits.	DQO 1 Comment Response "a": The PDI Work Plan includes samples intended to characterize and delineate the lateral and vertical extent of RAL/PTW threshold exceedances in SMAs such that remedies appropriate to future use or location can be designed. The Willbridge Cove Group is developing a weight of evidence approach to evaluate the physical and chemical stability of buried contamination in the WCPA using existing data and data to be gathered during the PDI. The approach and associated lines of evidence for the stability evaluations will be developed prior to the presentation of the PDI results. The last sentence of the comment response regarding the need to actively remediate PTW threshold exceedances in subsurface sediment is inconsistent with the description of the selected remedy in Sections 11 and 14.2 of the ROD and EPA's guidance documents as summarized below: Section 6.5.1 of the ROD states "*The PTW evaluation only included surface sediment, which poses the greatest risk of exposure given Site-specific conditions.*" Figure 8 of the ROD identifies PTW in surface sediments that are characterized as Source Material/NAPL, Not Reliably Contained and Highly Toxic. Section 11 of the ROD then goes on to state that "*If NAPL or PTW that is not reliably contained is identified in any of the remediation areas under the Selected Remedy, it will be addressed as specified in Section 14.2.*" This sentence does not address Highly Toxic PTW nor areas with no surface RAL or PTW threshold exceedances. We agree that the ROD states that surface sediment exceeding PTW thresholds must be remediated, but the ROD does not address subsurface sediments in areas without a surface sediment exceedance of RALs or PTW thresholds. The Statutory Determination (page iv) states that where there are surface sediment exceedances of RALs or PTW thresholds, "[t]*he Selected Remedy will address all principal threat waste (PTW) by excavation and off-site disposal, or, if left in place, with augmented reactive caps to provide in-situ treatment.*" Sections 14.2.2 through 14.2.5 of the ROD state that "*NAPL or PTW that cannot be reliably contained will be dredged [or excavated]…".* Subsurface PTW in areas with no surface RAL or PTW threshold exceedances is not addressed. Hence, ROD statements apply to PTW in surface sediments and subsurface PTW in areas with surface RAL exceedances. Further, the decision tree (ROD Figure 28) and Section 14.2 of the ROD differentiate between different types of PTW. It treats PTW characterized as Highly Toxic (based on PTW threshold exceedances) in surface sediments by following the same approach as surface sediments with RAL exceedances, i.e., "N" path for Highly Toxic PTW, while NAPL and PTW not reliably contained are treated differently by following the "Y" path. By applying the same rationale, subsurface sediments where PTW is characterized as Highly Toxic based on PTW threshold exceedances, but not as NAPL or not reliably contained must be treated following the same approach as subsurface sediments with RAL exceedances. This approach is consistent with the decision presented in Appendix B – Remedial Design Topics and Discussion (December 23, 2019), Topic 4 – Defining Depth of Contamination during SMA Delineation. This appendix is part of the *Remedial Design Guidelines and Considerations – Portland Harbor Superfund Site* (dated February 28, 2020). The decision presented at the conclusion of the Topic 4 discussion states: "***For sites where NAPL does not exist and/or the sampling location is not within a dredging area, sediments with concentrations exceeding RALs or PTW thresholds at depth may be left in place if it can be demonstrated that the subsurface sediments will remain stable.*** *In such cases, the physical (i.e., erosive conditions, slope stability) and chemical (i.e., advective and diffusive flux due to seepage) stability of the buried contamination will need to be demonstrated by conducting appropriate evaluations to assess whether the material needs to be capped or to support cap design"* (emphasis added). Thus, where there are no surface RAL or PTW threshold exceedances and no NAPL, subsurface exceedances of PTW thresholds will be treated identically to subsurface RAL exceedances and the physical and chemical stability of the buried contamination will be evaluated to determine whether remedial action is required. Thus, step 5 of Table 1-2 has been revised to reflect the above discussion. DQO 2: a/b. See response to DQO 1 Comment Response "a" regarding SMA delineation and weight of evidence approach to be used for stability evaluations. Both empirical data and modeled results will be considered as lines of evidence in the evaluations. This will be developed prior to presentation of the PDI results. c. Depth profiles of the Table 21 contaminants will be used as a line of evidence in evaluating the physical and chemical stability. Radiometric dating is not proposed because given development of the cove and the associated maintenance dredging history these types of data are not expected to produce adequate resolution or be useful for remedial design. d. Comment acknowledged. Hydrodynamic modeling, where needed, will be performed in accordance with ROD requirements and the RD guidance, as applicable (and will be incorporated as part of physical stability protocol as appropriate). e. Comment acknowledged. A project area monitoring plan is a required element of the SOW and will be prepared as part of the remedial design. f. Comment acknowledged. These lines of evidence will be incorporated into the evaluation of chemical and physical stability, as appropriate.

Comment ID	Comment & Response History/**Comment**	**Response**
PDI WP Specific Comment 7	**EPA Specific Comment 7 (October 26, 2020)** This section should be renamed to "Summary of Sediment and Riverbank Soil Results" and a new subsection (Section 2.2.3 Riverbank Soil) should be developed to summarize the existing riverbank soil data collected during the source control evaluations and as part of the riverbank DDT removal action. This section should include a discussion of how the existing riverbank soil data pertain to the PDI WP (RAL/PTW threshold exceedances present, areas removed, and confirmation sampling results) and how these data informed the proposed PDI riverbank soil sample collection. *Willbridge Cove Group's Response (December 10, 2020)* Section has been renamed Summary of Existing Data and a section has been added that discusses existing soil data collected during the source control evaluations (Section 2.2.4). The new section includes additional information about conditions in the vicinity of the DDT removal area beneath the KMLT dock. However, because the objective of river bank sampling is to determine whether the adjacent preliminary SMA (SMA 4) is contiguous with river bank soil contamination and the DDT removal area is not adjacent to the SMA, the existing results are not considered in the river bank sampling design. Note that the under- dock removal action will include collection of post-removal confirmation samples. These post-removal results will be considered in the remedial design for the river bank if one is necessary. *EPA Response (January 21, 2021)* EPA expects to review the results of the riverbank source control measure as part of remedial design. Dependent upon the completeness of the source control measure and the PDI riverbank soil sampling there may be additional data gaps for RD.	The results of the river bank source control measures will be provided to EPA when the work is completed. These results will also be incorporated into the Basis of Design Report (BODR), as appropriate.
PDI WP Specific Comment 13	**EPA Specific Comment 13 (October 26, 2020)** The McCall Oil riverbank adjacent to preliminary SMA 1 is identified as a riverbank pending characterization in EPA's RDGC Appendix D. Revise the text to state that the riverbank is a potential data gap pending PDI sediment sampling and SMA refinement. From the riverbank source control data provided in Table A-3D, it appears that the sediment and riverbank to the southwest of the historic RAL exceedance have not been sampled below the top of the bank. SMA refinement will require sampling within 150-ft of the original exceedance and riverbanks contiguous with an SMA are subject to remedial action as part of the in-water remedy. Additionally, this section suggests that G407 is no longer representative of surface conditions, based on the inferred deposition of 4 to 5 feet of sediment since 2004. This sedimentation rate is substantially higher than the range presented in site figures or indicated in cross-sections of the 2009 dredge area, and the channel edge slope in the G407 area does not seem conducive to natural sedimentation. Additional clarification should be provided in the revised PDI WP. *Willbridge Cove Group's Response (December 10, 2020)* The data evaluation process used to identify river banks for characterization is described in Section 1.4.3. Section 2.5 describes the existing sample results used and the outcomes of the evaluation process for the McCall river bank and the Chevron/Phillips 66 river bank. The evaluation for the McCall river bank includes the results for river bank samples collected from the bank adjacent to Preliminary SMA 1 (see Figure 2.21 and Appendix A, Table A-3). With regard to the representativeness of G407 and depositional rates, Section 2.3.1 describes conditions in the vicinity of the sample and states that there [is] uncertainty associated with the inferred amount of deposition. Section 3.1.2 details how current surface conditions at G407 will be assessed by reoccupying the location and collecting a surface grab and a core. Two other surface grab samples will also be obtained in the immediate vicinity of G407 to provide additional data for the area and support possible data replacement. The elevation of the mudline at each location will be recorded during sampling and this elevation data can be compared to earlier measurements to refine sedimentation rates. *EPA Response (January 21, 2021)* Note that, pending the results of PDI sediment sampling at Preliminary SMA 1, there may be a data gap shoreward of PD02, PD03, and PD04 that EPA expects would be filled in a supplemental PDI.	A step-out grab and core location has been added shoreward of PD02, PD03, and PD04.

Comment ID	Comment & Response History/**Comment**	**Response**
PDI WP Specific Comment 18	**EPA Specific Comment 18 (October 26, 2020)** Include subsurface sediment sampling in addition to the planned surface sediment sampling. Per EPA's RDGC Section 1.4, SMAs will be delineated by surface and subsurface contamination above RALs and PTW thresholds as discussed in PDI WP Specific Comments 12d and 17 above. Failure to vertically delineate contamination will result in a data gap that will need to be addressed at a later stage. See PDI WP General Comment 3 regarding subsurface sediment and PDI WP General Comment 2 regarding COCs. *Willbridge Cove Group's Response (December 10, 2020)* See responses to General Comments 1a, 2, and 3. Revised sampling designs are provided in Section 3 of the PDI Work Plan. *EPA Response (January 21, 2021)* EPA notes that the analytical schedule provided in Table 3-2 of the Revised Draft PDI WP has been modified from the schedule in Table 3-3 of the Draft PDI WP. The sample intervals from 1 to 5 feet of primary locations, along with the composite surface grab sample, should be submitted for analysis immediately. The 0 to 1-foot sample interval and lower intervals may be archived for analysis pending the results from the top intervals and the composite surface grab sample.	The analysis and archiving scheme has been revised as follows: Location determines which primary sample/core intervals will be analyzed immediately: **Intermediate/shallow areas and not regularly dredged FMD areas* (5 intervals)** - Immediately analyze upper 5 feet of sediment (surface grab plus 4 one-ft sample intervals from 1 to 5 feet below mudline). * Current elevation in FMD areas that are not regularly dredged are within 2 ft of the operational elevation. Therefore, characterizing the upper 5 feet of sediment will bracket the operational elevation. • **Regularly dredged FMD areas (5 intervals)** – Immediately analyze surface grabs and the four one-ft intervals bracketing the operational elevation.
FSP Overview	Unless otherwise noted, Willbridge Cove Group's responses to EPA's comments on Appendix B FSP of the Draft PDI WP and the Revised Draft PDI WP are acceptable. However, EPA submits the following clarification, supplemental information and correction requests for Specific Comment 8.	No response required
FSP Specific Comment 8	**EPA Specific Comment 8 (October 26, 2020)** Visual assessment of NAPL needs to be performed for the riverbank soil samples similar to the sediment samples. This needs to be stated in Section 4.2, which should also state that the Evidence of Contamination procedures from SOP-5 will be followed when collecting and processing riverbank soil samples. *Willbridge Cove Group's Response (December 10, 2020)* Text edited to include field screening for NAPL in river bank soil, similar to that being conducted for sediment samples. SOP-12 was updated to include evaluation of evidence of contamination, consistent with SOP-5. *EPA Response (January 21, 2021)* EPA notes in the Revised Draft PDI WP, Appendix B FSP, SOP-5, Section 5.3.3, Evidence of Contamination subsection that the descriptors for NAPL were modified from those in the Draft PDI WP. The revisions are generally acceptable. However, under the Sheen bullet a fourth descriptor should be included as follows: "Dark: dark metallic (reflects/mirrors the color of the sky) or brown/black colored." This descriptor should also be included in SOP-12, Section 12.3, J. Evidence of Contamination.	SOPs 5,8, and 12 have been modified as requested and included in the PDI Work Plan Addendum.
QAPP Overview	Unless otherwise noted, Willbridge Cove Group's responses to EPA's comments on Appendix C QAPP of the Draft PDI WP and the Revised Draft PDI WP are acceptable. However, EPA submits the following clarifications, supplemental information and/or correction requests for Specific Comments 9 and 10.	No response required
QAPP Specific Comment 9	**EPA Specific Comment 9 (October 26, 2020)** Indicate who will conduct these audits and define the scope of authority of auditor, e.g., including stop work orders, and when auditors are authorized to act. Note that field audits are independent reviews which qualitatively document the degree to which the procedures and processes specified in the approved QAPP are being implemented. *Willbridge Cove Group's Response (December 10, 2020)* Section 8.1.2 edited to note field audits will be performed once a year, or as necessary, by the FTL or local field task lead to verify the execution of field procedures. *EPA Response (January 21, 2021)* The audit should be completed by an independent auditor who is not a part of the field team. The Field Team Lead (FTL) is part of the field team and should be conducting oversight of field activities throughout the year.	The QAPP has been revised to indicate an independent auditor will perform the field audit. The revised QAPP will be provided to EPA after the laboratory procurement and will include laboratory addresses, points of contact, certifications, and standard operating procedures.

Comment ID	Comment & Response History/**Comment**	**Response**
QAPP Specific Comment 10	**EPA Specific Comment 10 (October 26, 2020)** This section states that annual audits of the laboratory will be conducted by the laboratory's QA officer and that all audit findings will be documented in QA reports to management. Clarify if management refers solely to laboratory management or includes overall project management and QA management (e.g., Jacobs Project Manager and QA Manager) and if audit findings will be shared with the EPA Project Coordinator or Remedial Project Manager. In addition, indicate if internal audits of other elements of overall execution of the project (e.g., report preparation) are planned. *Willbridge Cove Group's Response (December 10, 2020)* Section 8.2.3 edited to note management is laboratory management. Also added the internal audit reports will be made available to the Jacobs PM and QAM, as well as to the EPA Project Coordinator upon request. *EPA Response (January 21, 2021)* EPA requests that the audit be provided to the EPA Project Coordinator (PC) within 10 days of receipt by Jacobs.	The QAPP has been revised as requested.
HASP Overview	Unless otherwise noted, Willbridge Cove Group's responses to EPA's comments on Appendix D HASP with ERP of the Draft PDI WP and the Revised Draft PDI WP are acceptable. However, EPA submits the following clarifications and supplemental information for the Willbridge Cove Group's response to EPA's Appendix D Specific Comments. *Willbridge Cove Group's Response (December 10, 2020)* The HSP is primarily an internal document with specific language and procedures developed by Jacobs H&S professionals. Most of the suggested changes have been incorporated into the revised document, but in some cases, the AHAs and protocols suggested by EPA are the purview of our subcontractors and cannot be provided until the work is contracted. *EPA General Responsiveness Comment (January 21, 2021)* EPA expects to be provided any Activity Hazard Analysis and/or HASP updates prior to the start of field work. This includes the Dive Safety Plan, which according to the PDI WP will be produced prior to mobilization of the sediment sampling program and will be attached to the HASP. Note that EPA does not approve HASPs, but EPA comments should be addressed.	Activity hazard analyses (AHAs) and any applicable HASP updates will be provided to EPA prior to field mobilization. The HASP has not been updated at this time.
EPA Comments on November 20, 2020 Comments to Willbridge Cove Group	After the November 18, 2020 teleconference meeting with the Willbridge Cove Group, EPA sent two comments via email to the Willbridge Cove Group on November 20, 2020 pertaining to buried contamination and data replacement. EPA submits the following clarifications and supplemental information for the Willbridge Cove Group's response to EPA's comments provided on November 20, 2020.	No response required
	EPA Comment (November 20, 2020) **Buried Contamination:** As stated in the Remedial Design Guidelines, whether or not buried contamination areas will be included in an SMA is dependent on the chemical and physical stability of the buried material and reasonably anticipated future uses. Stability determinations will be based on, but not limited to the following factors: erosion/scour potential; chemical concentration compared to RAL thresholds; depth of contamination; and advective transport. The Guidelines also state that fate and transport modeling will be required to support these evaluations. EPA is concerned that there is currently not enough information to make a buried contamination stability conclusion for the RAL exceedances proposed in our call. Because the primary data quality objective of the PDI is SMA delineation, it is premature to exclude known RAL or PTW exceedances from PDI sampling based on an anticipated buried contamination conclusion that is expected to be evaluated and determined as part of the BODR. Therefore, we recommend the inclusion of additional sample points near the known subsurface RAL and PTW exceedances. *Willbridge Cove Group's Response (December 10, 2020)* The PDI Work Plan has been revised to include a physical/chemistry stability DQO (DQO 2) and describes the data requirements and evaluation process in Section 1.4.2 and the results of the related data gaps evaluations in Section 2.4. The data gaps evaluations conclude that the existing and planned PDI sampling data will be sufficient to characterize the chemical and physical stability of at-depth contamination in areas within and outside of the preliminary SMAs. No additional sampling specific to stability evaluations is needed. The combination of exiting data and data proposed to address DQO 1 will be sufficient to address DQO 2. *EPA Response (January 21, 2021)* EPA's conditional approval of the framework for evaluating physical and chemical stability as presented in DQO 2 in the Revised Draft PDI WP and modified by the above comments should not be considered an approval of the results of the evaluation in future RD deliverables.	Comment acknowledged. An addendum to the revised draft PDI Work Plan will be prepared and submitted to document the changes to the PDI sampling and analysis approach noted in previous responses. This addendum will include revised figures and the revised supporting tables with sampling, archiving, and analytical schedules. The framework for the physical and chemical stability evaluation will be more fully developed prior to the presentation of the PDI results.

Comment ID	Comment & Response History/**Comment**	**Response**
	EPA Comment (November 20, 2020) **Data Replacement.** We understand that the Willbridge Cove Group intends to use their forthcoming PDI data as well as the Pre-RD data as part of their surface sediment data replacement strategy. This is an acceptable approach but will require additional PDI surface sediment data than what was presented to EPA yesterday (11/18/2020) to achieve an adequate sample density to facilitate data replacement. The 150- by 150-foot sediment sampling grid is applicable for SMA delineation and is intended to augment RI/FS data to proceed towards the BODR with anticipation of higher resolution data to be collected for remedial design and data replacement purposes. As mentioned in the EPA comments, RI/FS data should only be replaced when concentrations have changed substantively over time and when spatial resolution is at least maintained or improved. EPA recommends the collection of additional samples to provide greater spatial density for the Willbridge Cove Group's future data replacement strategy. *Willbridge Cove Group's Response (December 10, 2020)* Section 1.4.1 added to describe the data replacement review process and includes references to EPA RD and data replacement guidance. Sampling designs in Section 3 have been revised to include additional grab surface sediment samples to provide greater spatial density in the three locations where data replacement is under consideration. Table 3-2 (now 3-1) was updated to reflect the revised sampling approach and identify which existing data may be replaced by newer data. *EPA Response (January 21, 2021)* EPA's conditional approval of the framework for evaluating data replacement as presented in PDI WP Section 1.4.1 should not be considered an approval of the results of the evaluation in future RD deliverables.	Comment acknowledged.

Appendix A
Analytical Data Tables

Appendix A. Sediment Data Tables

This appendix presents the data used to summarize the nature and extent in surface sediments within and near Willbridge Cove.

These data were compiled from the following U.S. Environmental Protection Agency-approved databases or tables downloaded from the Portland Harbor Data Portal (http://ph-public-data.com/):

- Feasibility Study Database (http://ph-public-data.com/document/CDMSmith2018/)
- Portland Harbor Pre-Design Investigation-Baseline (Pre-RD) Sample Database (http://ph-public-data.com/document/PHRD_2019/)
- Summary tables of the available surface sediment data used are as follows:
  - Table A-1—Feasibility Study (FS) Database Results for Willbridge Cove Area and Vicinity—Record of Decision (ROD) Table 17 and ROD Table 21 Contaminants of Concern (COCs) and Physical Parameters.
  - Table A-2—Pre-RD Results for Willbridge Cove Area and Vicinity—ROD Table 17 and ROD Table 21 COCs and Physical Parameters.
  - Tables A-3A through A-3D—River Bank Soil Results.

The summations included in the FS and Pre-RD databases follow the rules established during the Feasibility Study, which generally included non-detected results at half the reporting limit in the totaled results.

Where duplicate or split samples were collected from the identical location and sampling interval, the following rules were used for determining how values would be mapped:

- Two detected results—average value presented
- Detected and a non-detect result—detected value presented
- Both values non-detected—parameter mapped as a non-detect
- Rejected result and a non-rejected result—the non-rejected result was used.

Field replicates collected from slightly different locations are shown as separate samples, consistent with how the Remedial Investigation and FS treated those replicates.

Table A-1. RI/FS Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

CAS			Sample	Sample		Start	End	Depth			Elevation	1,2,3,4,7,8-Hexachlorodibenzofuran		1,2,3,7,8-Pentachlorodibenzo-p-dioxin		2,3,4,7,8-Pentachlorodibenzofuran		2,3,7,8-Tetrachlorodibenzofuran		2,3,7,8-Tetrachlorodibenzo-p-dioxin		Aldrin		Arsenic		Bis(2-ethylhexyl) phthalate		Cadmium							
												70648-26-9		40321-76-4		57117-31-4		51207-31-9		1746-01-6		309-00-2		7440-38-2		117-81-7		7440-43-9							
Matrix	Location ID	Sample Name	Type	Date	Depth Class	Depth	Depth	Units	X Coordinate	Y coordinate	(NAVD88)	µg/kg		µg/kg		µg/kg		µg/kg		µg/kg		µg/kg		mg/kg		µg/kg		mg/kg							
											Sediment/Bank Soil CUL	**0.0004**		**0.0002**		**0.0003**		**0.0004**		**0.0002**		**2**		**3**		**135**		**0.51**							
											Sitewide RAL	**NA**		**0.0008**		**0.2**		**NA**		**0.0006**		**--**		**--**		**--**		**--**							
											PTW Threshold	**0.04**		**0.01**		**0.2**		**0.6**		**0.01**		**--**		**--**		**--**		**--**							
SE	LW2-B019	LW2-B019	N	7/26/2004	Surface Sediment	0	15	cm	7628948	700051	9	***0.000706***	***J***	0.000197	J	0.000141	U	0.000108	J	0.00004	J	0.0281	UJ	***3.82***		14	U	0.11							
SE	LW2-C377	LW2-C377-B	N	11/10/2004	Subsurface Sediment	30	153	cm	7628521	701143	-5	0.000745	U	0.000045	U	0.000191	J	***0.000494***		0.000045	J	0.0411	UJ	***3.53***	***J***	94	U	0.098							
SE	LW2-C377	LW2-C377-C	N	11/10/2004	Subsurface Sediment	153	273	cm	7628521	701143	-5	***0.002695***		0.000132	J	***0.001002***	***J***	***0.002318***		0.000067	J	0.218	UJ	***4.38***	***J***	240	U	0.146							
SE	LW2-C377	LW2-C377-D	N	11/10/2004	Subsurface Sediment	273	369	cm	7628521	701143	-5	***0.0561***		***0.000489***	***J***	***0.00614***		***0.00658***		***0.000296***	***J***	--		--		--		--							
SE	LW2-C377	LW2-C377-E	N	11/10/2004	Subsurface Sediment	369	482	cm	7628521	701143	-5	***0.02375***		***0.001461***		***0.004628***		***0.006994***		***0.000297***		0.391	UJ	***3.81***	***J***	62	U	0.498							
SE	LW2-C401	LW2-C401-B	N	11/10/2004	Subsurface Sediment	30	69	cm	7629031	700153	-10	--		--		--		--		--		0.185	UJ	***5.43***	***J***	300	U	***2.07***							
SE	LW2-C401	LW2-C401-C	N	11/10/2004	Subsurface Sediment	69	188	cm	7629031	700153	-10	--		--		--		--		--		0.158	UJ	***4.79***	***J***	210	U	0.217							
SE	LW2-C401	LW2-C401-D	N	11/10/2004	Subsurface Sediment	188	278	cm	7629031	700153	-10	--		--		--		--		--		0.155	UJ	***3.6***	***J***	190	U	0.177							
SE	LW2-C401	LW2-C401-E	N	11/10/2004	Subsurface Sediment	278	367	cm	7629031	700153	-10	--		--		--		--		--		0.183	UJ	***4.78***	***J***	150	U	0.274							
SE	LW2-C401	LW2-C401-F	N	11/10/2004	Subsurface Sediment	367	504	cm	7629031	700153	-10	--		--		--		--		--		0.14	UJ	--		--		--							
SE	LW2-C403	LW2-C403-B	N	11/10/2004	Subsurface Sediment	30	153	cm	7629841	700123	-6	0.000153	U	0.000042	J	0.000066	U	0.000087	U	0.000017	U	0.0327	UJ	2.35	J	10	U	0.123	J						
SE	LW2-C403	LW2-C403-C	N	11/10/2004	Subsurface Sediment	153	242	cm	7629841	700123	-6	0.000016	U	0.000011	U	0.000007	U	0.00002	U	0.000016	U	0.0327	UJ	2.4	J	7.7	U	0.056	J						
SE	LW2-C403	LW2-C403-D	N	11/10/2004	Subsurface Sediment	242	361	cm	7629841	700123	-6	0.000007	U	0.00001	U	0.00001	U	0.000021	U	0.000026	U	0.037	UJ	2.88	J	15	U	0.136	J						
SE	LW2-C403	LW2-C403-F	N	11/10/2004	Subsurface Sediment	483	533	cm	7629841	700123	-6	--		--		--		--		--		--		***3.2***		--		0.232							
SE	LW2-C413	LW2-C413-B1	N	10/26/2004	Subsurface Sediment	30	158	cm	7630354	699540	2	--		--		--		--		--		0.217	UJ	***4.78***	***J***	35	U	0.407							
SE	LW2-C413	LW2-C413-C1	N	10/26/2004	Subsurface Sediment	158	299	cm	7630354	699540	2	--		--		--		--		--		0.207	UJ	***3.61***	***J***	***650***		0.454							
SE	LW2-C413-2	LW2-C413-B2	FR	10/26/2004	Subsurface Sediment	30	152	cm	7630359	699540	1	--		--		--		--		--		0.204	UJ	***4.11***	***J***	76		0.441							
SE	LW2-C413-2	LW2-C413-C2	FR	10/26/2004	Subsurface Sediment	152	274	cm	7630359	699540	1	--		--		--		--		--		0.211	UJ	***4.39***	***J***	42	U	0.466							
SE	LW2-C413-2	LW2-C413-D2	N	10/26/2004	Subsurface Sediment	274	349	cm	7630359	699540	1	--		--		--		--		--		0.203	UJ	***3.63***	***JT***	18	J	0.41	JT						
SE	LW2-C532	LW2-C532-A	N	10/18/2005	Surface Sediment	0	30	cm	7629257	700289	-21	--		--		--		--		--		0.64	NJ	***5.01***		24	U	0.413							
SE	LW2-C532	LW2-C532-B	N	10/18/2005	Subsurface Sediment	30	99	cm	7629257	700289	-21	--		--		--		--		--		0.2	UJ	***3.85***		15	U	0.362							
SE	LW2-C532	LW2-C532-C	N	10/18/2005	Subsurface Sediment	99	198	cm	7629257	700289	-21	--		--		--		--		--		0.17	UJ	***4.74***		17	U	***0.535***							
SE	LW2-C532	LW2-C532-D	N	10/18/2005	Subsurface Sediment	198	293	cm	7629257	700289	-21	--		--		--		--		--		--		--		--		--							
SE	LW2-C532	LW2-C532-E	N	10/18/2005	Subsurface Sediment	293	338	cm	7629257	700289	-21	--		--		--		--		--		0.16	UJ	***5.08***		15	U	***0.642***							
SE	LW2-C532	LW2-C532-F	N	10/18/2005	Subsurface Sediment	338	372	cm	7629257	700289	-21	--		--		--		--		--		0.13	UJ	***4.18***		4.1	U	0.208							
SE	LW2-G389	LW2-G389	N	10/29/2004	Surface Sediment	0	20	cm	7628548	700673	4	--		--		--		--		--		0.0375	UJ	1.89		6.9	UJ	0.101	U						
SE	LW2-G394	LW2-G394	N	8/27/2004	Surface Sediment	0	25	cm	7628836	700366	-25	--		--		--		--		--		0.972	J	***4.23***		150	U	***0.731***	***J***						
SE	LW2-G399	LW2-G399	N	8/27/2004	Surface Sediment	0	22	cm	7629573	700201	-1	--		--		--		--		--		0.182	UJ	***5.38***		67		0.202	J						
SE	LW2-G401	LW2-G401	N	10/22/2004	Surface Sediment	0	27	cm	7629036	700154	-10	--		--		--		--		--		0.218	UJ	***4.54***		***290***		0.339							
SE	LW2-G403	LW2-G403	N	10/22/2004	Surface Sediment	0	26	cm	7629824	700114	-4	0.000171	U	0.000024	U	0.00006	J	0.000093	U	0.00002	U	0.0322	UJ	***3.72***		20	U	0.116							
SE	LW2-G404	LW2-G404	N	8/27/2004	Surface Sediment	0	25	cm	7629091	700096	-9	--		--		--		--		--		0.721	NJ	***4.24***		110		0.274	J						
SE	LW2-G407	LW2-G407	N	9/9/2004	Surface Sediment	0	29	cm	7630154	699834	-12	--		--		--		--		--		0.0429	UJ	***3.53***	***JT***	64	UJ	0.271	T						
SE	LW2-G413	LW2-G413	N	10/22/2004	Surface Sediment	0	27	cm	7630351	699536	2	--		--		--		--		--		0.0305	UJ	2.39		78		0.271							
SE	LW2-GBT020	LW2-GBT020	N	12/6/2005	Surface Sediment	0	10	cm	7628495	701060	-13	***0.00278***		***0.000494***	***J***	***0.000748***	***J***	***0.001313***		***0.000226***	***J***	0.0655	J	***3.49***		30	U	0.248							
SE	LW2-GBT021	LW2-GBT021	N	12/13/2005	Surface Sediment	0	10	cm	7629274	700117	-6	0.000156	J	0.000053	J	0.00007	J	0.000256	J	0.000011	U	0.047	J	***4.25***		45	U	0.173							
SE	LW2-NA3A	NA-3A-0005	N	10/22/2004	Surface Sediment	0	5	cm	7629429	700328	-21	--		--		--		--		--		--		2.8		--		0.176							
SE	LW2-NA3A	NA-3A-0508	N	10/22/2004	Subsurface Sediment	5	8	cm	7629429	700328	-21	--		--		--		--		--		--		***3.03***		--		0.17							
SE	LW2-NA3A	NA-3A-0812	N	10/22/2004	Subsurface Sediment	8	12	cm	7629429	700328	-21	--		--		--		--		--		--		***4.79***		--		0.315							
SE	LW2-NA3A	NA-3A-1216	N	10/22/2004	Subsurface Sediment	12	16	cm	7629429	700328	-21	--		--		--		--		--		--		***6.33***		--		0.418							
SE	LW2-NA3A	NA-3A-1620	N	10/22/2004	Subsurface Sediment	16	20	cm	7629429	700328	-21	--		--		--		--		--		--		***4.52***		--		0.309							
SE	LW2-NA3A	NA-3A-2024	N	10/22/2004	Subsurface Sediment	20	24	cm	7629429	700328	-21	--		--		--		--		--		--		2.84	J	--		0.194							
SE	LW2-NA3A	NA-3A-2428	N	10/22/2004	Subsurface Sediment	24	28	cm	7629429	700328	-21	--		--		--		--		--		--		***3.9***	***J***	--		0.265							
SE	LW2-NA3A	NA-3A-2832	N	10/22/2004	Subsurface Sediment	28	32	cm	7629429	700328	-21	--		--		--		--		--		--		2.57	J	--		0.167							
SE	LW2-NA3A	NA-3A-3238	N	10/22/2004	Subsurface Sediment	32	38	cm	7629429	700328	-21	--		--		--		--		--		--		2.45	JT	--		0.159	T						
SE	LW2-NA3A	NA-3A-3844	N	10/22/2004	Subsurface Sediment	38	44	cm	7629429	700328	-21	--		--		--		--		--		--		***3.16***	***J***	--		0.224							
SE	LW2-NA3A	NA-3A-4854	N	10/22/2004	Subsurface Sediment	48	54	cm	7629429	700328	-21	--		--		--		--		--		--		***4.03***	***J***	--		0.29							
SE	LW2-NA3A	NA-3A-5864	N	10/22/2004	Subsurface Sediment	58	64	cm	7629429	700328	-21	--		--		--		--		--		--		***4.35***	***J***	--		0.31							
SE	LW2-NA3A	NA-3A-6874	N	10/22/2004	Subsurface Sediment	68	74	cm	7629429	700328	-21	--		--		--		--		--		--		***4.42***	***J***	--		0.409							
SE	LW2-NA3A	NA-3A-7884	N	10/22/2004	Subsurface Sediment	78	84	cm	7629429	700328	-21	--		--		--		--		--		--		***4.79***	***J***	--		***0.547***							
SE	LW2-NA3A	NA-3A-8894	N	10/22/2004	Subsurface Sediment	88	94	cm	7629429	700328	-21	--		--		--		--		--		--		***4.07***	***J***	--		0.346							
SE	LW2-NA3B	NA-3B-0034	N	10/22/2004	Surface Sediment	0	34	cm	7629433	700322	-20	--		--		--		--		--		0.0363	UJ	--		66	U	--							
SE	LW2-NA3B	NA-3B-3464	N	10/22/2004	Subsurface Sediment	34	64	cm	7629433	700322	-20	--		--		--		--		--		0.0343	UJ	--		22	U	--							
SE	LW2-NA3B	NA-3B-6494	N	10/22/2004	Subsurface Sediment	64	94	cm	7629433	700322	-20	--		--		--		--		--		0.0371	UJ	--		18	U	--							
SE	LW3-C688	LW3-C688-B	N	1/15/2008	Subsurface Sediment	30	114	cm	7628760	701367	-5	***0.00286***	***J***	***0.000273***	***J***	***0.00035***	***J***	***0.000723***		0.000155	U	0.27		***3.52***		51		0.179							
SE	LW3-C688	LW3-C688-C	N	1/15/2008	Subsurface Sediment	114	221	cm	7628760	701367	-5	***0.00536***		***0.000211***	***J***	***0.000758***	***J***	***0.00147***		0.0000754	U	0.6	U	***3.85***		93		0.288							
SE	LW3-C688	LW3-C688-D	N	1/15/2008	Subsurface Sediment	221	281	cm	7628760	701367	-5	***0.0126***		***0.000576***	***J***	***0.00208***	***J***	***0.00267***		***0.000355***	***J***	0.6	U	***3.79***		***170***	***J***	0.245							
SE	LW3-C688	LW3-C688-F	N	1/15/2008	Subsurface Sediment	404	457	cm	7628760	701367	-5	***0.025***		***0.00122***	***J***	***0.00572***		***0.0045***		***0.000594***		0.6	U	***3.91***		350	U	***0.534***							
SE	LW3-C690	LW3-C690-B	N	1/15/2008	Subsurface Sediment	30	151	cm	7628470	701000	-11	***0.00066***	***J***	0.0000146	U	***0.000647***	***J***	***0.00168***		0.00000801	U	0.12	U	2.95		86	J	0.18							
SE	LW3-C690	LW3-C690-C	N	1/15/2008	Subsurface Sediment	151	274	cm	7628470	701000	-11	***0.00556***		***0.000412***	***J***	***0.00134***	***J***	***0.00327***		0.000174	J	0.12	U	***3.95***		***180***	***J***	0.282							
SE	LW3-C690	LW3-C690-D	N	1/15/2008	Subsurface Sediment	274	346	cm	7628470	701000	-11	***0.0427***		***0.000809***	***J***	***0.00961***		***0.0128***		***0.000441***	***J***	0.6	U	***4.04***		350	U	0.491							
SE	LW3-C690	LW3-C690-E	N	1/15/2008	Subsurface Sediment	346	471	cm	7628470	701000	-11	***0.0565***		***0.00741***		***0.0128***		***0.0199***		***0.00117***		3.6	U	***3.83***		700	U	0.497							
SE	LW3-C695	LW3-C695-B	N	12/20/2007	Subsurface Sediment	30	95	cm	7629190	700056	-6	--		--		--		--		--		0.12	U	***5.5***		***250***		0.355							
SE	LW3-C695	LW3-C695-D	N	12/20/2007	Subsurface Sediment	221	343	cm	7629190	700056	-6	--		--		--		--		--		0.87	U	***5.21***		***450***		0.401							
SE	LW3-C695	LW3-C695-E	N	12/20/2007	Subsurface Sediment	343	384	cm	7629190	700056	-6	--		--		--		--		--		0.84	U	***4.06***		***180***	***J***	0.398							
SE	LW3-C698	LW3-C698-B	N	12/20/2007	Subsurface Sediment	30	152	cm	7630300	699718	-16	--		--		--		--		--		0.12	U	***3.68***		82		0.277							
SE	LW3-G692-1	LW3-G692-1	N	12/4/2007	Surface Sediment	0	30	cm	7628730	701055	-20	***0.000711***	***J***	0.0000181	U	0.000185	J	***0.000656***	***J***	0.0000155	U	0.14	U	***3.01***		***140***	***J***	0.264							
SE	LW3-G692-2	LW3-G692-2	FR	12/4/2007	Surface Sediment	0	28	cm	7628730	701048	-20	***0.00226***	***JT***	0.000154	JT	***0.000569***	***JT***	***0.00092***	***T***	0.000019	UT	0.22	UT	2.83	T	110	JT	0.259	T						
SE	LW3-G694	LW3-G694	N	11/16/2007	Surface Sediment	0	20	cm	7629340	700318	-23	--		--		--		--		--		0.12	U	***3.49***	***J***	34		0.226							
SE	LW3-GSP07W	LW3-GSP07W	N	10/15/2007	Surface Sediment	0	19	cm	7628812	700370	-14	0.000381	J	0.0000989	U	0.0000859	U	0.000184	U	0.000109	U	0.12	U	2.09		25	U	0.081	U						

Table A-1. RI/FS Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

CAS													1,2,3,4,7,8-Hexachlorodibenzofuran		1,2,3,7,8-Pentachlorodibenzo-p-dioxin		2,3,4,7,8-Pentachlorodibenzofuran		2,3,7,8-Tetrachlorodibenzofuran		2,3,7,8-Tetrachlorodibenzo-p-dioxin		Aldrin		Arsenic		Bis(2-ethylhexyl) phthalate		Cadmium	
			Sample	Sample		Start	End	Depth			Elevation		70648-26-9		40321-76-4		57117-31-4		51207-31-9		1746-01-6		309-00-2		7440-38-2		117-81-7		7440-43-9	
Matrix	Location ID	Sample Name	Type	Date	Depth Class	Depth	Depth	Units	X Coordinate	Y coordinate	(NAVD88)		µg/kg		µg/kg		µg/kg		µg/kg		µg/kg		µg/kg		mg/kg		µg/kg		mg/kg	
											Sediment/Bank Soil CUL		**0.0004**		**0.0002**		**0.0003**		**0.0004**		**0.0002**		**2**		**3**		**135**		**0.51**	
											Sitewide RAL		**NA**		**0.0008**		**0.2**		**NA**		**0.0006**		**--**		**--**		**--**		**--**	
											PTW Threshold		**0.04**		**0.01**		**0.2**		**0.6**		**0.01**		**--**		**--**		**--**		**--**	
SE	LWG0108R002SDS015C00	LWG0108R002SDS015C00	N	10/17/2002	Surface Sediment	0	15	cm	7630259	699666	1		--		--		--		--		--		0.19	U	2.7		100	U	0.17	
SE	TOSC099DMMU1	TOSC099DMMU1SDO1	N	1/21/1999	Subsurface Sediment	0	304	cm	7629016	700391	-24		--		--		--		--		--		2	U	***5***	***J***	***370***		1	U
SE	TOSC099DMMU1	TOSC099DMMU1SDO3	FR	1/21/1999	Subsurface Sediment	0	304	cm	7629016	700391	-24		--		--		--		--		--		2	UJ	***5***	***J***	***420***		1	U
SE	TOSC099DMMU2	TOSC099DMMU2SDO4	N	1/21/1999	Subsurface Sediment	0	164	cm	7629278	700566	-29		--		--		--		--		--		2	U	***4***	***J***	***140***		1	U
SE	W-09-A-PG	LWG2-PG-W9A	N	11/30/2005	Surface Sediment	0	23	cm	7628899	700250	3		--		--		--		--		--		--		2.32		6.7	U	0.121	
SE	W-09-C-PG	LWG2-PG-W9C	N	11/30/2005	Surface Sediment	0	30	cm	7628988	700306	-20		--		--		--		--		--		--		***3.52***		110	J	0.284	
SE	WLCASF97S001	WLCASF97S001W4159	N	6/12/1997	Surface Sediment	0	10	cm	7630379	699598	-9		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S002	WLCASF97S002W4158	N	6/12/1997	Surface Sediment	0	10	cm	7630047	700105	-32		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S003	WLCASF97S003W4157	N	6/12/1997	Surface Sediment	0	10	cm	7629493	700297	-17		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S004	WLCASF97S004W4156	N	6/12/1997	Surface Sediment	0	10	cm	7629322	700277	-23		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S005	WLCASF97S005W4155	N	6/12/1997	Surface Sediment	0	10	cm	7629019	700128	-5		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S006	WLCASF97S006W4154	N	6/12/1997	Surface Sediment	0	10	cm	7628791	700365	-2		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S008	WLCASF97S008W4152	N	6/12/1997	Surface Sediment	0	10	cm	7629266	700704	-35		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S009	WLCASF97S009W4172	N	6/11/1997	Surface Sediment	0	10	cm	7628648	700612	-32		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S010	WLCASF97S010W4171	N	6/11/1997	Surface Sediment	0	10	cm	7629081	700977	-34		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S011	WLCASF97S011W4170	N	6/11/1997	Surface Sediment	0	10	cm	7628514	700853	-19		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S012	WLCASF97S012W4168	N	6/11/1997	Surface Sediment	0	10	cm	7628858	701245	-20		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S012	WLCASF97S012W4169	FR	6/11/1997	Surface Sediment	0	10	cm	7628858	701245	-20		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08ANCTPD03	WLCCWI08ANCTPD03	N	5/29/2009	Surface Sediment	0	2	cm	7629152	700952	-39		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08ANCTPD06	WLCCWI08ANCTPD06	N	5/29/2009	Surface Sediment	0	2	cm	7629255	700767	-38		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU1A	WLCCWI08DMMU1A	N	5/29/2009	Surface Sediment	0	2	cm	7628747	700605	-35		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU1A	WLCCWI08DMMU1A4041	N	9/2/2008	Subsurface Sediment	91	121	cm	7628747	700605	-35		--		--		--		--		--		--		2.01	UJ	***6.1***		***189***	***J***	0.265	
SE	WLCCWI08DMMU1A	WLCCWI08DMMU1A4142	N	9/2/2008	Subsurface Sediment	121	152	cm	7628747	700605	-35		--		--		--		--		--		--		2.45	UJ	***5.76***	***J***	7,310	UJ	0.919	UJ
SE	WLCCWI08DMMU1ABC	WLCCWI08DMMU1ABC	N	9/4/2008	Subsurface Sediment	91	182	cm	7628839	700680	-33		--		--		--		--		--		--		2.26	UJT	***5.36***	***T***	***183***	***JT***	0.226	T
SE	WLCCWI08DMMU1B	WLCCWI08DMMU1B	N	5/29/2009	Surface Sediment	0	2	cm	7628834	700677	-33		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU1B	WLCCWI08DMMU1B4041	N	9/3/2008	Subsurface Sediment	121	152	cm	7628834	700677	-33		--		--		--		--		--		--		1.99	UJ	***5.13***		***180***	***J***	0.245	
SE	WLCCWI08DMMU1B	WLCCWI08DMMU1B4142	N	9/3/2008	Subsurface Sediment	152	182	cm	7628834	700677	-33		--		--		--		--		--		--		2.34	UJ	***4.79***	***J***	6,930	UJ	0.857	UJ
SE	WLCCWI08DMMU1C	WLCCWI08DMMU1C	N	5/29/2009	Surface Sediment	0	5	cm	7628936	700759	-33		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU1C	WLCCWI08DMMU1C4041	N	9/4/2008	Subsurface Sediment	91	121	cm	7628936	700759	-33		--		--		--		--		--		--		1.98	UJ	***5.9***		***148***	***J***	0.248	
SE	WLCCWI08DMMU1C	WLCCWI08DMMU1C4142	N	9/4/2008	Subsurface Sediment	121	152	cm	7628936	700759	-33		--		--		--		--		--		--		2.33	UJ	***4.58***	***J***	186	UJ	0.874	UJ
SE	WLCCWI08DMMU2A	WLCCWI08DMMU2A	N	5/29/2009	Surface Sediment	0	2	cm	7628879	700461	-33		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU2A	WLCCWI08DMMU2A4041	N	9/5/2008	Subsurface Sediment	152	182	cm	7628879	700461	-33		--		--		--		--		--		--		1.94	U	***5.54***		***172***	***J***	0.232	J
SE	WLCCWI08DMMU2A	WLCCWI08DMMU2A4142	N	9/5/2008	Subsurface Sediment	182	213	cm	7628879	700461	-33		--		--		--		--		--		--		2.5	U	***5.57***		7,440	U	0.92	U
SE	WLCCWI08DMMU2B	WLCCWI08DMMU2B	N	5/29/2009	Surface Sediment	0	2	cm	7628979	700542	-33		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU2B	WLCCWI08DMMU2B4041	N	9/5/2008	Subsurface Sediment	91	121	cm	7628979	700542	-33		--		--		--		--		--		--		1.92	U	***5.34***		***166***	***J***	0.254	J
SE	WLCCWI08DMMU2B	WLCCWI08DMMU2B4142	N	9/5/2008	Subsurface Sediment	121	152	cm	7628979	700542	-33		--		--		--		--		--		--		2.39	UJ	***5.76***	***J***	***191***	***J***	0.886	UJ
SE	WLCCWI08DMMU2C	WLCCWI08DMMU2C	N	5/29/2009	Surface Sediment	0	2	cm	7629098	700580	-33		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU2C	WLCCWI08DMMU2C4041	N	9/5/2008	Subsurface Sediment	60	91	cm	7629098	700580	-33		--		--		--		--		--		--		2.28	U	***5.77***		***203***	***J***	0.23	J
SE	WLCCWI08DMMU2C	WLCCWI08DMMU2C4142	N	9/5/2008	Subsurface Sediment	91	121	cm	7629098	700580	-33		--		--		--		--		--		--		2.34	UJ	***4.98***	***J***	7,010	UJ	0.888	UJ
SE	WLCCWI08DMMU3A	WLCCWI08DMMU3A	N	5/29/2009	Surface Sediment	0	2	cm	7628650	700651	-24		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU3A	WLCCWI08DMMU3A13	N	9/8/2008	Subsurface Sediment	30	91	cm	7628650	700651	-24		--		--		--		--		--		--		2.53	UJ	***4.96***	***J***	3,760	UJ	0.939	UJ
SE	WLCCWI08DMMU3B	WLCCWI08DMMU3B	N	5/29/2009	Surface Sediment	0	2	cm	7628779	700408	-18		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU3B	WLCCWI08DMMU3B13	N	9/8/2008	Subsurface Sediment	30	91	cm	7628779	700408	-18		--		--		--		--		--		--		4.11	UJ	***6***		328	U	0.606	U
SE	WLCCWI08DMMU3D	WLCCWI08DMMU3D	N	5/29/2009	Surface Sediment	0	2	cm	7628873	700325	-13		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU3D	WLCCWI08DMMU3D13	N	9/8/2008	Subsurface Sediment	30	91	cm	7628873	700325	-13		--		--		--		--		--		--		1.57	UJ	***5.29***	***J***	4,690	UJ	0.577	UJ
SE	WLCCWI08DMMU3E	WLCCWI08DMMU3E	N	5/29/2009	Surface Sediment	0	2	cm	7628981	700402	-24		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU3E	WLCCWI08DMMU3E13	N	9/5/2008	Subsurface Sediment	30	91	cm	7628981	700402	-24		--		--		--		--		--		--		2.35	U	***4.85***		188	U	0.875	U
SE	WLCDRD05PG074	WLCDRD05PG07474	N	5/25/2005	Surface Sediment	0	30	cm	7628952	701290	-24		--		--		--		--		--		--		0.25	U	***3.92***		35	U	0.287	
SE	WLCOFJ022201	WLCOFJ02220122010	N	10/21/2002	Surface Sediment	0	10	cm	7629006	700111	-2		--		--		--		--		--		--		1.07	UJ	***3.47***		164	U	0.00177	U
SE	WLCOFJ022202	WLCOFJ02220222020	N	10/21/2002	Surface Sediment	0	15	cm	7629048	700077	-3		--		--		--		--		--		--		1.23	UJ	***4.27***		113	J	0.0156	J
SE	WLCOFJ022203	WLCOFJ02220322030	N	10/21/2002	Surface Sediment	0	15	cm	7628994	700157	-4		--		--		--		--		--		--		1.24	UJ	***3.8***		***622***		0.00199	U
SE	WLCOFJ0222031	WLCOFJ022203122031	FR	10/21/2002	Surface Sediment	0	15	cm	7628991	700159	-4		--		--		--		--		--		--		1.24	UJ	***3.91***		***234***	***J***	0.00182	U
SE	WLCOFJ022204	WLCOFJ02220422040	N	10/21/2002	Surface Sediment	0	12	cm	7629090	700117	-16		--		--		--		--		--		--		1.73	UJ	***4.26***		279	U	0.00292	U
SE	WLCOFJ022205	WLCOFJ02220522050	N	10/21/2002	Surface Sediment	0	12	cm	7629070	700151	-18		--		--		--		--		--		--		1.79	UJ	***4.29***		***263***	***J***	0.00262	U
SE	WLCOFJ022206	WLCOFJ02220622060	N	10/21/2002	Surface Sediment	0	10	cm	7629040	700177	-19		--		--		--		--		--		--		1.62	UJ	***3.88***		***369***	***J***	0.00266	U
SE	WLCPWL09APS-1	WLCPWL09APS-1B	N	12/29/2009	Subsurface Sediment	60	121	cm	7628973	700319	-21		--		--		--		--		--		--		0.97	UJ	***3.99***		***140***	***J***	0.353	
SE	WLCPWL09APS-1	WLCPWL09APS-1T	N	12/29/2009	Subsurface Sediment	0	60	cm	7628973	700319	-21		--		--		--		--		--		--		1	UJ	***3.63***		76	J	0.239	
SE	WLCPWL09APS-2	WLCPWL09APS-2B	N	12/29/2009	Subsurface Sediment	60	121	cm	7629116	700296	-15		--		--		--		--		--		--		0.9	UJ	***3.63***		88	J	0.261	
SE	WLCPWL09APS-2	WLCPWL09APS-2T	N	12/29/2009	Subsurface Sediment	0	60	cm	7629116	700296	-15		--		--		--		--		--		--		1.1	UJ	***3.73***		***430***	***J***	0.281	
SE	WLCPWL09APS-3	WLCPWL09APS-3B	N	12/29/2009	Subsurface Sediment	60	121	cm	7629238	700264	-20		--		--		--		--		--		--		0.88	UJ	***3.94***		20	J	0.321	
SE	WLCPWL09APS-3	WLCPWL09APS-3T	N	12/29/2009	Subsurface Sediment	0	60	cm	7629238	700264	-20		--		--		--		--		--		--		0.9	UJ	***4.28***		40	J	0.337	
SE	WLCPWL09DRB-1	WLCPWL09DRB-1Z	N	12/29/2009	Subsurface Sediment	320	350	cm	7629031	700383	-22		--		--		--		--		--		--		0.86	UJ	***3.68***		130	J	0.474	
SE	WLCPWL09DRB-2	WLCPWL09DRB-2Z	N	12/29/2009	Subsurface Sediment	307	338	cm	7629105	700447	-25		--		--		--		--		--		--		0.86	UJ	***3.49***		110	J	0.418	
SE	WLCPWL09DRB-3	WLCPWL09DRB-3Z	N	12/29/2009	Subsurface Sediment	280	310	cm	7629193	700495	-25		--		--		--		--		--		--		0.89	UJ	***3.52***		130	J	0.433	
SE	WLCPWL09DRB-D	WLCPWL09DRB-DP	N	12/29/2009	Subsurface Sediment	0	302	cm	7629110	700442	-24		--		--		--		--		--		--		0.94	UJ	***3.88***		130	J	0.358	
SE	WLCPWL09URB-1	WLCPWL09URB-1Z	N	12/29/2009	Subsurface Sediment	295	326	cm	7629179	700364	-17		--		--		--		--		--		--		0.9	UJ	***4.06***		83	J	***0.521***	
SE	WLCPWL09URB-2	WLCPWL09URB-2Z	N	12/29/2009	Subsurface Sediment	118	149	cm	7629295	700478	-23		--		--		--		--		--		--		0.88	UJ	***3.94***		110	J	***0.592***	
SE	WLCPWL09URB-3	WLCPWL09URB-3Z	N	12/29/2009	Subsurface Sediment	167	198	cm	7629268	700417	-21		--		--		--		--		--		--		0.88	UJ	***3.88***		45	J	0.487	
SE	WLCPWL09URB-D	WLCPWL09URB-DP	N	12/29/2009	Subsurface Sediment	0	194	cm	7629248	700420	-20		--		--		--		--		--		--		0.91	UJT	***3.86***	***T***	76	JT	0.349	T

Table A-1. RI/FS Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

CAS													1,2,3,4,7,8-Hexachlorodibenzofuran		1,2,3,7,8-Pentachlorodibenzo-p-dioxin		2,3,4,7,8-Pentachlorodibenzofuran		2,3,7,8-Tetrachlorodibenzofuran		2,3,7,8-Tetrachlorodibenzo-p-dioxin		Aldrin		Arsenic		Bis(2-ethylhexyl) phthalate		Cadmium	
			Sample	Sample		Start	End	Depth			Elevation		70648-26-9		40321-76-4		57117-31-4		51207-31-9		1746-01-6		309-00-2		7440-38-2		117-81-7		7440-43-9	
Matrix	Location ID	Sample Name	Type	Date	Depth Class	Depth	Depth	Units	X Coordinate	Y coordinate	(NAVD88)		µg/kg		µg/kg		µg/kg		µg/kg		µg/kg		µg/kg		mg/kg		µg/kg		mg/kg	
											Sediment/Bank Soil CUL		**0.0004**		**0.0002**		**0.0003**		**0.0004**		**0.0002**		**2**		**3**		**135**		**0.51**	
											Sitewide RAL		**NA**		**0.0008**		**0.2**		**NA**		**0.0006**		--		--		--		--	
											PTW Threshold		**0.04**		**0.01**		**0.2**		**0.6**		**0.01**		--		--		--		--	
SE	WLCWTG02ANCTPD01	WLCWTG02ANCTPD01A	N	7/16/2002	Surface Sediment	0	2	cm	7628756	700622	-35		--		--		--		--		--		2.6	U	***4.1***		510	U	0.36	
SE	WLCWTG02ANCTPD01	WLCWTG02ANCTPD01B	N	7/16/2002	Surface Sediment	0	15	cm	7628756	700622	-35		--		--		--		--		--		2.2	U	***3.8***		430	U	0.32	
SE	WLCWTG02ANCTPD02	WLCWTG02ANCTPD02A	N	7/16/2002	Surface Sediment	0	2	cm	7628958	700792	-34		--		--		--		--		--		2.3	U	***3.5***		490	U	0.3	
SE	WLCWTG02ANCTPD02	WLCWTG02ANCTPD02B	N	7/16/2002	Surface Sediment	0	15	cm	7628958	700792	-34		--		--		--		--		--		2	U	***3.8***		***6,700***		0.3	
SE	WLCWTG02ANCTPD03	WLCWTG02ANCTPD03A	N	7/16/2002	Surface Sediment	0	2	cm	7629152	700954	-39		--		--		--		--		--		2.8	U	***3.9***		590	U	0.26	
SE	WLCWTG02ANCTPD03	WLCWTG02ANCTPD03B	N	7/16/2002	Surface Sediment	0	15	cm	7629152	700954	-39		--		--		--		--		--		1.8	U	***5.5***		370	U	0.32	
SE	WLCWTG02ANCTPD05	WLCWTG02ANCTPD09R	FR	7/16/2002	Surface Sediment	0	15	cm	7629438	700917	-40		--		--		--		--		--		2.3	U	***3.6***		460	U	0.28	
SE	WLCWTG02ANCTPD06	WLCWTG02ANCTPD06A	N	7/16/2002	Surface Sediment	0	2	cm	7629255	700769	-38		--		--		--		--		--		2.6	U	***3.2***		520	U	0.33	
SE	WLCWTG02ANCTPD06	WLCWTG02ANCTPD06B	N	7/16/2002	Surface Sediment	0	15	cm	7629255	700769	-38		--		--		--		--		--		2.1	UT	***3.8***	***T***	460	UT	0.365	T
SE	WLCWTG02ANCTPD07	WLCWTG02ANCTPD07A	N	7/16/2002	Surface Sediment	0	2	cm	7629084	700617	-34		--		--		--		--		--		2.8	U	***3.9***		580	U	0.3	
SE	WLCWTG02ANCTPD07	WLCWTG02ANCTPD07B	N	7/16/2002	Surface Sediment	0	15	cm	7629084	700617	-34		--		--		--		--		--		2.3	U	***3.3***		460	U	0.28	
SE	WLCWTG02ANCTPD08	WLCWTG02ANCTPD08A	N	7/16/2002	Surface Sediment	0	2	cm	7628892	700455	-33		--		--		--		--		--		3.2	U	***4.8***		670	U	0.31	
SE	WLCWTG02ANCTPD08	WLCWTG02ANCTPD08B	N	7/16/2002	Surface Sediment	0	15	cm	7628892	700455	-33		--		--		--		--		--		2.6	U	***4.3***		540	U	0.31	
SE	WLCWTI00N20	WLCWTI00N20N20	N	9/22/2000	Subsurface Sediment	15	30	cm	7628972	700109	9		--		--		--		--		--		--		--		--		--		--	
SE	WLCWTI00N40	WLCWTI00N40N40	N	9/22/2000	Subsurface Sediment	15	30	cm	7628959	700126	8		--		--		--		--		--		--		--		--		--		--	
SE	WLCWTI00S20	WLCWTI00S20S20	N	9/22/2000	Subsurface Sediment	15	30	cm	7628998	700078	8		--		--		--		--		--		--		--		--		--		--	
SE	WLCWTI00S40	WLCWTI00S40S40	N	9/22/2000	Subsurface Sediment	15	30	cm	7629011	700062	9		--		--		--		--		--		--		--		--		--		--	
SE	WLCWTI00TP11	WLCWTI00TP11TP11	N	9/21/2000	Subsurface Sediment	122	137	cm	7628955	700089	13		--		--		--		--		--		--		--		--		--		--	
SE	WLRWTF98ATXSD3	WLRWTF98ATXSD3ATXSD3	N	12/18/1998	Surface Sediment	0	12	cm	7628416	701026	-8		--		--		--		--		--		3.35	U	--		--		--		--	
SE	WLRWTF98ATXSD4	WLRWTF98ATXSD4ATXSD4	N	12/18/1998	Surface Sediment	0	12	cm	7628460	700769	5		--		--		--		--		--		6.7	U	--		--		--		--	
SE	WLRWTF98ATXSD5	WLRWTF98ATXSD5ATXSD5	N	12/18/1998	Surface Sediment	0	12	cm	7628584	700620	5		--		--		--		--		--		6.7	U	2.52		2,000	UJ	***5.05***		
SE	WLRWTF98HEVSD1	WLRWTF98HEVSD1HEVSD1	N	12/17/1998	Surface Sediment	0	12	cm	7628832	700321	1		--		--		--		--		--		--		1.25		2,000	UJ	***1.21***		
SE	WLRWTF98HEVSD2	WLRWTF98HEVSD2HEVSD2	N	12/17/1998	Surface Sediment	0	12	cm	7628783	700396	-15		--		--		--		--		--		3.35	U	1.22		2,000	UJ	***0.651***		
SE	WLRWTF98HEVSD3	WLRWTF98HEVSD3HEVSD3	N	12/17/1998	Surface Sediment	0	12	cm	7628708	700471	0		--		--		--		--		--		3.35	U	2.42		2,000	UJ	***1.07***		
SE	WLRWTF98TOSSD1	WLRWTF98TOSSD1TOSSD1	N	12/17/1998	Surface Sediment	0	12	cm	7628882	700247	6		--		--		--		--		--		--		2.6		2,000	UJ	***1.3***		
SE	WLRWTF98TOSSD2	WLRWTF98TOSSD2TOSSD2	N	12/17/1998	Surface Sediment	0	12	cm	7628955	700135	7		--		--		--		--		--		--		--		--		--		--	
SE	WLRWTF98TOSSD3	WLRWTF98TOSSD3TOSSD3	N	12/17/1998	Surface Sediment	0	12	cm	7629084	700168	-12		--		--		--		--		--		--		***6.18***		4,800	UJ	***1.56***		
SE	WLRWTF98TOSSD4	WLRWTF98TOSSD4TOSSD4	N	12/17/1998	Surface Sediment	0	12	cm	7629054	699987	13		--		--		--		--		--		--		--		--		--		--	
SE	WLRWTF98TOSSD5	WLRWTF98TOSSD5TOSSD5	N	12/18/1998	Surface Sediment	0	12	cm	7629232	699945	9		--		--		--		--		--		--		***6.53***	***J***	2,000	UJ	***5.79***		
SE	WR-WSI98SD104	WR-WSI98SD1040	N	9/23/1997	Surface Sediment	0	10	cm	7628572	701218	-1		--		--		--		--		--		--		5	U	***320***		0.3		
SE	WR-WSI98SD105	WR-WSI98SD1050	N	9/23/1997	Surface Sediment	0	10	cm	7628878	701254	-20		--		--		--		--		--		--		6	U	***270***		0.3		
SE	WR-WSI98SD107	WR-WSI98SD1070	N	9/23/1997	Surface Sediment	0	10	cm	7628533	700849	-19		--		--		--		--		--		--		6	U	***430***		0.5		
SE	WR-WSI98SD110	WR-WSI98SD1100	N	9/23/1997	Surface Sediment	0	10	cm	7629093	700983	-35		--		--		--		--		--		--		6	U	130		0.5		
SE	WR-WSI98SD113	WR-WSI98SD1130	N	9/23/1997	Surface Sediment	0	10	cm	7629298	700704	-35		--		--		--		--		--		--		6	U	***210***		0.4		
SE	WR-WSI98SD114	WR-WSI98SD1140	N	9/23/1997	Surface Sediment	0	10	cm	7629023	700135	-8		--		--		--		--		--		--		3	U	***250***		***0.7***		
SE	WR-WSI98SD115	WR-WSI98SD1150	N	9/23/1997	Surface Sediment	0	10	cm	7629434	700515	-24		--		--		--		--		--		--		5	U	120		0.4		
SE	WR-WSI98SD117	WR-WSI98SD1170000A	N	10/16/1997	Subsurface Sediment	0	90	cm	7629340	700260	-22		--		--		--		--		--		0.95	U	4	U	90	UJ	0.5		
SE	WR-WSI98SD117	WR-WSI98SD1170000CC	N	9/23/1997	Surface Sediment	0	10	cm	7629340	700260	-22		--		--		--		--		--		0.98	UT	4	UT	122	T	0.4	T	
SE	WR-WSI98SD118	WR-WSI98SD1180	N	9/23/1997	Surface Sediment	0	10	cm	7629521	700296	-16		--		--		--		--		--		--		4	U	59		0.3		
SE	WR-WSI98SD123	WR-WSI98SD1230	N	9/24/1997	Surface Sediment	0	10	cm	7630398	699583	-8		--		--		--		--		--		--		5	U	91		0.4		

Table A-1. RI/FS Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

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												LWG RA Total Chlordane (Calculated U = 1/2)		Chlordane (cis & trans)		Chlordane (technical)		Chlorobenzene		Copper		Dieldrin		Diesel Range Hydrocarbons		Lead		Lindane (gamma-Hexachlorocyclohexane)	
CAS			Sample	Sample		Start	End	Depth			Elevation			57-74-9		12789-03-6		108-90-7		7440-50-8		60-57-1		68334-30-5		7439-92-1			
Matrix	Location ID	Sample Name	Type	Date	Depth Class	Depth	Depth	Units	X Coordinate	Y coordinate	(NAVD88)	µg/kg		µg/kg		µg/kg		µg/kg		mg/kg		µg/kg		mg/kg		mg/kg		µg/kg	
											Sediment/Bank Soil CUL	**1.4**		**1.4**		**1.4**		**NA**		**359**		**0.07**		**91**		**196**		**5**	
											Sitewide RAL	--		--		--		**NA**		--		--		--		--		--	
											PTW Threshold	--		--		--		**320**		--		--		--		--		--	
SE	LW2-B019	LW2-B019	N	7/26/2004	Surface Sediment	0	15	cm	7628948	700051	9	0.094	JT	--		--		--		20.9	J	0.046	U	--		7.88		0.0687	U
SE	LW2-C377	LW2-C377-B	N	11/10/2004	Subsurface Sediment	30	153	cm	7628521	701143	-5	0.894	NJT	--		--		--		31.1		0.0672	U	***120***	***J***	16.8	J	0.1	U
SE	LW2-C377	LW2-C377-C	N	11/10/2004	Subsurface Sediment	153	273	cm	7628521	701143	-5	***2.9***	***NJT***	--		--		--		38.4		0.357	U	***1,200***	***J***	28	J	0.532	U
SE	LW2-C377	LW2-C377-D	N	11/10/2004	Subsurface Sediment	273	369	cm	7628521	701143	-5	--		--		--		--		--		--		--		--		--	
SE	LW2-C377	LW2-C377-E	N	11/10/2004	Subsurface Sediment	369	482	cm	7628521	701143	-5	0.52	UT	--		--		--		36.8		0.639	U	***4,500***	***J***	35.5	J	0.953	U
SE	LW2-C401	LW2-C401-B	N	11/10/2004	Subsurface Sediment	30	69	cm	7629031	700153	-10	***2***	***NJT***	--		--		--		27.8		***0.725***	***NJ***	***810***	***J***	29.5	J	0.45	U
SE	LW2-C401	LW2-C401-C	N	11/10/2004	Subsurface Sediment	69	188	cm	7629031	700153	-10	***2***	***NJT***	--		--		--		23.1		0.259	U	***1,900***	***J***	23.5	J	0.387	U
SE	LW2-C401	LW2-C401-D	N	11/10/2004	Subsurface Sediment	188	278	cm	7629031	700153	-10	1.3	JT	--		--		--		19.3		0.254	UJ	***1,100***	***J***	21.7	J	0.379	UJ
SE	LW2-C401	LW2-C401-E	N	11/10/2004	Subsurface Sediment	278	367	cm	7629031	700153	-10	***2.22***	***NJT***	--		--		--		28.1		0.299	U	***1,900***	***J***	24.8	J	0.446	U
SE	LW2-C401	LW2-C401-F	N	11/10/2004	Subsurface Sediment	367	504	cm	7629031	700153	-10	0.097	UJT	--		--		--		--		0.12	UJ	--		--		0.14	UJ
SE	LW2-C403	LW2-C403-B	N	11/10/2004	Subsurface Sediment	30	153	cm	7629841	700123	-6	0.0435	UT	--		--		0.098	U	16.1		0.0534	U	***100***	***J***	5.71		0.0797	U
SE	LW2-C403	LW2-C403-C	N	11/10/2004	Subsurface Sediment	153	242	cm	7629841	700123	-6	0.044	UT	--		--		0.09	U	14.7		0.0535	U	20	T	2.74		0.0797	U
SE	LW2-C403	LW2-C403-D	N	11/10/2004	Subsurface Sediment	242	361	cm	7629841	700123	-6	0.049	UT	--		--		0.11	U	30		0.0606	U	13	U	5.21		0.0903	U
SE	LW2-C403	LW2-C403-F	N	11/10/2004	Subsurface Sediment	483	533	cm	7629841	700123	-6	--		--		--		0.12	U	36.2		--		--		5.43		--	
SE	LW2-C413	LW2-C413-B1	N	10/26/2004	Subsurface Sediment	30	158	cm	7630354	699540	2	***2.1***	***JT***	--		--		--		46.1		0.355	U	--		25.7		0.529	U
SE	LW2-C413	LW2-C413-C1	N	10/26/2004	Subsurface Sediment	158	299	cm	7630354	699540	2	0.85	NJT	--		--		--		43.4		0.339	U	--		29.4		0.506	U
SE	LW2-C413-2	LW2-C413-B2	FR	10/26/2004	Subsurface Sediment	30	152	cm	7630359	699540	1	1.37	JT	--		--		--		37.5		0.333	U	--		37.2		0.497	U
SE	LW2-C413-2	LW2-C413-C2	FR	10/26/2004	Subsurface Sediment	152	274	cm	7630359	699540	1	0.281	UT	--		--		--		47.6		0.345	U	--		31.3		0.514	U
SE	LW2-C413-2	LW2-C413-D2	N	10/26/2004	Subsurface Sediment	274	349	cm	7630359	699540	1	***7.1***	***JT***	--		--		--		42.7	T	0.331	U	--		27	T	0.494	U
SE	LW2-C532	LW2-C532-A	N	10/18/2005	Surface Sediment	0	30	cm	7629257	700289	-21	***1.6***	***JT***	--		--		0.14	U	53.6		0.38	UJ	***260***	***J***	25.7		0.28	U
SE	LW2-C532	LW2-C532-B	N	10/18/2005	Subsurface Sediment	30	99	cm	7629257	700289	-21	1.1	JT	--		--		0.13	U	40.2		0.85	UJ	***290***	***J***	22.1		0.92	NJ
SE	LW2-C532	LW2-C532-C	N	10/18/2005	Subsurface Sediment	99	198	cm	7629257	700289	-21	1.1	JT	--		--		0.13	U	53.3		0.41	UJ	***740***	***J***	32.9		0.49	U
SE	LW2-C532	LW2-C532-D	N	10/18/2005	Subsurface Sediment	198	293	cm	7629257	700289	-21	--		--		--		--		--		--		***3,800***	***JT***	--		--	
SE	LW2-C532	LW2-C532-E	N	10/18/2005	Subsurface Sediment	293	338	cm	7629257	700289	-21	1.2	JT	--		--		0.12	U	53.2		0.38	UJ	***1,500***	***J***	38.4		0.38	U
SE	LW2-C532	LW2-C532-F	N	10/18/2005	Subsurface Sediment	338	372	cm	7629257	700289	-21	0.09	UJT	--		--		0.094	U	19.5		0.11	UJ	4.6	UT	7.42		0.13	U
SE	LW2-G389	LW2-G389	N	10/29/2004	Surface Sediment	0	20	cm	7628548	700673	4	0.05	UJT	--		--		--		15.8		0.0614	U	--		6.34	J	0.0915	U
SE	LW2-G394	LW2-G394	N	8/27/2004	Surface Sediment	0	25	cm	7628836	700366	-25	***18***	***JNT***	--		--		--		49.8		0.343	UJ	--		71.7	J	0.512	UJ
SE	LW2-G399	LW2-G399	N	8/27/2004	Surface Sediment	0	22	cm	7629573	700201	-1	1.14	JNT	--		--		--		31.9		0.297	UJ	--		11.4	J	0.444	UJ
SE	LW2-G401	LW2-G401	N	10/22/2004	Surface Sediment	0	27	cm	7629036	700154	-10	0.82	NJT	--		--		--		36.1		0.356	U	***240***	***J***	19.8		0.531	U
SE	LW2-G403	LW2-G403	N	10/22/2004	Surface Sediment	0	26	cm	7629824	700114	-4	0.0429	UJT	--		--		0.099	U	16.3		0.0527	U	20	J	11.2		0.0786	U
SE	LW2-G404	LW2-G404	N	8/27/2004	Surface Sediment	0	25	cm	7629091	700096	-9	1.4	JNT	--		--		0.15	U	39.8		***0.768***	***NJ***	***170***	***J***	16	J	0.119	U
SE	LW2-G407	LW2-G407	N	9/9/2004	Surface Sediment	0	29	cm	7630154	699834	-12	1.1	JNT	--		--		--		39.5	T	0.0701	U	--		14.9	JT	0.105	U
SE	LW2-G413	LW2-G413	N	10/22/2004	Surface Sediment	0	27	cm	7630351	699536	2	0.642	JT	--		--		--		28		0.0498	U	--		15.3		1.69	NJ
SE	LW2-GBT020	LW2-GBT020	N	12/6/2005	Surface Sediment	0	10	cm	7628495	701060	-13	0.999	JT	--		--		0.16	U	33.6		***0.36***		***420***	***J***	16.9		0.0153	U
SE	LW2-GBT021	LW2-GBT021	N	12/13/2005	Surface Sediment	0	10	cm	7629274	700117	-6	0.557	JT	--		--		0.14	U	37.4		***0.107***	***J***	***130***	***J***	12.2	J	0.0089	J
SE	LW2-NA3A	NA-3A-0005	N	10/22/2004	Surface Sediment	0	5	cm	7629429	700328	-21	--		--		--		--		26.3		--		--		12.4		--	
SE	LW2-NA3A	NA-3A-0508	N	10/22/2004	Subsurface Sediment	5	8	cm	7629429	700328	-21	--		--		--		--		26.5		--		--		11.9		--	
SE	LW2-NA3A	NA-3A-0812	N	10/22/2004	Subsurface Sediment	8	12	cm	7629429	700328	-21	--		--		--		--		44		--		--		24.6		--	
SE	LW2-NA3A	NA-3A-1216	N	10/22/2004	Subsurface Sediment	12	16	cm	7629429	700328	-21	--		--		--		--		41.9		--		--		29.1		--	
SE	LW2-NA3A	NA-3A-1620	N	10/22/2004	Subsurface Sediment	16	20	cm	7629429	700328	-21	--		--		--		--		35		--		--		21.4		--	
SE	LW2-NA3A	NA-3A-2024	N	10/22/2004	Subsurface Sediment	20	24	cm	7629429	700328	-21	--		--		--		--		24.9		--		--		13.4		--	
SE	LW2-NA3A	NA-3A-2428	N	10/22/2004	Subsurface Sediment	24	28	cm	7629429	700328	-21	--		--		--		--		34.7		--		--		18.6		--	
SE	LW2-NA3A	NA-3A-2832	N	10/22/2004	Subsurface Sediment	28	32	cm	7629429	700328	-21	--		--		--		--		19.8		--		--		12.6		--	
SE	LW2-NA3A	NA-3A-3238	N	10/22/2004	Subsurface Sediment	32	38	cm	7629429	700328	-21	--		--		--		--		23	T	--		--		12	T	--	
SE	LW2-NA3A	NA-3A-3844	N	10/22/2004	Subsurface Sediment	38	44	cm	7629429	700328	-21	--		--		--		--		26.4		--		--		16.7		--	
SE	LW2-NA3A	NA-3A-4854	N	10/22/2004	Subsurface Sediment	48	54	cm	7629429	700328	-21	--		--		--		--		29.3		--		--		19.5		--	
SE	LW2-NA3A	NA-3A-5864	N	10/22/2004	Subsurface Sediment	58	64	cm	7629429	700328	-21	--		--		--		--		38.7		--		--		20.8		--	
SE	LW2-NA3A	NA-3A-6874	N	10/22/2004	Subsurface Sediment	68	74	cm	7629429	700328	-21	--		--		--		--		39.6		--		--		25.5		--	
SE	LW2-NA3A	NA-3A-7884	N	10/22/2004	Subsurface Sediment	78	84	cm	7629429	700328	-21	--		--		--		--		86.3		--		--		44.4		--	
SE	LW2-NA3A	NA-3A-8894	N	10/22/2004	Subsurface Sediment	88	94	cm	7629429	700328	-21	--		--		--		--		51		--		--		22.8		--	
SE	LW2-NA3B	NA-3B-0034	N	10/22/2004	Surface Sediment	0	34	cm	7629433	700322	-20	***2.63***	***JNT***	--		--		0.11	U	--		0.0595	U	***140***	***J***	--		0.0887	U
SE	LW2-NA3B	NA-3B-3464	N	10/22/2004	Subsurface Sediment	34	64	cm	7629433	700322	-20	0.69	JNT	--		--		0.11	U	--		0.0561	U	80	JT	--		0.0836	U
SE	LW2-NA3B	NA-3B-6494	N	10/22/2004	Subsurface Sediment	64	94	cm	7629433	700322	-20	***1.92***	***NJT***	--		--		0.12	U	--		0.0608	U	***200***	***J***	--		0.0906	U
SE	LW3-C688	LW3-C688-B	N	1/15/2008	Subsurface Sediment	30	114	cm	7628760	701367	-5	1	JT	--		--		--		32.8		***0.37***		56	J	14.2		0.2	U
SE	LW3-C688	LW3-C688-C	N	1/15/2008	Subsurface Sediment	114	221	cm	7628760	701367	-5	***1.9***	***JT***	--		--		--		39.4		0.19	U	***190***	***J***	22.5		0.31	U
SE	LW3-C688	LW3-C688-D	N	1/15/2008	Subsurface Sediment	221	281	cm	7628760	701367	-5	***5.8***	***JT***	--		--		--		33.5		0.15	U	***540***	***J***	25.5		0.86	U
SE	LW3-C688	LW3-C688-F	N	1/15/2008	Subsurface Sediment	404	457	cm	7628760	701367	-5	2.3	UT	--		--		--		39.3		0.15	U	***850***	***J***	29.6		0.31	U
SE	LW3-C690	LW3-C690-B	N	1/15/2008	Subsurface Sediment	30	151	cm	7628470	701000	-11	1.1	NJT	--		--		--		25.6		0.28	U	89.5	JT	17.4		0.2	U
SE	LW3-C690	LW3-C690-C	N	1/15/2008	Subsurface Sediment	151	274	cm	7628470	701000	-11	***2.2***	***NJT***	--		--		--		38.9		***0.34***	***J***	***320***	***J***	22.3		0.19	U
SE	LW3-C690	LW3-C690-D	N	1/15/2008	Subsurface Sediment	274	346	cm	7628470	701000	-11	4.4	UT	--		--		--		39.8		0.86	U	***1,300***	***J***	43.2		1.3	U
SE	LW3-C690	LW3-C690-E	N	1/15/2008	Subsurface Sediment	346	471	cm	7628470	701000	-11	6.4	UT	--		--		--		41.6		1.7	U	***3,800***	***J***	43.6		0.62	U
SE	LW3-C695	LW3-C695-B	N	12/20/2007	Subsurface Sediment	30	95	cm	7629190	700056	-6	0.82	UT	--		--		--		64.3	J	0.33	U	***720***	***J***	29.5	J	0.24	U
SE	LW3-C695	LW3-C695-D	N	12/20/2007	Subsurface Sediment	221	343	cm	7629190	700056	-6	***5.9***	***JT***	--		--		--		40.8	J	***6.9***	***NJ***	***2,900***	***J***	37.2	J	5.6	U
SE	LW3-C695	LW3-C695-E	N	12/20/2007	Subsurface Sediment	343	384	cm	7629190	700056	-6	***5***	***T***	--		--		--		49.6	J	2.7	U	***2,700***	***J***	40.3	J	***7.9***	***J***
SE	LW3-C698	LW3-C698-B	N	12/20/2007	Subsurface Sediment	30	152	cm	7630300	699718	-16	***1.7***	***JT***	--		--		--		44.8	J	0.52	U	***170***	***J***	20.8	J	0.067	U
SE	LW3-G692-1	LW3-G692-1	N	12/4/2007	Surface Sediment	0	30	cm	7628730	701055	-20	1.3	JNT	--		--		--		38.7		***0.49***		***130***	***J***	15.3		0.28	U
SE	LW3-G692-2	LW3-G692-2	FR	12/4/2007	Surface Sediment	0	28	cm	7628730	701048	-20	0.52	UT	--		--		--		37.9	T	0.43	UT	***115***	***JT***	15.1	T	0.2	UT
SE	LW3-G694	LW3-G694	N	11/16/2007	Surface Sediment	0	20	cm	7629340	700318	-23	***1.5***	***JT***	--		--		--		31.5		0.03	U	***186***	***JT***	16		0.32	U
SE	LW3-GSP07W	LW3-GSP07W	N	10/15/2007	Surface Sediment	0	19	cm	7628812	700370	-14	0.087	UT	--		--		--		20.4		0.03	U	--		5.84		0.062	U

Table A-1. RI/FS Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

													LWG RA Total Chlordane (Calculated U = 1/2)		Chlordane (cis & trans)		Chlordane (technical)		Chlorobenzene		Copper		Dieldrin		Diesel Range Hydrocarbons		Lead		Lindane (gamma-Hexachlorocyclohexane)	
CAS			Sample	Sample		Start	End	Depth			Elevation				57-74-9		12789-03-6		108-90-7		7440-50-8		60-57-1		68334-30-5		7439-92-1			
Matrix	Location ID	Sample Name	Type	Date	Depth Class	Depth	Depth	Units	X Coordinate	Y coordinate	(NAVD88)		µg/kg		µg/kg		µg/kg		µg/kg		mg/kg		µg/kg		mg/kg		mg/kg		µg/kg	
											Sediment/Bank Soil CUL		**1.4**		**1.4**		**1.4**		**NA**		**359**		**0.07**		**91**		**196**		**5**	
											Sitewide RAL		--		--		--		**NA**		--		--		--		--		--	
											PTW Threshold		--		--		--		**320**		--		--		--		--		--	
SE	LWG0108R002SDS015C00	LWG0108R002SDS015C00	N	10/17/2002	Surface Sediment	0	15	cm	7630259	699666	1		0.39	UT	--		--		--		23		0.39	U	--		15		0.19	U
SE	TOSC099DMMU1	TOSC099DMMU1SDO1	N	1/21/1999	Subsurface Sediment	0	304	cm	7629016	700391	-24		2	UT	--		--		--		46		***10***	***J***	--		29		2	U
SE	TOSC099DMMU1	TOSC099DMMU1SDO3	FR	1/21/1999	Subsurface Sediment	0	304	cm	7629016	700391	-24		2	UJT	--		--		--		48		***8***	***J***	--		28		2	UJ
SE	TOSC099DMMU2	TOSC099DMMU2SDO4	N	1/21/1999	Subsurface Sediment	0	164	cm	7629278	700566	-29		2	UT	--		--		--		44		***3***	***J***	--		21		2	U
SE	W-09-A-PG	LWG2-PG-W9A	N	11/30/2005	Surface Sediment	0	23	cm	7628899	700250	3		--		--		--		0.11	U	30.8		--		4.9	U	5.96		--	
SE	W-09-C-PG	LWG2-PG-W9C	N	11/30/2005	Surface Sediment	0	30	cm	7628988	700306	-20		--		--		--		0.17	U	43.2		--		***180***	***J***	18.6		--	
SE	WLCASF97S001	WLCASF97S001W4159	N	6/12/1997	Surface Sediment	0	10	cm	7630379	699598	-9		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S002	WLCASF97S002W4158	N	6/12/1997	Surface Sediment	0	10	cm	7630047	700105	-32		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S003	WLCASF97S003W4157	N	6/12/1997	Surface Sediment	0	10	cm	7629493	700297	-17		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S004	WLCASF97S004W4156	N	6/12/1997	Surface Sediment	0	10	cm	7629322	700277	-23		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S005	WLCASF97S005W4155	N	6/12/1997	Surface Sediment	0	10	cm	7629019	700128	-5		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S006	WLCASF97S006W4154	N	6/12/1997	Surface Sediment	0	10	cm	7628791	700365	-2		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S008	WLCASF97S008W4152	N	6/12/1997	Surface Sediment	0	10	cm	7629266	700704	-35		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S009	WLCASF97S009W4172	N	6/11/1997	Surface Sediment	0	10	cm	7628648	700612	-32		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S010	WLCASF97S010W4171	N	6/11/1997	Surface Sediment	0	10	cm	7629081	700977	-34		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S011	WLCASF97S011W4170	N	6/11/1997	Surface Sediment	0	10	cm	7628514	700853	-19		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S012	WLCASF97S012W4168	N	6/11/1997	Surface Sediment	0	10	cm	7628858	701245	-20		--		--		--		--		--		--		--		--		--	
SE	WLCASF97S012	WLCASF97S012W4169	FR	6/11/1997	Surface Sediment	0	10	cm	7628858	701245	-20		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08ANCTPD03	WLCCWI08ANCTPD03	N	5/29/2009	Surface Sediment	0	2	cm	7629152	700952	-39		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08ANCTPD06	WLCCWI08ANCTPD06	N	5/29/2009	Surface Sediment	0	2	cm	7629255	700767	-38		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU1A	WLCCWI08DMMU1A	N	5/29/2009	Surface Sediment	0	2	cm	7628747	700605	-35		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU1A	WLCCWI08DMMU1A4041	N	9/2/2008	Subsurface Sediment	91	121	cm	7628747	700605	-35		2.01	UJT	--		45.8	UJ	6.2	UJ	43.6	J	2.01	UJ	79.8	J	29.7		2.01	UJ
SE	WLCCWI08DMMU1A	WLCCWI08DMMU1A4142	N	9/2/2008	Subsurface Sediment	121	152	cm	7628747	700605	-35		2.45	UJT	--		54.9	UJ	--		46.7	J	2.45	UJ	52.8	J	15.9	J	2.45	UJ
SE	WLCCWI08DMMU1ABC	WLCCWI08DMMU1ABC	N	9/4/2008	Subsurface Sediment	91	182	cm	7628839	700680	-33		2.27	UJT	--		51.4	UJT	6.84	UJT	40.4	JT	2.26	UJT	***98.3***	***JT***	13.7	T	2.26	UJT
SE	WLCCWI08DMMU1B	WLCCWI08DMMU1B	N	5/29/2009	Surface Sediment	0	2	cm	7628834	700677	-33		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU1B	WLCCWI08DMMU1B4041	N	9/3/2008	Subsurface Sediment	121	152	cm	7628834	700677	-33		1.99	UJT	--		45.2	UJ	6.01	UJ	40.7	J	1.99	UJ	65.4	J	14.3		1.99	UJ
SE	WLCCWI08DMMU1B	WLCCWI08DMMU1B4142	N	9/3/2008	Subsurface Sediment	152	182	cm	7628834	700677	-33		2.34	UJT	--		52.3	UJ	--		41.6	J	2.34	UJ	22.2	UJ	13.8	J	2.34	UJ
SE	WLCCWI08DMMU1C	WLCCWI08DMMU1C	N	5/29/2009	Surface Sediment	0	5	cm	7628936	700759	-33		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU1C	WLCCWI08DMMU1C4041	N	9/4/2008	Subsurface Sediment	91	121	cm	7628936	700759	-33		1.98	UJT	--		45	UJ	6.11	UJ	41.9	J	1.98	UJ	56.3	J	14.9		1.98	UJ
SE	WLCCWI08DMMU1C	WLCCWI08DMMU1C4142	N	9/4/2008	Subsurface Sediment	121	152	cm	7628936	700759	-33		2.33	UJT	--		52.1	UJ	167	UJ	41	J	2.33	UJ	22.1	UJ	13.4	J	2.33	UJ
SE	WLCCWI08DMMU2A	WLCCWI08DMMU2A	N	5/29/2009	Surface Sediment	0	2	cm	7628879	700461	-33		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU2A	WLCCWI08DMMU2A4041	N	9/5/2008	Subsurface Sediment	152	182	cm	7628879	700461	-33		1.94	UT	--		44.2	U	6.03	U	40.1	J	1.94	U	***151***		15.3		1.94	U
SE	WLCCWI08DMMU2A	WLCCWI08DMMU2A4142	N	9/5/2008	Subsurface Sediment	182	213	cm	7628879	700461	-33		2.5	UT	--		56	U	--		50		2.5	U	67.6		18.2		2.5	U
SE	WLCCWI08DMMU2B	WLCCWI08DMMU2B	N	5/29/2009	Surface Sediment	0	2	cm	7628979	700542	-33		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU2B	WLCCWI08DMMU2B4041	N	9/5/2008	Subsurface Sediment	91	121	cm	7628979	700542	-33		1.92	UT	--		43.7	U	6	U	42.6	J	1.92	U	***132***		15.1		1.92	U
SE	WLCCWI08DMMU2B	WLCCWI08DMMU2B4142	N	9/5/2008	Subsurface Sediment	121	152	cm	7628979	700542	-33		2.39	UJT	--		53.6	UJ	--		57.6	J	2.39	UJ	66.8	J	24.6	J	2.39	UJ
SE	WLCCWI08DMMU2C	WLCCWI08DMMU2C	N	5/29/2009	Surface Sediment	0	2	cm	7629098	700580	-33		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU2C	WLCCWI08DMMU2C4041	N	9/5/2008	Subsurface Sediment	60	91	cm	7629098	700580	-33		2.28	UT	--		51.9	U	7.1	U	45	J	2.28	U	***103***		14.3		2.28	U
SE	WLCCWI08DMMU2C	WLCCWI08DMMU2C4142	N	9/5/2008	Subsurface Sediment	91	121	cm	7629098	700580	-33		2.34	UJT	--		52.4	UJ	--		44.2	J	2.34	UJ	71.3	J	15.8	J	2.34	UJ
SE	WLCCWI08DMMU3A	WLCCWI08DMMU3A	N	5/29/2009	Surface Sediment	0	2	cm	7628650	700651	-24		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU3A	WLCCWI08DMMU3A13	N	9/8/2008	Subsurface Sediment	30	91	cm	7628650	700651	-24		2.53	UJT	--		56.7	UJ	--		48	J	2.53	UJ	***110***	***J***	19.9	J	2.53	UJ
SE	WLCCWI08DMMU3B	WLCCWI08DMMU3B	N	5/29/2009	Surface Sediment	0	2	cm	7628779	700408	-18		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU3B	WLCCWI08DMMU3B13	N	9/8/2008	Subsurface Sediment	30	91	cm	7628779	700408	-18		4.11	UJT	--		92.1	UJ	119	U	21.8		4.11	UJ	***1,570***		23.2		4.11	UJ
SE	WLCCWI08DMMU3D	WLCCWI08DMMU3D	N	5/29/2009	Surface Sediment	0	2	cm	7628873	700325	-13		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU3D	WLCCWI08DMMU3D13	N	9/8/2008	Subsurface Sediment	30	91	cm	7628873	700325	-13		1.57	UJT	--		35.2	UJ	--		16.3	J	1.57	UJ	***4,440***	***J***	30.3	J	1.57	UJ
SE	WLCCWI08DMMU3E	WLCCWI08DMMU3E	N	5/29/2009	Surface Sediment	0	2	cm	7628981	700402	-24		--		--		--		--		--		--		--		--		--	
SE	WLCCWI08DMMU3E	WLCCWI08DMMU3E13	N	9/5/2008	Subsurface Sediment	30	91	cm	7628981	700402	-24		2.35	UT	--		52.6	U	169	U	44.2		2.35	U	67.7		14.8		2.35	U
SE	WLCDRD05PG074	WLCDRD05PG07474	N	5/25/2005	Surface Sediment	0	30	cm	7628952	701290	-24		1.1	UT	12	U	--		0.18	U	36.5	J	0.12	U	***110***	***J***	15.3	J	0.56	U
SE	WLCOFJ022201	WLCOFJ02220122010	N	10/21/2002	Surface Sediment	0	10	cm	7629006	700111	-2		2.5	UJT	3.48	UJ	--		--		14.4	J	0.812	UJ	***501***		17.6	J	0.946	UJ
SE	WLCOFJ022202	WLCOFJ02220222020	N	10/21/2002	Surface Sediment	0	15	cm	7629048	700077	-3		2.84	UJT	4	UJ	--		--		61.9	J	0.932	UJ	***242***		18	J	1.09	UJ
SE	WLCOFJ022203	WLCOFJ02220322030	N	10/21/2002	Surface Sediment	0	15	cm	7628994	700157	-4		2.88	UJT	4.06	UJ	--		--		20	J	0.946	UJ	***320***		15.9	J	1.1	UJ
SE	WLCOFJ0222031	WLCOFJ022203122031	FR	10/21/2002	Surface Sediment	0	15	cm	7628991	700159	-4		2.87	UJT	4.04	UJ	--		--		21.5	J	0.942	UJ	***488***		14.6	J	1.1	UJ
SE	WLCOFJ022204	WLCOFJ02220422040	N	10/21/2002	Surface Sediment	0	12	cm	7629090	700117	-16		4.01	UJT	5.64	UJ	--		--		39.1	J	1.31	UJ	***238***		16	J	1.53	UJ
SE	WLCOFJ022205	WLCOFJ02220522050	N	10/21/2002	Surface Sediment	0	12	cm	7629070	700151	-18		4.14	UJT	5.83	UJ	--		--		39.8	J	1.36	UJ	***124***		15.3	J	1.58	UJ
SE	WLCOFJ022206	WLCOFJ02220622060	N	10/21/2002	Surface Sediment	0	10	cm	7629040	700177	-19		3.76	UJT	5.29	UJ	--		--		33.3	J	1.23	UJ	***246***		15.1	J	1.44	UJ
SE	WLCPWL09APS-1	WLCPWL09APS-1B	N	12/29/2009	Subsurface Sediment	60	121	cm	7628973	700319	-21		1	JT	--		--		--		49.1		0.97	UJ	***350***		21.2		0.97	UJ
SE	WLCPWL09APS-1	WLCPWL09APS-1T	N	12/29/2009	Subsurface Sediment	0	60	cm	7628973	700319	-21		0.87	JT	--		--		--		41.3		1	UJ	***140***		15.4		1	UJ
SE	WLCPWL09APS-2	WLCPWL09APS-2B	N	12/29/2009	Subsurface Sediment	60	121	cm	7629116	700296	-15		0.67	JT	--		--		--		43.5		0.9	UJ	***140***		18.3		0.9	UJ
SE	WLCPWL09APS-2	WLCPWL09APS-2T	N	12/29/2009	Subsurface Sediment	0	60	cm	7629116	700296	-15		0.76	JT	--		--		--		41.8		1.1	UJ	***120***		14.9		1.1	UJ
SE	WLCPWL09APS-3	WLCPWL09APS-3B	N	12/29/2009	Subsurface Sediment	60	121	cm	7629238	700264	-20		***1.8***	***JT***	--		--		--		38.3		1.7	UJ	***240***		22.1		0.37	J
SE	WLCPWL09APS-3	WLCPWL09APS-3T	N	12/29/2009	Subsurface Sediment	0	60	cm	7629238	700264	-20		1.4	JT	--		--		--		38.5		0.9	UJ	***240***		21.6		0.9	UJ
SE	WLCPWL09DRB-1	WLCPWL09DRB-1Z	N	12/29/2009	Subsurface Sediment	320	350	cm	7629031	700383	-22		***1.8***	***JT***	--		--		--		51		0.86	UJ	***980***		36.7		0.86	UJ
SE	WLCPWL09DRB-2	WLCPWL09DRB-2Z	N	12/29/2009	Subsurface Sediment	307	338	cm	7629105	700447	-25		***1.6***	***JT***	--		--		--		50.4		0.86	UJ	***490***		32.8		0.86	UJ
SE	WLCPWL09DRB-3	WLCPWL09DRB-3Z	N	12/29/2009	Subsurface Sediment	280	310	cm	7629193	700495	-25		***1.9***	***JT***	--		--		--		49.9		0.89	UJ	***720***		33.4		0.32	J
SE	WLCPWL09DRB-D	WLCPWL09DRB-DP	N	12/29/2009	Subsurface Sediment	0	302	cm	7629110	700442	-24		1.1	JT	--		--		--		50.1		0.94	UJ	***400***		23.2		0.94	UJ
SE	WLCPWL09URB-1	WLCPWL09URB-1Z	N	12/29/2009	Subsurface Sediment	295	326	cm	7629179	700364	-17		***2***	***JT***	--		--		--		50.4		0.9	UJ	***470***		29.6		0.51	J
SE	WLCPWL09URB-2	WLCPWL09URB-2Z	N	12/29/2009	Subsurface Sediment	118	149	cm	7629295	700478	-23		***4.2***	***JT***	--		--		--		58.9		0.88	UJ	***2,200***		41.9		0.88	UJ
SE	WLCPWL09URB-3	WLCPWL09URB-3Z	N	12/29/2009	Subsurface Sediment	167	198	cm	7629268	700417	-21		0.92	JT	--		--		--		49.2		0.88	UJ	***500***		28.5		0.88	UJ
SE	WLCPWL09URB-D	WLCPWL09URB-DP	N	12/29/2009	Subsurface Sediment	0	194	cm	7629248	700420	-20		1.3	JT	--		--		--		46.1	T	0.91	UJT	***615***	***T***	22.1	T	0.21	JT

Table A-1. RI/FS Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

												LWG RA Total Chlordane (Calculated U = 1/2)		Chlordane (cis & trans)		Chlordane (technical)		Chlorobenzene		Copper		Dieldrin		Diesel Range Hydrocarbons		Lead		Lindane (gamma-Hexachlorocyclohexane)		
CAS														57-74-9		12789-03-6		108-90-7		7440-50-8		60-57-1		68334-30-5		7439-92-1				
Matrix	Location ID	Sample Name	Sample Type	Sample Date	Depth Class	Start Depth	End Depth	Depth Units	X Coordinate	Y coordinate	Elevation (NAVD88)	µg/kg		µg/kg		µg/kg		µg/kg		mg/kg		µg/kg		mg/kg		mg/kg		µg/kg		
											Sediment/Bank Soil CUL	**1.4**		**1.4**		**1.4**		**NA**		**359**		**0.07**		**91**		**196**		**5**		
											Sitewide RAL	--		--		--		**NA**		--		--		--		--		--		
											PTW Threshold	--		--		--		**320**		--		--		--		--		--		
SE	WLCWTG02ANCTPD01	WLCWTG02ANCTPD01A	N	7/16/2002	Surface Sediment	0	2	cm	7628756	700622	-35	2.6	UT	--		--		--		40.9		2.6	U	--		21.6		2.6	U	
SE	WLCWTG02ANCTPD01	WLCWTG02ANCTPD01B	N	7/16/2002	Surface Sediment	0	15	cm	7628756	700622	-35	2.2	UT	--		--		--		36.4		2.2	U	--		18.5		2.2	U	
SE	WLCWTG02ANCTPD02	WLCWTG02ANCTPD02A	N	7/16/2002	Surface Sediment	0	2	cm	7628958	700792	-34	2.3	UT	--		--		--		33.8		2.3	U	--		15.7		2.3	U	
SE	WLCWTG02ANCTPD02	WLCWTG02ANCTPD02B	N	7/16/2002	Surface Sediment	0	15	cm	7628958	700792	-34	2	UT	--		--		--		34.7		2	U	--		15.1		2	U	
SE	WLCWTG02ANCTPD03	WLCWTG02ANCTPD03A	N	7/16/2002	Surface Sediment	0	2	cm	7629152	700954	-39	2.8	UT	--		--		--		33.7		2.8	U	--		15.5		2.8	U	
SE	WLCWTG02ANCTPD03	WLCWTG02ANCTPD03B	N	7/16/2002	Surface Sediment	0	15	cm	7629152	700954	-39	1.8	UT	--		--		--		30.6		1.8	U	--		22.5		1.8	U	
SE	WLCWTG02ANCTPD05	WLCWTG02ANCTPD09R	FR	7/16/2002	Surface Sediment	0	15	cm	7629438	700917	-40	2.3	UT	--		--		--		33.3		2.3	U	--		13		2.3	U	
SE	WLCWTG02ANCTPD06	WLCWTG02ANCTPD06A	N	7/16/2002	Surface Sediment	0	2	cm	7629255	700769	-38	2.6	UT	--		--		--		36.2		2.6	U	--		17.9		2.6	U	
SE	WLCWTG02ANCTPD06	WLCWTG02ANCTPD06B	N	7/16/2002	Surface Sediment	0	15	cm	7629255	700769	-38	2.1	UT	--		--		--		37.9	T	2.1	UT	--		20.4	T	2.1	UT	
SE	WLCWTG02ANCTPD07	WLCWTG02ANCTPD07A	N	7/16/2002	Surface Sediment	0	2	cm	7629084	700617	-34	2.8	UT	--		--		--		35.7		2.8	U	--		16.5		2.8	U	
SE	WLCWTG02ANCTPD07	WLCWTG02ANCTPD07B	N	7/16/2002	Surface Sediment	0	15	cm	7629084	700617	-34	2.3	UT	--		--		--		36		2.3	U	--		14.6		2.3	U	
SE	WLCWTG02ANCTPD08	WLCWTG02ANCTPD08A	N	7/16/2002	Surface Sediment	0	2	cm	7628892	700455	-33	3.2	UT	--		--		--		38.5		3.2	U	--		17		3.2	U	
SE	WLCWTG02ANCTPD08	WLCWTG02ANCTPD08B	N	7/16/2002	Surface Sediment	0	15	cm	7628892	700455	-33	2.6	UT	--		--		--		37.1		2.6	U	--		17.3		2.6	U	
SE	WLCWTI00N20	WLCWTI00N20N20	N	9/22/2000	Subsurface Sediment	15	30	cm	7628972	700109	9	--		--		--		--		--		--		***703***		--		--		
SE	WLCWTI00N40	WLCWTI00N40N40	N	9/22/2000	Subsurface Sediment	15	30	cm	7628959	700126	8	--		--		--		--		--		--		50	U	--		--		
SE	WLCWTI00S20	WLCWTI00S20S20	N	9/22/2000	Subsurface Sediment	15	30	cm	7628998	700078	8	--		--		--		--		--		--		***153***		--		--		
SE	WLCWTI00S40	WLCWTI00S40S40	N	9/22/2000	Subsurface Sediment	15	30	cm	7629011	700062	9	--		--		--		--		--		--		50	U	--		--		
SE	WLCWTI00TP11	WLCWTI00TP11TP11	N	9/21/2000	Subsurface Sediment	122	137	cm	7628955	700089	13	--		--		--		--		--		--		50	U	--		--		
SE	WLRWTF98ATXSD3	WLRWTF98ATXSD3ATXSD3	N	12/18/1998	Surface Sediment	0	12	cm	7628416	701026	-8	3.35	UT	75	U	--		--		--		3.35	U	--		--		3.35	U	
SE	WLRWTF98ATXSD4	WLRWTF98ATXSD4ATXSD4	N	12/18/1998	Surface Sediment	0	12	cm	7628460	700769	5	6.7	UT	150	U	--		--		--		3.35	U	--		--		3.35	U	
SE	WLRWTF98ATXSD5	WLRWTF98ATXSD5ATXSD5	N	12/18/1998	Surface Sediment	0	12	cm	7628584	700620	5	6.7	UT	150	U	--		--		16.8		3.35	U	--		40.7		3.35	U	
SE	WLRWTF98HEVSD1	WLRWTF98HEVSD1HEVSD1	N	12/17/1998	Surface Sediment	0	12	cm	7628832	700321	1	--		--		--		--		35.8		--		--		15.5	J	--		
SE	WLRWTF98HEVSD2	WLRWTF98HEVSD2HEVSD2	N	12/17/1998	Surface Sediment	0	12	cm	7628783	700396	-15	3.35	UT	75	U	--		--		11.3		3.35	U	--		10	J	3.35	U	
SE	WLRWTF98HEVSD3	WLRWTF98HEVSD3HEVSD3	N	12/17/1998	Surface Sediment	0	12	cm	7628708	700471	0	3.35	UT	75	U	--		--		23.8		3.35	U	--		12.9	J	3.35	U	
SE	WLRWTF98TOSSD1	WLRWTF98TOSSD1TOSSD1	N	12/17/1998	Surface Sediment	0	12	cm	7628882	700247	6	--		--		--		--		35.1		--		--		13.7	J	--		
SE	WLRWTF98TOSSD2	WLRWTF98TOSSD2TOSSD2	N	12/17/1998	Surface Sediment	0	12	cm	7628955	700135	7	--		--		--		--		--		--		--		--		--		
SE	WLRWTF98TOSSD3	WLRWTF98TOSSD3TOSSD3	N	12/17/1998	Surface Sediment	0	12	cm	7629084	700168	-12	--		--		--		--		47.9		--		--		42	J	--		
SE	WLRWTF98TOSSD4	WLRWTF98TOSSD4TOSSD4	N	12/17/1998	Surface Sediment	0	12	cm	7629054	699987	13	--		--		--		--		--		--		--		--		--		
SE	WLRWTF98TOSSD5	WLRWTF98TOSSD5TOSSD5	N	12/18/1998	Surface Sediment	0	12	cm	7629232	699945	9	--		--		--		--		19.3		--		--		35.5		--		
SE	WR-WSI98SD104	WR-WSI98SD1040	N	9/23/1997	Surface Sediment	0	10	cm	7628572	701218	-1	--		--		--		--		30.8		--		--		10		--		
SE	WR-WSI98SD105	WR-WSI98SD1050	N	9/23/1997	Surface Sediment	0	10	cm	7628878	701254	-20	--		--		--		--		43.3		--		--		11		--		
SE	WR-WSI98SD107	WR-WSI98SD1070	N	9/23/1997	Surface Sediment	0	10	cm	7628533	700849	-19	--		--		--		--		44.8		--		--		16		--		
SE	WR-WSI98SD110	WR-WSI98SD1100	N	9/23/1997	Surface Sediment	0	10	cm	7629093	700983	-35	--		--		--		--		48.2		--		--		14		--		
SE	WR-WSI98SD113	WR-WSI98SD1130	N	9/23/1997	Surface Sediment	0	10	cm	7629298	700704	-35	--		--		--		--		45.9		--		--		13		--		
SE	WR-WSI98SD114	WR-WSI98SD1140	N	9/23/1997	Surface Sediment	0	10	cm	7629023	700135	-8	--		--		--		--		28.7		--		--		47		--		
SE	WR-WSI98SD115	WR-WSI98SD1150	N	9/23/1997	Surface Sediment	0	10	cm	7629434	700515	-24	--		--		--		--		45.1		--		--		14		--		
SE	WR-WSI98SD117	WR-WSI98SD1170000A	N	10/16/1997	Subsurface Sediment	0	90	cm	7629340	700260	-22	0.95	UJT	--		--		--		42.7		1.9	U	--		22		0.95	U	
SE	WR-WSI98SD117	WR-WSI98SD1170000CC	N	9/23/1997	Surface Sediment	0	10	cm	7629340	700260	-22	0.98	UT	--		--		--		29.7	T	2	UT	--		15.5	T	0.98	UT	
SE	WR-WSI98SD118	WR-WSI98SD1180	N	9/23/1997	Surface Sediment	0	10	cm	7629521	700296	-16	--		--		--		--		27.8		--		--		12		--		
SE	WR-WSI98SD123	WR-WSI98SD1230	N	9/24/1997	Surface Sediment	0	10	cm	7630398	699583	-8	--		--		--		--		41.5		--		--		14		--		

Table A-1. RI/FS Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

CAS												LWG RA Sum DDD (Calculated U = 1/2)		LWG RA Sum DDE (Calculated U = 1/2)		LWG RA Sum DDT (Calculated U = 1/2)		LWG RA Total cPAH/BaPE		LWG RA Total DDx (Calculated U = 1/2)		LWG RA Total PAH (Calculated U = 1/2)		LWG RA Total PCB Aroclors (Calculated U = 1/2)	
Matrix	Location ID	Sample Name	Sample Type	Sample Date	Depth Class	Start Depth	End Depth	Depth Units	X Coordinate	Y coordinate	Elevation (NAVD88)	LRASum_DDD_N		LRASum_DDE_N		LRASum_DDT_N		LRAtcPAHTEF7_N		LRAtDDT_N		LRAtPAH_17_N		LRAtPCB_N	
												µg/kg		µg/kg		µg/kg		µg/kg		µg/kg		µg/kg		µg/kg	
											Sediment/Bank Soil CUL	**114**		**50**		**246**		**774**		**6.1**		**23,000**		**9**	
											Sitewide RAL	--		--		--		**NA**		**160**		**30,000**		**75**	
											PTW Threshold	--		--		--		**774,000**		**7,050**		**NA**		**200**	
SE	LW2-B019	LW2-B019	N	7/26/2004	Surface Sediment	0	15	cm	7628948	700051	9	0.614	NJT	0.0406	UT	2.9	JNT	3.3	JT	3.54	JNT	24	JT	2.1	UT
SE	LW2-C377	LW2-C377-B	N	11/10/2004	Subsurface Sediment	30	153	cm	7628521	701143	-5	29	JNT	4.79	NJT	28	JNT	39	T	***62***	***JNT***	330	T	***39.6***	***JT***
SE	LW2-C377	LW2-C377-C	N	11/10/2004	Subsurface Sediment	153	273	cm	7628521	701143	-5	69	JNT	10.1	NJT	20	JT	38	T	***99***	***JNT***	430	T	***280***	***T***
SE	LW2-C377	LW2-C377-D	N	11/10/2004	Subsurface Sediment	273	369	cm	7628521	701143	-5	--		--		--		110	T	--		2,100	T	--	
SE	LW2-C377	LW2-C377-E	N	11/10/2004	Subsurface Sediment	369	482	cm	7628521	701143	-5	***241***	***JNT***	14.7	NJT	3.2	JT	380	T	***260***	***JNT***	6,200	T	--	
SE	LW2-C401	LW2-C401-B	N	11/10/2004	Subsurface Sediment	30	69	cm	7629031	700153	-10	9.36	NJT	3.9	NJT	9.79	JT	360	T	***23***	***JNT***	2,600	T	***51.6***	***JT***
SE	LW2-C401	LW2-C401-C	N	11/10/2004	Subsurface Sediment	69	188	cm	7629031	700153	-10	7.36	NJT	1.67	NJT	0.229	AUT	170	T	***9.14***	***ANJT***	2,200	T	***36***	***JT***
SE	LW2-C401	LW2-C401-D	N	11/10/2004	Subsurface Sediment	188	278	cm	7629031	700153	-10	13	T	3.14	JT	2.5	JNT	140	T	***19***	***JNT***	2,100	T	***300***	***JT***
SE	LW2-C401	LW2-C401-E	N	11/10/2004	Subsurface Sediment	278	367	cm	7629031	700153	-10	17.6	JNT	13.2	NJT	0.977	JT	72	T	***31.8***	***JNT***	1,900	T	--	
SE	LW2-C401	LW2-C401-F	N	11/10/2004	Subsurface Sediment	367	504	cm	7629031	700153	-10	0.2	UJT	0.081	UJT	0.21	UJT	12	T	0.21	UJT	130	JT	1.6	UT
SE	LW2-C403	LW2-C403-B	N	11/10/2004	Subsurface Sediment	30	153	cm	7629841	700123	-6	0.42	JNT	0.236	T	0.0581	UJT	40	T	0.7	JNT	400	T	***11***	***JT***
SE	LW2-C403	LW2-C403-C	N	11/10/2004	Subsurface Sediment	153	242	cm	7629841	700123	-6	0.0503	UT	0.356	NJT	0.0582	UJT	3.5	JT	0.45	NJT	21	JT	2.42	UT
SE	LW2-C403	LW2-C403-D	N	11/10/2004	Subsurface Sediment	242	361	cm	7629841	700123	-6	0.057	UT	0.0535	UT	0.0659	UJT	0.45	JT	0.066	UJT	7.5	JT	7.9	T
SE	LW2-C403	LW2-C403-F	N	11/10/2004	Subsurface Sediment	483	533	cm	7629841	700123	-6	--		--		--		--		--		--		--	
SE	LW2-C413	LW2-C413-B1	N	10/26/2004	Subsurface Sediment	30	158	cm	7630354	699540	2	13.9	NJT	10.8	NJT	0.966	JT	36	T	***25.7***	***JNT***	390	T	***132***	***JT***
SE	LW2-C413	LW2-C413-C1	N	10/26/2004	Subsurface Sediment	158	299	cm	7630354	699540	2	21.8	NJT	9.89	JNT	1.07	JT	37	T	***32.8***	***JNT***	460	T	***96***	***JT***
SE	LW2-C413-2	LW2-C413-B2	FR	10/26/2004	Subsurface Sediment	30	152	cm	7630359	699540	1	12.8	NJT	7.73	NJT	1.86	JT	62	T	***22.4***	***JNT***	650	T	***310***	***T***
SE	LW2-C413-2	LW2-C413-C2	FR	10/26/2004	Subsurface Sediment	152	274	cm	7630359	699540	1	13.5	NJT	12	NJT	1.1	NJT	25	T	***26***	***NJT***	290	T	***410***	***JT***
SE	LW2-C413-2	LW2-C413-D2	N	10/26/2004	Subsurface Sediment	274	349	cm	7630359	699540	1	110	NJT	12.2	NJT	140	JT	62	T	***270***	***JNT***	940	T	***77***	***JT***
SE	LW2-C532	LW2-C532-A	N	10/18/2005	Surface Sediment	0	30	cm	7629257	700289	-21	4.6	JNT	8.1	JT	1.7	UJT	64	T	***14***	***JNT***	800	T	***150***	***T***
SE	LW2-C532	LW2-C532-B	N	10/18/2005	Subsurface Sediment	30	99	cm	7629257	700289	-21	6.9	JT	5.8	JT	4.1	JT	50	T	***17***	***JT***	630	T	***180***	***JT***
SE	LW2-C532	LW2-C532-C	N	10/18/2005	Subsurface Sediment	99	198	cm	7629257	700289	-21	8.3	JNT	5.9	JT	3.1	JT	56	T	***17***	***JNT***	670	T	***120***	***JT***
SE	LW2-C532	LW2-C532-D	N	10/18/2005	Subsurface Sediment	198	293	cm	7629257	700289	-21	--		--		--		--		--		--		--	
SE	LW2-C532	LW2-C532-E	N	10/18/2005	Subsurface Sediment	293	338	cm	7629257	700289	-21	21	JT	13	NJT	1.5	JT	210	T	***35***	***JNT***	2,500	T	***49***	***JT***
SE	LW2-C532	LW2-C532-F	N	10/18/2005	Subsurface Sediment	338	372	cm	7629257	700289	-21	0.18	JT	0.13	JT	0.19	UJT	0.43	UT	0.44	JT	6.3	JT	1.5	UT
SE	LW2-G389	LW2-G389	N	10/29/2004	Surface Sediment	0	20	cm	7628548	700673	4	0.252	NJT	0.514	JNT	0.528	JT	0.47	JT	1.29	JNT	6.3	JT	2.7	UT
SE	LW2-G394	LW2-G394	N	8/27/2004	Surface Sediment	0	25	cm	7628836	700366	-25	***385***	***JNT***	***68.4***	***JT***	5.27	JT	190	T	***459***	***JNT***	5,100	T	***790***	***JT***
SE	LW2-G399	LW2-G399	N	8/27/2004	Surface Sediment	0	22	cm	7629573	700201	-1	6.07	JT	2.13	JT	3.7	NJT	240	T	***12***	***JNT***	2,300	T	--	
SE	LW2-G401	LW2-G401	N	10/22/2004	Surface Sediment	0	27	cm	7629036	700154	-10	4.88	NJT	4.08	NJT	2.8	JNT	440	T	***11.8***	***JNT***	4,200	T	--	
SE	LW2-G403	LW2-G403	N	10/22/2004	Surface Sediment	0	26	cm	7629824	700114	-4	0.975	JNT	0.374	NJT	0.12	JT	19	JT	1.5	JNT	210	JT	2.35	UT
SE	LW2-G404	LW2-G404	N	8/27/2004	Surface Sediment	0	25	cm	7629091	700096	-9	4.9	JNT	3.6	JT	2.79	JT	140	T	***11***	***JNT***	1,200	T	***53***	***JT***
SE	LW2-G407	LW2-G407	N	9/9/2004	Surface Sediment	0	29	cm	7630154	699834	-12	2.3	JNT	2.13	JT	0.33	NJT	31	T	4.8	JNT	340	T	***100***	***JT***
SE	LW2-G413	LW2-G413	N	10/22/2004	Surface Sediment	0	27	cm	7630351	699536	2	0.501	JNT	0.38	JT	0.12	NJT	13	JT	0.99	JNT	120	JT	***55.5***	***JT***
SE	LW2-GBT020	LW2-GBT020	N	12/6/2005	Surface Sediment	0	10	cm	7628495	701060	-13	23.8	T	3.7	T	125	T	36	T	***150***	***T***	360	JT	--	
SE	LW2-GBT021	LW2-GBT021	N	12/13/2005	Surface Sediment	0	10	cm	7629274	700117	-6	5.14	T	2.6	JT	0.62	JT	51	T	***8.4***	***JT***	430	T	--	
SE	LW2-NA3A	NA-3A-0005	N	10/22/2004	Surface Sediment	0	5	cm	7629429	700328	-21	--		--		--		--		--		--		--	
SE	LW2-NA3A	NA-3A-0508	N	10/22/2004	Subsurface Sediment	5	8	cm	7629429	700328	-21	--		--		--		--		--		--		--	
SE	LW2-NA3A	NA-3A-0812	N	10/22/2004	Subsurface Sediment	8	12	cm	7629429	700328	-21	--		--		--		--		--		--		--	
SE	LW2-NA3A	NA-3A-1216	N	10/22/2004	Subsurface Sediment	12	16	cm	7629429	700328	-21	--		--		--		--		--		--		--	
SE	LW2-NA3A	NA-3A-1620	N	10/22/2004	Subsurface Sediment	16	20	cm	7629429	700328	-21	--		--		--		--		--		--		--	
SE	LW2-NA3A	NA-3A-2024	N	10/22/2004	Subsurface Sediment	20	24	cm	7629429	700328	-21	--		--		--		--		--		--		--	
SE	LW2-NA3A	NA-3A-2428	N	10/22/2004	Subsurface Sediment	24	28	cm	7629429	700328	-21	--		--		--		--		--		--		--	
SE	LW2-NA3A	NA-3A-2832	N	10/22/2004	Subsurface Sediment	28	32	cm	7629429	700328	-21	--		--		--		--		--		--		--	
SE	LW2-NA3A	NA-3A-3238	N	10/22/2004	Subsurface Sediment	32	38	cm	7629429	700328	-21	--		--		--		--		--		--		--	
SE	LW2-NA3A	NA-3A-3844	N	10/22/2004	Subsurface Sediment	38	44	cm	7629429	700328	-21	--		--		--		--		--		--		--	
SE	LW2-NA3A	NA-3A-4854	N	10/22/2004	Subsurface Sediment	48	54	cm	7629429	700328	-21	--		--		--		--		--		--		--	
SE	LW2-NA3A	NA-3A-5864	N	10/22/2004	Subsurface Sediment	58	64	cm	7629429	700328	-21	--		--		--		--		--		--		--	
SE	LW2-NA3A	NA-3A-6874	N	10/22/2004	Subsurface Sediment	68	74	cm	7629429	700328	-21	--		--		--		--		--		--		--	
SE	LW2-NA3A	NA-3A-7884	N	10/22/2004	Subsurface Sediment	78	84	cm	7629429	700328	-21	--		--		--		--		--		--		--	
SE	LW2-NA3A	NA-3A-8894	N	10/22/2004	Subsurface Sediment	88	94	cm	7629429	700328	-21	--		--		--		--		--		--		--	
SE	LW2-NA3B	NA-3B-0034	N	10/22/2004	Surface Sediment	0	34	cm	7629433	700322	-20	7.5	JNT	5.11	JT	0.51	JT	91	JT	***13***	***JNT***	1,100	JT	***68.9***	***JT***
SE	LW2-NA3B	NA-3B-3464	N	10/22/2004	Subsurface Sediment	34	64	cm	7629433	700322	-20	5.03	NJT	2.1	T	0.061	UT	66	JT	***7.2***	***NJT***	730	JT	***26***	***JT***
SE	LW2-NA3B	NA-3B-6494	N	10/22/2004	Subsurface Sediment	64	94	cm	7629433	700322	-20	11.2	JT	4.61	JT	1.4	JT	88	JT	***17.2***	***JT***	1,300	JT	***74***	***JT***
SE	LW3-C688	LW3-C688-B	N	1/15/2008	Subsurface Sediment	30	114	cm	7628760	701367	-5	2.6	NJT	2.4	JT	10	T	29	T	***15***	***JNT***	250	JT	--	
SE	LW3-C688	LW3-C688-C	N	1/15/2008	Subsurface Sediment	114	221	cm	7628760	701367	-5	3.8	T	4	T	22	T	25	T	***30***	***T***	250	JT	--	
SE	LW3-C688	LW3-C688-D	N	1/15/2008	Subsurface Sediment	221	281	cm	7628760	701367	-5	***200***	***JT***	***450***	***T***	***670***	***T***	48	T	***1,300***	***JT***	540	T	--	
SE	LW3-C688	LW3-C688-F	N	1/15/2008	Subsurface Sediment	404	457	cm	7628760	701367	-5	46	T	7.9	UT	30	T	160	JT	***81***	***T***	2,700	JT	--	
SE	LW3-C690	LW3-C690-B	N	1/15/2008	Subsurface Sediment	30	151	cm	7628470	701000	-11	10	JT	4.6	JT	3.4	JT	46	T	***18***	***JT***	540	T	--	
SE	LW3-C690	LW3-C690-C	N	1/15/2008	Subsurface Sediment	151	274	cm	7628470	701000	-11	25	JT	11	JT	29	JT	73	T	***64***	***JT***	850	T	--	
SE	LW3-C690	LW3-C690-D	N	1/15/2008	Subsurface Sediment	274	346	cm	7628470	701000	-11	41	JT	31	JNT	11	T	150	T	***83***	***JNT***	2,000	JT	--	
SE	LW3-C690	LW3-C690-E	N	1/15/2008	Subsurface Sediment	346	471	cm	7628470	701000	-11	64	NJT	17	T	11	JT	360	T	***92***	***JNT***	6,600	T	--	
SE	LW3-C695	LW3-C695-B	N	12/20/2007	Subsurface Sediment	30	95	cm	7629190	700056	-6	7.3	NJT	4.3	JT	15	T	140	T	***27***	***JNT***	1,500	T	***73***	***JT***
SE	LW3-C695	LW3-C695-D	N	12/20/2007	Subsurface Sediment	221	343	cm	7629190	700056	-6	16	T	19	NJT	21	T	200	T	***57***	***NJT***	3,000	T	***750***	***JT***
SE	LW3-C695	LW3-C695-E	N	12/20/2007	Subsurface Sediment	343	384	cm	7629190	700056	-6	14	T	21	NJT	27	NJT	110	T	***62***	***NJT***	2,500	T	***930***	***T***
SE	LW3-C698	LW3-C698-B	N	12/20/2007	Subsurface Sediment	30	152	cm	7630300	699718	-16	3.7	NJT	3.7	JT	3	JT	31	T	***10***	***JNT***	330	T	***56***	***JT***
SE	LW3-G692-1	LW3-G692-1	N	12/4/2007	Surface Sediment	0	30	cm	7628730	701055	-20	7.5	T	3.7	T	11	T	61	T	***23***	***T***	750	T	--	
SE	LW3-G692-2	LW3-G692-2	FR	12/4/2007	Surface Sediment	0	28	cm	7628730	701048	-20	5.9	JT	2.8	JT	7.3	JT	56	T	***16***	***JT***	650	T	--	
SE	LW3-G694	LW3-G694	N	11/16/2007	Surface Sediment	0	20	cm	7629340	700318	-23	3.7	T	6.3	JT	4.6	JT	100	T	***15***	***JT***	1,300	T	***89***	***JT***
SE	LW3-GSP07W	LW3-GSP07W	N	10/15/2007	Surface Sediment	0	19	cm	7628812	700370	-14	0.46	JT	0.37	NJT	0.18	UT	4.2	JT	0.98	JNT	60	JT	--	

Table A-1. RI/FS Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

CAS			Sample	Sample		Start	End	Depth			Elevation	LWG RA Sum DDD (Calculated U = 1/2)		LWG RA Sum DDE (Calculated U = 1/2)		LWG RA Sum DDT (Calculated U = 1/2)		LWG RA Total cPAH/BaPE		LWG RA Total DDx (Calculated U = 1/2)		LWG RA Total PAH (Calculated U = 1/2)		LWG RA Total PCB Aroclors (Calculated U = 1/2)	
Matrix	Location ID	Sample Name	Type	Date	Depth Class	Depth	Depth	Units	X Coordinate	Y coordinate	(NAVD88)	LRASum_DDD_N		LRASum_DDE_N		LRASum_DDT_N		LRAtcPAHTEF7_N		LRAtDDT_N		LRAtPAH_17_N		LRAtPCB_N	
												µg/kg		µg/kg		µg/kg		µg/kg		µg/kg		µg/kg		µg/kg	
											Sediment/Bank Soil CUL	**114**		**50**		**246**		**774**		**6.1**		**23,000**		**9**	
											Sitewide RAL	--		--		--		**NA**		**160**		**30,000**		**75**	
											PTW Threshold	--		--		--		**774,000**		**7,050**		**NA**		**200**	
SE	LWG0108R002SDS015C00	LWG0108R002SDS015C00	N	10/17/2002	Surface Sediment	0	15	cm	7630259	699666	1	0.92	UT	1.3	UT	1.2	UT	36	T	1.3	UT	420	T	***36***	***T***
SE	TOSC099DMMU1	TOSC099DMMU1SDO1	N	1/21/1999	Subsurface Sediment	0	304	cm	7629016	700391	-24	11	AJT	8	AJT	2	AUJT	250	T	***20***	***AJT***	3,300	T	***260***	***JT***
SE	TOSC099DMMU1	TOSC099DMMU1SDO3	FR	1/21/1999	Subsurface Sediment	0	304	cm	7629016	700391	-24	12	AJT	8	AJT	13	AJT	190	T	***33***	***AJT***	3,200	T	***350***	***JT***
SE	TOSC099DMMU2	TOSC099DMMU2SDO4	N	1/21/1999	Subsurface Sediment	0	164	cm	7629278	700566	-29	20	AJT	8	AJT	24	AJT	110	T	***52***	***AJT***	1,600	T	***99***	***JT***
SE	W-09-A-PG	LWG2-PG-W9A	N	11/30/2005	Surface Sediment	0	23	cm	7628899	700250	3	--		--		--		0.82	JT	--		12	JT	--	
SE	W-09-C-PG	LWG2-PG-W9C	N	11/30/2005	Surface Sediment	0	30	cm	7628988	700306	-20	--		--		--		57	T	--		580	T	--	
SE	WLCASF97S001	WLCASF97S001W4159	N	6/12/1997	Surface Sediment	0	10	cm	7630379	699598	-9	--		--		--		75	T	--		730	T	--	
SE	WLCASF97S002	WLCASF97S002W4158	N	6/12/1997	Surface Sediment	0	10	cm	7630047	700105	-32	--		--		--		38	T	--		420	T	--	
SE	WLCASF97S003	WLCASF97S003W4157	N	6/12/1997	Surface Sediment	0	10	cm	7629493	700297	-17	--		--		--		82	T	--		1,100	T	--	
SE	WLCASF97S004	WLCASF97S004W4156	N	6/12/1997	Surface Sediment	0	10	cm	7629322	700277	-23	--		--		--		110	T	--		1,700	T	--	
SE	WLCASF97S005	WLCASF97S005W4155	N	6/12/1997	Surface Sediment	0	10	cm	7629019	700128	-5	--		--		--		***852***	***T***	--		9,000	T	--	
SE	WLCASF97S006	WLCASF97S006W4154	N	6/12/1997	Surface Sediment	0	10	cm	7628791	700365	-2	--		--		--		66	T	--		1,000	T	--	
SE	WLCASF97S008	WLCASF97S008W4152	N	6/12/1997	Surface Sediment	0	10	cm	7629266	700704	-35	--		--		--		48	T	--		800	T	--	
SE	WLCASF97S009	WLCASF97S009W4172	N	6/11/1997	Surface Sediment	0	10	cm	7628648	700612	-32	--		--		--		120	T	--		1,400	T	--	
SE	WLCASF97S010	WLCASF97S010W4171	N	6/11/1997	Surface Sediment	0	10	cm	7629081	700977	-34	--		--		--		72	T	--		1,400	T	--	
SE	WLCASF97S011	WLCASF97S011W4170	N	6/11/1997	Surface Sediment	0	10	cm	7628514	700853	-19	--		--		--		110	T	--		2,800	T	--	
SE	WLCASF97S012	WLCASF97S012W4168	N	6/11/1997	Surface Sediment	0	10	cm	7628858	701245	-20	--		--		--		56	T	--		680	T	--	
SE	WLCASF97S012	WLCASF97S012W4169	FR	6/11/1997	Surface Sediment	0	10	cm	7628858	701245	-20	--		--		--		56	T	--		680	T	--	
SE	WLCCWI08ANCTPD03	WLCCWI08ANCTPD03	N	5/29/2009	Surface Sediment	0	2	cm	7629152	700952	-39	--		--		--		--		--		--		4	UT
SE	WLCCWI08ANCTPD06	WLCCWI08ANCTPD06	N	5/29/2009	Surface Sediment	0	2	cm	7629255	700767	-38	--		--		--		--		--		--		3.9	UT
SE	WLCCWI08DMMU1A	WLCCWI08DMMU1A	N	5/29/2009	Surface Sediment	0	2	cm	7628747	700605	-35	--		--		--		--		--		--		***19***	***T***
SE	WLCCWI08DMMU1A	WLCCWI08DMMU1A4041	N	9/2/2008	Subsurface Sediment	91	121	cm	7628747	700605	-35	3.31	AJT	3.02	AJT	13.4	AUJT	91.8	JT	***13***	***AJT***	1,500	JT	***37.9***	***JT***
SE	WLCCWI08DMMU1A	WLCCWI08DMMU1A4142	N	9/2/2008	Subsurface Sediment	121	152	cm	7628747	700605	-35	3.25	AJT	2.45	AUJT	12.3	AUJT	120	JT	***10.6***	***AJT***	1,600	JT	12.2	UT
SE	WLCCWI08DMMU1ABC	WLCCWI08DMMU1ABC	N	9/4/2008	Subsurface Sediment	91	182	cm	7628839	700680	-33	3.11	AJT	2.91	AJT	16.7	AJT	110	JT	***22.7***	***AJT***	1,700	JT	15.3	UT
SE	WLCCWI08DMMU1B	WLCCWI08DMMU1B	N	5/29/2009	Surface Sediment	0	2	cm	7628834	700677	-33	--		--		--		--		--		--		***20***	***T***
SE	WLCCWI08DMMU1B	WLCCWI08DMMU1B4041	N	9/3/2008	Subsurface Sediment	121	152	cm	7628834	700677	-33	2.03	AJT	2.63	AJT	13.3	AUJT	75.4	JT	***11.3***	***AJT***	1,400	JT	13.5	UT
SE	WLCCWI08DMMU1B	WLCCWI08DMMU1B4142	N	9/3/2008	Subsurface Sediment	152	182	cm	7628834	700677	-33	3.81	AJT	2.44	AJT	11.7	AUJT	136	JT	***12.1***	***AJT***	1,740	JT	11.6	UT
SE	WLCCWI08DMMU1C	WLCCWI08DMMU1C	N	5/29/2009	Surface Sediment	0	5	cm	7628936	700759	-33	--		--		--		--		--		--		***18***	***T***
SE	WLCCWI08DMMU1C	WLCCWI08DMMU1C4041	N	9/4/2008	Subsurface Sediment	91	121	cm	7628936	700759	-33	4.02	AUJT	5.33	AJT	13.2	AUJT	64.7	JT	***13.9***	***AJT***	1,400	JT	***34.3***	***JT***
SE	WLCCWI08DMMU1C	WLCCWI08DMMU1C4142	N	9/4/2008	Subsurface Sediment	121	152	cm	7628936	700759	-33	2.89	AJT	2.93	AJT	11.6	AUJT	79.1	JT	***11.6***	***AJT***	996.4	JT	11.6	UT
SE	WLCCWI08DMMU2A	WLCCWI08DMMU2A	N	5/29/2009	Surface Sediment	0	2	cm	7628879	700461	-33	--		--		--		--		--		--		4	UT
SE	WLCCWI08DMMU2A	WLCCWI08DMMU2A4041	N	9/5/2008	Subsurface Sediment	152	182	cm	7628879	700461	-33	1.98	AJT	2.61	AJT	6.48	AUT	100	JT	***7.83***	***AJT***	1,500	JT	13.1	UT
SE	WLCCWI08DMMU2A	WLCCWI08DMMU2A4142	N	9/5/2008	Subsurface Sediment	182	213	cm	7628879	700461	-33	3.68	T	2.5	AUT	12.5	AUT	132	T	***11***	***AT***	1,600	T	12.5	UT
SE	WLCCWI08DMMU2B	WLCCWI08DMMU2B	N	5/29/2009	Surface Sediment	0	2	cm	7628979	700542	-33	--		--		--		--		--		--		3.9	UT
SE	WLCCWI08DMMU2B	WLCCWI08DMMU2B4041	N	9/5/2008	Subsurface Sediment	91	121	cm	7628979	700542	-33	4.04	T	3.83	AJT	9.93	AJT	160	JT	***17.8***	***AJT***	2,700	JT	13	UT
SE	WLCCWI08DMMU2B	WLCCWI08DMMU2B4142	N	9/5/2008	Subsurface Sediment	121	152	cm	7628979	700542	-33	6.14	AJT	3.59	AJT	8.51	AJT	150	JT	***18.2***	***AJT***	2,300	JT	11.8	UT
SE	WLCCWI08DMMU2C	WLCCWI08DMMU2C	N	5/29/2009	Surface Sediment	0	2	cm	7629098	700580	-33	--		--		--		--		--		--		***17***	***T***
SE	WLCCWI08DMMU2C	WLCCWI08DMMU2C4041	N	9/5/2008	Subsurface Sediment	60	91	cm	7629098	700580	-33	2.62	AJT	2.72	AJT	10.8	AJT	310	T	***16.1***	***AJT***	2,800	JT	15.5	UT
SE	WLCCWI08DMMU2C	WLCCWI08DMMU2C4142	N	9/5/2008	Subsurface Sediment	91	121	cm	7629098	700580	-33	3.96	AJT	2.54	AJT	11.7	AUJT	123	JT	***12.4***	***AJT***	1,840	JT	11.7	UT
SE	WLCCWI08DMMU3A	WLCCWI08DMMU3A	N	5/29/2009	Surface Sediment	0	2	cm	7628650	700651	-24	--		--		--		--		--		--		***19***	***T***
SE	WLCCWI08DMMU3A	WLCCWI08DMMU3A13	N	9/8/2008	Subsurface Sediment	30	91	cm	7628650	700651	-24	3.69	AJT	2.53	AUJT	12.7	AUJT	216	JT	***11.3***	***AJT***	2,700	JT	12.6	UT
SE	WLCCWI08DMMU3B	WLCCWI08DMMU3B	N	5/29/2009	Surface Sediment	0	2	cm	7628779	700408	-18	--		--		--		--		--		--		***560***	***T***
SE	WLCCWI08DMMU3B	WLCCWI08DMMU3B13	N	9/8/2008	Subsurface Sediment	30	91	cm	7628779	700408	-18	***655***	***AJT***	40.7	AJT	16.5	AUJT	297	T	***704***	***AJT***	3,530	T	***261***	***JT***
SE	WLCCWI08DMMU3D	WLCCWI08DMMU3D	N	5/29/2009	Surface Sediment	0	2	cm	7628873	700325	-13	--		--		--		--		--		--		3.9	UT
SE	WLCCWI08DMMU3D	WLCCWI08DMMU3D13	N	9/8/2008	Subsurface Sediment	30	91	cm	7628873	700325	-13	2.15	AJT	1.57	AUJT	7.86	AUJT	300	JT	***6.87***	***AJT***	8,600	JT	7.79	UT
SE	WLCCWI08DMMU3E	WLCCWI08DMMU3E	N	5/29/2009	Surface Sediment	0	2	cm	7628981	700402	-24	--		--		--		--		--		--		***19***	***T***
SE	WLCCWI08DMMU3E	WLCCWI08DMMU3E13	N	9/5/2008	Subsurface Sediment	30	91	cm	7628981	700402	-24	4.2	T	3.34	T	11.7	AUT	122	T	***13***	***AT***	1,200	T	11.8	UT
SE	WLCDRD05PG074	WLCDRD05PG07474	N	5/25/2005	Surface Sediment	0	30	cm	7628952	701290	-24	4.5	NJT	4.3	T	9	JT	27	T	***18***	***JNT***	400	JT	***46***	***T***
SE	WLCOFJ022201	WLCOFJ02220122010	N	10/21/2002	Surface Sediment	0	10	cm	7629006	700111	-2	2.47	UJT	2.5	UJT	2.47	UJT	53.5	JT	2.5	UJT	450	JT	--	
SE	WLCOFJ022202	WLCOFJ02220222020	N	10/21/2002	Surface Sediment	0	15	cm	7629048	700077	-3	5.85	JT	3.17	JT	4.6	JT	169	JT	***13.6***	***JT***	2,400	JT	--	
SE	WLCOFJ022203	WLCOFJ02220322030	N	10/21/2002	Surface Sediment	0	15	cm	7628994	700157	-4	9.95	JT	4.61	JT	4.86	JT	***3,600***	***T***	***19.4***	***JT***	***28,000***	***T***	--	
SE	WLCOFJ0222031	WLCOFJ022203122031	FR	10/21/2002	Surface Sediment	0	15	cm	7628991	700159	-4	7.45	JT	3.08	JT	13.3	JT	243	JT	***23.8***	***JT***	1,760	JT	--	
SE	WLCOFJ022204	WLCOFJ02220422040	N	10/21/2002	Surface Sediment	0	12	cm	7629090	700117	-16	4.01	UJT	4.01	UJT	4.01	UJT	190	T	4.01	UJT	1,900	JT	--	
SE	WLCOFJ022205	WLCOFJ02220522050	N	10/21/2002	Surface Sediment	0	12	cm	7629070	700151	-18	7.76	JT	6	JT	4.14	UJT	570	T	***16.4***	***JT***	6,400	JT	--	
SE	WLCOFJ022206	WLCOFJ02220622060	N	10/21/2002	Surface Sediment	0	10	cm	7629040	700177	-19	5.47	JT	3.76	UJT	3.76	UJT	180	JT	***10.1***	***JT***	1,600	JT	--	
SE	WLCPWL09APS-1	WLCPWL09APS-1B	N	12/29/2009	Subsurface Sediment	60	121	cm	7628973	700319	-21	2.3	AJT	2.5	AJT	5.1	AJT	120	JT	***9.9***	***AJT***	1,300	JT	***56***	***JT***
SE	WLCPWL09APS-1	WLCPWL09APS-1T	N	12/29/2009	Subsurface Sediment	0	60	cm	7628973	700319	-21	2	AJT	2.4	AJT	1	AUJT	97	JT	4.9	AJT	1,100	JT	***49***	***JT***
SE	WLCPWL09APS-2	WLCPWL09APS-2B	N	12/29/2009	Subsurface Sediment	60	121	cm	7629116	700296	-15	0.87	AJT	1.2	AJT	0.9	AUJT	100	JT	2.5	AJT	1,100	JT	***40***	***JT***
SE	WLCPWL09APS-2	WLCPWL09APS-2T	N	12/29/2009	Subsurface Sediment	0	60	cm	7629116	700296	-15	1.2	AJT	1.9	AJT	1.1	AUJT	86	JT	3.7	AJT	1,400	JT	***51***	***JT***
SE	WLCPWL09APS-3	WLCPWL09APS-3B	N	12/29/2009	Subsurface Sediment	60	121	cm	7629238	700264	-20	4.1	AJT	7.6	AJT	1.9	AJT	100	JT	***14***	***AJT***	1,500	JT	***110***	***JT***
SE	WLCPWL09APS-3	WLCPWL09APS-3T	N	12/29/2009	Subsurface Sediment	0	60	cm	7629238	700264	-20	2.7	AJT	6.2	AJT	1.6	AJT	120	JT	***11***	***AJT***	1,700	JT	***86***	***JT***
SE	WLCPWL09DRB-1	WLCPWL09DRB-1Z	N	12/29/2009	Subsurface Sediment	320	350	cm	7629031	700383	-22	1.6	AJT	4.3	AJT	2.9	AJT	81	JT	***8.8***	***AJT***	1,400	JT	***79***	***JT***
SE	WLCPWL09DRB-2	WLCPWL09DRB-2Z	N	12/29/2009	Subsurface Sediment	307	338	cm	7629105	700447	-25	1.5	AJT	3.6	AJT	2.3	AJT	130	JT	***7.4***	***AJT***	2,300	JT	***66***	***JT***
SE	WLCPWL09DRB-3	WLCPWL09DRB-3Z	N	12/29/2009	Subsurface Sediment	280	310	cm	7629193	700495	-25	2.1	AJT	4.2	AJT	2.8	AJT	280	JT	***9.1***	***AJT***	4,100	JT	***82***	***JT***
SE	WLCPWL09DRB-D	WLCPWL09DRB-DP	N	12/29/2009	Subsurface Sediment	0	302	cm	7629110	700442	-24	1.9	AJT	2.7	AJT	20	AJT	130	JT	***25***	***AJT***	1,700	JT	***54***	***JT***
SE	WLCPWL09URB-1	WLCPWL09URB-1Z	N	12/29/2009	Subsurface Sediment	295	326	cm	7629179	700364	-17	4.3	AJT	5.3	AJT	2.3	AJT	170	JT	***12***	***AJT***	2,200	JT	***130***	***JT***
SE	WLCPWL09URB-2	WLCPWL09URB-2Z	N	12/29/2009	Subsurface Sediment	118	149	cm	7629295	700478	-23	64	AJT	44	AJT	19	AJT	420	JT	***130***	***AJT***	9,900	JT	***200***	***JT***
SE	WLCPWL09URB-3	WLCPWL09URB-3Z	N	12/29/2009	Subsurface Sediment	167	198	cm	7629268	700417	-21	3.3	AJT	3.8	AJT	1.1	AJT	120	JT	***8.2***	***AJT***	1,400	JT	***69***	***JT***
SE	WLCPWL09URB-D	WLCPWL09URB-DP	N	12/29/2009	Subsurface Sediment	0	194	cm	7629248	700420	-20	2.5	AJT	4.2	AJT	2.9	AJT	110	JT	***9.6***	***AJT***	2,100	JT	***83***	***TJ***

Table A-1. RI/FS Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

CAS			Sample	Sample		Start	End	Depth				LWG RA Sum DDD (Calculated U = 1/2)		LWG RA Sum DDE (Calculated U = 1/2)		LWG RA Sum DDT (Calculated U = 1/2)		LWG RA Total cPAH/BaPE		LWG RA Total DDx (Calculated U = 1/2)		LWG RA Total PAH (Calculated U = 1/2)		LWG RA Total PCB Aroclors (Calculated U = 1/2)	
Matrix	Location ID	Sample Name	Type	Date	Depth Class	Depth	Depth	Units	X Coordinate	Y coordinate	Elevation (NAVD88)	LRASum_DDD_N		LRASum_DDE_N		LRASum_DDT_N		LRAtcPAHTEF7_N		LRAtDDT_N		LRAtPAH_17_N		LRAtPCB_N	
												µg/kg		µg/kg		µg/kg		µg/kg		µg/kg		µg/kg		µg/kg	
											Sediment/Bank Soil CUL	**114**		**50**		**246**		**774**		**6.1**		**23,000**		**9**	
											Sitewide RAL	--		--		--		**NA**		**160**		**30,000**		**75**	
											PTW Threshold	--		--		--		**774,000**		**7,050**		**NA**		**200**	
SE	WLCWTG02ANCTPD01	WLCWTG02ANCTPD01A	N	7/16/2002	Surface Sediment	0	2	cm	7628756	700622	-35	5.7	T	5.4	T	7.6	T	97	T	***19***	***T***	1,400	T	***120***	***JT***
SE	WLCWTG02ANCTPD01	WLCWTG02ANCTPD01B	N	7/16/2002	Surface Sediment	0	15	cm	7628756	700622	-35	8.3	T	5.5	T	13	T	130	T	***27***	***T***	1,900	T	***110***	***JT***
SE	WLCWTG02ANCTPD02	WLCWTG02ANCTPD02A	N	7/16/2002	Surface Sediment	0	2	cm	7628958	700792	-34	6.2	T	4.9	T	3.4	T	77	T	***15***	***T***	1,100	T	***110***	***JT***
SE	WLCWTG02ANCTPD02	WLCWTG02ANCTPD02B	N	7/16/2002	Surface Sediment	0	15	cm	7628958	700792	-34	2.1	T	4.6	T	2	AUT	49	T	***7.7***	***AT***	690	T	***91***	***JT***
SE	WLCWTG02ANCTPD03	WLCWTG02ANCTPD03A	N	7/16/2002	Surface Sediment	0	2	cm	7629152	700954	-39	2.8	AUT	3.5	T	8.8	T	96	T	***14***	***AT***	1,700	T	***130***	***JT***
SE	WLCWTG02ANCTPD03	WLCWTG02ANCTPD03B	N	7/16/2002	Surface Sediment	0	15	cm	7629152	700954	-39	3	T	6.2	T	3.4	T	35	T	***13***	***T***	460	T	***140***	***T***
SE	WLCWTG02ANCTPD05	WLCWTG02ANCTPD09R	FR	7/16/2002	Surface Sediment	0	15	cm	7629438	700917	-40	2.3	AUT	3.3	T	2.3	AUT	26	T	5.6	AT	210	T	***91***	***JT***
SE	WLCWTG02ANCTPD06	WLCWTG02ANCTPD06A	N	7/16/2002	Surface Sediment	0	2	cm	7629255	700769	-38	3.4	T	3.2	T	11	T	140	T	***18***	***T***	1,400	T	***130***	***JT***
SE	WLCWTG02ANCTPD06	WLCWTG02ANCTPD06B	N	7/16/2002	Surface Sediment	0	15	cm	7629255	700769	-38	3.2	AT	3.6	AT	4.5	AT	84	T	***11***	***AT***	1,100	T	***110***	***JT***
SE	WLCWTG02ANCTPD07	WLCWTG02ANCTPD07A	N	7/16/2002	Surface Sediment	0	2	cm	7629084	700617	-34	2.8	AUT	4.5	T	2.8	AUT	63	T	***7.3***	***AT***	880	T	***120***	***JT***
SE	WLCWTG02ANCTPD07	WLCWTG02ANCTPD07B	N	7/16/2002	Surface Sediment	0	15	cm	7629084	700617	-34	5	T	4.1	T	2.3	AUT	33	T	***10***	***AT***	320	T	***92***	***JT***
SE	WLCWTG02ANCTPD08	WLCWTG02ANCTPD08A	N	7/16/2002	Surface Sediment	0	2	cm	7628892	700455	-33	3.2	AUT	3.6	T	3.2	T	77	T	***8.4***	***AT***	1,100	T	***130***	***JT***
SE	WLCWTG02ANCTPD08	WLCWTG02ANCTPD08B	N	7/16/2002	Surface Sediment	0	15	cm	7628892	700455	-33	3.2	T	3.3	T	4.2	T	67	T	***11***	***T***	1,100	T	***120***	***JT***
SE	WLCWTI00N20	WLCWTI00N20N20	N	9/22/2000	Subsurface Sediment	15	30	cm	7628972	700109	9	--		--		--		--		--		--		--	
SE	WLCWTI00N40	WLCWTI00N40N40	N	9/22/2000	Subsurface Sediment	15	30	cm	7628959	700126	8	--		--		--		--		--		--		--	
SE	WLCWTI00S20	WLCWTI00S20S20	N	9/22/2000	Subsurface Sediment	15	30	cm	7628998	700078	8	--		--		--		--		--		--		--	
SE	WLCWTI00S40	WLCWTI00S40S40	N	9/22/2000	Subsurface Sediment	15	30	cm	7629011	700062	9	--		--		--		--		--		--		--	
SE	WLCWTI00TP11	WLCWTI00TP11TP11	N	9/21/2000	Subsurface Sediment	122	137	cm	7628955	700089	13	--		--		--		--		--		--		--	
SE	WLRWTF98ATXSD3	WLRWTF98ATXSD3ATXSD3	N	12/18/1998	Surface Sediment	0	12	cm	7628416	701026	-8	10.6	T	3.35	AUT	3.35	AUT	--		***14***	***AT***	--		--	
SE	WLRWTF98ATXSD4	WLRWTF98ATXSD4ATXSD4	N	12/18/1998	Surface Sediment	0	12	cm	7628460	700769	5	6.7	AUT	6.7	AUT	3.35	AUT	8.2	T	6.7	AUT	73	T	--	
SE	WLRWTF98ATXSD5	WLRWTF98ATXSD5ATXSD5	N	12/18/1998	Surface Sediment	0	12	cm	7628584	700620	5	6.7	AUT	6.7	AUT	3.35	AUT	7	UJT	6.7	AUT	500	UJT	--	
SE	WLRWTF98HEVSD1	WLRWTF98HEVSD1HEVSD1	N	12/17/1998	Surface Sediment	0	12	cm	7628832	700321	1	--		--		--		7	UJT	--		500	UJT	--	
SE	WLRWTF98HEVSD2	WLRWTF98HEVSD2HEVSD2	N	12/17/1998	Surface Sediment	0	12	cm	7628783	700396	-15	3.35	AUT	3.35	AUT	3.35	AUT	7	UJT	3.35	AUT	500	UJT	--	
SE	WLRWTF98HEVSD3	WLRWTF98HEVSD3HEVSD3	N	12/17/1998	Surface Sediment	0	12	cm	7628708	700471	0	3.35	AUT	3.35	AUT	3.35	AUT	7	UJT	3.35	AUT	500	UJT	--	
SE	WLRWTF98TOSSD1	WLRWTF98TOSSD1TOSSD1	N	12/17/1998	Surface Sediment	0	12	cm	7628882	700247	6	--		--		--		7	UJT	--		500	UJT	--	
SE	WLRWTF98TOSSD2	WLRWTF98TOSSD2TOSSD2	N	12/17/1998	Surface Sediment	0	12	cm	7628955	700135	7	--		--		--		13.4	UT	--		125	T	--	
SE	WLRWTF98TOSSD3	WLRWTF98TOSSD3TOSSD3	N	12/17/1998	Surface Sediment	0	12	cm	7629084	700168	-12	--		--		--		205	JT	--		3,400	JT	--	
SE	WLRWTF98TOSSD4	WLRWTF98TOSSD4TOSSD4	N	12/17/1998	Surface Sediment	0	12	cm	7629054	699987	13	--		--		--		6.7	UT	--		6.7	UT	--	
SE	WLRWTF98TOSSD5	WLRWTF98TOSSD5TOSSD5	N	12/18/1998	Surface Sediment	0	12	cm	7629232	699945	9	--		--		--		8.7	JT	--		350	JT	--	
SE	WR-WSI98SD104	WR-WSI98SD1040	N	9/23/1997	Surface Sediment	0	10	cm	7628572	701218	-1	--		--		--		73	T	--		600	T	--	
SE	WR-WSI98SD105	WR-WSI98SD1050	N	9/23/1997	Surface Sediment	0	10	cm	7628878	701254	-20	--		--		--		56	T	--		640	T	--	
SE	WR-WSI98SD107	WR-WSI98SD1070	N	9/23/1997	Surface Sediment	0	10	cm	7628533	700849	-19	--		--		--		110	T	--		2,000	T	--	
SE	WR-WSI98SD110	WR-WSI98SD1100	N	9/23/1997	Surface Sediment	0	10	cm	7629093	700983	-35	--		--		--		65	T	--		1,000	T	--	
SE	WR-WSI98SD113	WR-WSI98SD1130	N	9/23/1997	Surface Sediment	0	10	cm	7629298	700704	-35	--		--		--		71	T	--		940	T	--	
SE	WR-WSI98SD114	WR-WSI98SD1140	N	9/23/1997	Surface Sediment	0	10	cm	7629023	700135	-8	--		--		--		240	T	--		2,000	NT	--	
SE	WR-WSI98SD115	WR-WSI98SD1150	N	9/23/1997	Surface Sediment	0	10	cm	7629434	700515	-24	--		--		--		49	T	--		430	T	--	
SE	WR-WSI98SD117	WR-WSI98SD1170000A	N	10/16/1997	Subsurface Sediment	0	90	cm	7629340	700260	-22	20	T	5.2	T	22	T	99	T	***47***	***T***	1,600	T	***160***	***JT***
SE	WR-WSI98SD117	WR-WSI98SD1170000CC	N	9/23/1997	Surface Sediment	0	10	cm	7629340	700260	-22	3.3	AJT	2	AJT	3.25	AJT	99	T	***8.6***	***AJT***	930	T	***96***	***T***
SE	WR-WSI98SD118	WR-WSI98SD1180	N	9/23/1997	Surface Sediment	0	10	cm	7629521	700296	-16	--		--		--		270	T	--		2,300	JT	--	
SE	WR-WSI98SD123	WR-WSI98SD1230	N	9/24/1997	Surface Sediment	0	10	cm	7630398	699583	-8	--		--		--		35	T	--		340	T	--	

Table A-1. RI/FS Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

CAS			Sample	Sample		Start	End	Depth			Elevation	LWG RA Total PCB Congener (Calculated U = 1/2)		Mercury		Naphthalene		Tributyltin ion		Zinc		Total Organic Carbon		Fines (silt + clay)		Total Clay		Total Silt		Total Gravel		Total Sand
Matrix	Location ID	Sample Name	Sample Type	Sample Date	Depth Class	Start Depth	End Depth	Depth Units	X Coordinate	Y coordinate	(NAVD88)	LRAtPCBCong_N		7439-97-6		91-20-3		36643-28-4		7440-66-6		TOC		GS-FINES		T-Clay		T-Silt		T-Gravel		T-Sand
												µg/kg		mg/kg		µg/kg		µg/kg		mg/kg		%		%		%		%		%		%
											Sediment/Bank Soil CUL	**9**		**0.085**		**NA**		**3,080**		**459**												
											Sitewide RAL	**75**		--		**NA**		--		--												
											PTW Threshold	**200**		--		**140,000**		--		--												
SE	LW2-B019	LW2-B019	N	7/26/2004	Surface Sediment	0	15	cm	7628948	700051	9	--		0.017	J	1.1	U	--		76.9		0.12		15.6		--		11.9		16.3		70.1
SE	LW2-C377	LW2-C377-B	N	11/10/2004	Subsurface Sediment	30	153	cm	7628521	701143	-5	--		0.049	J	10	U	--		82.7		2.05		75.5		18.1		57.4		0.3		21.7
SE	LW2-C377	LW2-C377-C	N	11/10/2004	Subsurface Sediment	153	273	cm	7628521	701143	-5	--		***0.096***	***J***	13	U	--		130		2.23		69.7		18.7		51.1		0.8		27.5
SE	LW2-C377	LW2-C377-D	N	11/10/2004	Subsurface Sediment	273	369	cm	7628521	701143	-5	--		--		44		--		--		2.61		73.3		15.4		57.9		0.4		25.0
SE	LW2-C377	LW2-C377-E	N	11/10/2004	Subsurface Sediment	369	482	cm	7628521	701143	-5	***65***	***JT***	***0.238***	***J***	170		--		127		2.3		64.2		10.0		54.3		0.3		34.2
SE	LW2-C401	LW2-C401-B	N	11/10/2004	Subsurface Sediment	30	69	cm	7629031	700153	-10	--		0.05	J	13	U	--		108		1.73		46.1		7.0		39.1		3.0		48.9
SE	LW2-C401	LW2-C401-C	N	11/10/2004	Subsurface Sediment	69	188	cm	7629031	700153	-10	--		0.058	J	150		--		114		0.9		22.3		5.1		17.2		1.5		76.0
SE	LW2-C401	LW2-C401-D	N	11/10/2004	Subsurface Sediment	188	278	cm	7629031	700153	-10	--		0.05	J	41	U	--		105		0.77		21.1		5.1		16.0		2.0		75.9
SE	LW2-C401	LW2-C401-E	N	11/10/2004	Subsurface Sediment	278	367	cm	7629031	700153	-10	***890***	***JT***	***0.089***	***J***	69		--		128		1.49		51.3		12.2		39.1		0.0		47.8
SE	LW2-C401	LW2-C401-F	N	11/10/2004	Subsurface Sediment	367	504	cm	7629031	700153	-10	--		--		8.6	U	--		--		0.43		76.4		15.3		61.0		--		24.1
SE	LW2-C403	LW2-C403-B	N	11/10/2004	Subsurface Sediment	30	153	cm	7629841	700123	-6	--		0.038		0.49	U	--		55.8		0.45		8.6		2.0		6.6		0.3		89.9
SE	LW2-C403	LW2-C403-C	N	11/10/2004	Subsurface Sediment	153	242	cm	7629841	700123	-6	--		0.009	U	0.45	U	--		49.7		0.05		0.8		0.3		0.5		0.1		97.6
SE	LW2-C403	LW2-C403-D	N	11/10/2004	Subsurface Sediment	242	361	cm	7629841	700123	-6	--		0.028		0.53	U	--		64.6		1.23		78.6		13.0		65.6		0.7		20.0
SE	LW2-C403	LW2-C403-F	N	11/10/2004	Subsurface Sediment	483	533	cm	7629841	700123	-6	--		--		0.57	U	--		68		1.65		90.6		20.1		70.5		--		4.0
SE	LW2-C413	LW2-C413-B1	N	10/26/2004	Subsurface Sediment	30	158	cm	7630354	699540	2	--		***0.213***		30		--		168		3.1		84.0		16.7		67.3		1.2		14.1
SE	LW2-C413	LW2-C413-C1	N	10/26/2004	Subsurface Sediment	158	299	cm	7630354	699540	2	--		***0.277***		25		--		177		2.05		77.0		16.1		60.8		1.1		19.1
SE	LW2-C413-2	LW2-C413-B2	FR	10/26/2004	Subsurface Sediment	30	152	cm	7630359	699540	1	--		***0.205***		39		--		198		2.19		54.0		7.6		46.4		2.1		38.0
SE	LW2-C413-2	LW2-C413-C2	FR	10/26/2004	Subsurface Sediment	152	274	cm	7630359	699540	1	--		***0.222***		22		--		201		2.61		78.0		14.3		63.8		1.6		20.7
SE	LW2-C413-2	LW2-C413-D2	N	10/26/2004	Subsurface Sediment	274	349	cm	7630359	699540	1	--		***0.357***	***T***	60		--		137	T	2.04		81.4		21.5		59.9		0.2		18.9
SE	LW2-C532	LW2-C532-A	N	10/18/2005	Surface Sediment	0	30	cm	7629257	700289	-21	--		***0.276***		62		--		170		2.56		83.3		11.1		72.2		0.1		15.2
SE	LW2-C532	LW2-C532-B	N	10/18/2005	Subsurface Sediment	30	99	cm	7629257	700289	-21	--		***0.261***		49		--		150		2.27		79.1		9.6		69.5		0.1		19.8
SE	LW2-C532	LW2-C532-C	N	10/18/2005	Subsurface Sediment	99	198	cm	7629257	700289	-21	--		***0.298***		50		--		208		2.96		92.3		14.5		77.8		0.1		7.7
SE	LW2-C532	LW2-C532-D	N	10/18/2005	Subsurface Sediment	198	293	cm	7629257	700289	-21	***380***	***JT***	--		--		--		--		2.86		--		--		--		--		--
SE	LW2-C532	LW2-C532-E	N	10/18/2005	Subsurface Sediment	293	338	cm	7629257	700289	-21	--		***0.299***		70		--		191		2.28		78.9		19.0		59.9		0.1		20.3
SE	LW2-C532	LW2-C532-F	N	10/18/2005	Subsurface Sediment	338	372	cm	7629257	700289	-21	--		0.019	JT	0.47	U	--		100		0.08		34.4		1.0		33.4		--		56.2
SE	LW2-G389	LW2-G389	N	10/29/2004	Surface Sediment	0	20	cm	7628548	700673	4	--		0.02		1.2	U	--		52.3		0.43		78.4		11.6		66.8		1.3		19.3
SE	LW2-G394	LW2-G394	N	8/27/2004	Surface Sediment	0	25	cm	7628836	700366	-25	--		***0.46***		95		--		244		2.87		62.3		13.8		48.5		0.3		37.6
SE	LW2-G399	LW2-G399	N	8/27/2004	Surface Sediment	0	22	cm	7629573	700201	-1	***38***	***JT***	0.05		31		--		105		1.32		30.4		2.8		27.6		0.3		69.6
SE	LW2-G401	LW2-G401	N	10/22/2004	Surface Sediment	0	27	cm	7629036	700154	-10	***29***	***JT***	0.07		29		--		140		2.22		62.3		5.4		56.9		2.0		32.5
SE	LW2-G403	LW2-G403	N	10/22/2004	Surface Sediment	0	26	cm	7629824	700114	-4	--		0.018		6.5		--		72.1		0.22		4.1		0.7		3.4		0.8		95.6
SE	LW2-G404	LW2-G404	N	8/27/2004	Surface Sediment	0	25	cm	7629091	700096	-9	--		0.06		1	U	--		120		2.1		75.3		7.5		67.8		1.1		26.1
SE	LW2-G407	LW2-G407	N	9/9/2004	Surface Sediment	0	29	cm	7630154	699834	-12	--		0.067		9.9	U	--		125	T	1.99		64.3		7.1		57.3		0.4		32.3
SE	LW2-G413	LW2-G413	N	10/22/2004	Surface Sediment	0	27	cm	7630351	699536	2	--		0.029		3.1	U	--		142		0.42		4.5		0.8		3.7		1.8		94.0
SE	LW2-GBT020	LW2-GBT020	N	12/6/2005	Surface Sediment	0	10	cm	7628495	701060	-13	***19***	***JT***	0.056		6.1		9.7		115		2.97		50.8		4.2		46.6		1.4		48.6
SE	LW2-GBT021	LW2-GBT021	N	12/13/2005	Surface Sediment	0	10	cm	7629274	700117	-6	***26***	***T***	0.068		8.7		9		130		1.39		41.6		6.4		35.3		0.8		57.9
SE	LW2-NA3A	NA-3A-0005	N	10/22/2004	Surface Sediment	0	5	cm	7629429	700328	-21	--		--		--		--		93.4		--		--		--		--		--		--		--
SE	LW2-NA3A	NA-3A-0508	N	10/22/2004	Subsurface Sediment	5	8	cm	7629429	700328	-21	--		--		--		--		90.6		--		--		--		--		--		--		--
SE	LW2-NA3A	NA-3A-0812	N	10/22/2004	Subsurface Sediment	8	12	cm	7629429	700328	-21	--		--		--		--		157		--		--		--		--		--		--		--
SE	LW2-NA3A	NA-3A-1216	N	10/22/2004	Subsurface Sediment	12	16	cm	7629429	700328	-21	--		--		--		--		160		--		--		--		--		--		--		--
SE	LW2-NA3A	NA-3A-1620	N	10/22/2004	Subsurface Sediment	16	20	cm	7629429	700328	-21	--		--		--		--		126		--		--		--		--		--		--		--
SE	LW2-NA3A	NA-3A-2024	N	10/22/2004	Subsurface Sediment	20	24	cm	7629429	700328	-21	--		--		--		--		86.7		--		--		--		--		--		--		--
SE	LW2-NA3A	NA-3A-2428	N	10/22/2004	Subsurface Sediment	24	28	cm	7629429	700328	-21	--		--		--		--		120		--		--		--		--		--		--		--
SE	LW2-NA3A	NA-3A-2832	N	10/22/2004	Subsurface Sediment	28	32	cm	7629429	700328	-21	--		--		--		--		72.2		--		--		--		--		--		--		--
SE	LW2-NA3A	NA-3A-3238	N	10/22/2004	Subsurface Sediment	32	38	cm	7629429	700328	-21	--		--		--		--		83	T	--		--		--		--		--		--		--
SE	LW2-NA3A	NA-3A-3844	N	10/22/2004	Subsurface Sediment	38	44	cm	7629429	700328	-21	--		--		--		--		101		--		--		--		--		--		--		--
SE	LW2-NA3A	NA-3A-4854	N	10/22/2004	Subsurface Sediment	48	54	cm	7629429	700328	-21	--		--		--		--		115		--		--		--		--		--		--		--
SE	LW2-NA3A	NA-3A-5864	N	10/22/2004	Subsurface Sediment	58	64	cm	7629429	700328	-21	--		--		--		--		131		--		--		--		--		--		--		--
SE	LW2-NA3A	NA-3A-6874	N	10/22/2004	Subsurface Sediment	68	74	cm	7629429	700328	-21	--		--		--		--		149		--		--		--		--		--		--		--
SE	LW2-NA3A	NA-3A-7884	N	10/22/2004	Subsurface Sediment	78	84	cm	7629429	700328	-21	--		--		--		--		329		--		--		--		--		--		--		--
SE	LW2-NA3A	NA-3A-8894	N	10/22/2004	Subsurface Sediment	88	94	cm	7629429	700328	-21	--		--		--		--		144		--		--		--		--		--		--		--
SE	LW2-NA3B	NA-3B-0034	N	10/22/2004	Surface Sediment	0	34	cm	7629433	700322	-20	--		--		82	J	--		--		1.87		72.8		5.2		67.6		0.4		20.7
SE	LW2-NA3B	NA-3B-3464	N	10/22/2004	Subsurface Sediment	34	64	cm	7629433	700322	-20	--		--		63	J	--		--		0.94		40.8		3.2		37.6		0.4		58.6
SE	LW2-NA3B	NA-3B-6494	N	10/22/2004	Subsurface Sediment	64	94	cm	7629433	700322	-20	--		--		110	J	--		--		2.01		60.0		5.5		54.6		0.7		38.5
SE	LW3-C688	LW3-C688-B	N	1/15/2008	Subsurface Sediment	30	114	cm	7628760	701367	-5	***13***	***JT***	0.057		5.1	U	--		88.2		2.14		79.1		19.1		60.1		1.1		16.2
SE	LW3-C688	LW3-C688-C	N	1/15/2008	Subsurface Sediment	114	221	cm	7628760	701367	-5	***71***	***JT***	***0.107***		10		--		155		2.01		89.4		27.0		62.4		1.0		14.6
SE	LW3-C688	LW3-C688-D	N	1/15/2008	Subsurface Sediment	221	281	cm	7628760	701367	-5	***230***	***JT***	0.085		16		--		152		1.54		57.5		15.6		41.8		1.9		45.5
SE	LW3-C688	LW3-C688-F	N	1/15/2008	Subsurface Sediment	404	457	cm	7628760	701367	-5	***54***	***JT***	***0.286***		95		--		154		2.08		65.3		18.1		47.2		5.7		27.6
SE	LW3-C690	LW3-C690-B	N	1/15/2008	Subsurface Sediment	30	151	cm	7628470	701000	-11	***17***	***T***	0.052		7		--		88		1.82		77.8		9.0		68.8		1.0		19.1
SE	LW3-C690	LW3-C690-C	N	1/15/2008	Subsurface Sediment	151	274	cm	7628470	701000	-11	***48***	***T***	***0.09***		24		--		136		2.24		88.8		4.8		84.0		0.7		13.6
SE	LW3-C690	LW3-C690-D	N	1/15/2008	Subsurface Sediment	274	346	cm	7628470	701000	-11	***220***	***T***	***0.158***		33		--		228		2.12		82.1		10.6		71.5		1.7		17.2
SE	LW3-C690	LW3-C690-E	N	1/15/2008	Subsurface Sediment	346	471	cm	7628470	701000	-11	***450***	***JT***	***0.349***		87		--		222		2.49		62.5		10.8		51.7		5.4		26.8
SE	LW3-C695	LW3-C695-B	N	12/20/2007	Subsurface Sediment	30	95	cm	7629190	700056	-6	--		***0.089***		50		--		122	J	1.37		55.9		31.8		24.1		0.9		56.2
SE	LW3-C695	LW3-C695-D	N	12/20/2007	Subsurface Sediment	221	343	cm	7629190	700056	-6	--		***0.172***		39		--		175	J	1.93		77.1		11.8		65.3		1.1		35.4
SE	LW3-C695	LW3-C695-E	N	12/20/2007	Subsurface Sediment	343	384	cm	7629190	700056	-6	--		***0.209***		67		--		167	J	2.18		82.4		13.6		68.8		0.4		18.1
SE	LW3-C698	LW3-C698-B	N	12/20/2007	Subsurface Sediment	30	152	cm	7630300	699718	-16	--		***0.102***		16		--		116	J	1.94		85.8		13.4		72.4		0.8		18.9
SE	LW3-G692-1	LW3-G692-1	N	12/4/2007	Surface Sediment	0	30	cm	7628730	701055	-20	***26***	***JT***	0.071		20		--		111		2.24		84.8		13.8		71.0		1.9		13.6
SE	LW3-G692-2	LW3-G692-2	FR	12/4/2007	Surface Sediment	0	28	cm	7628730	701048	-20	***21***	***JT***	***0.086***	***T***	12	T	--		110	T	2.25		79.9		10.3		69.5		1.2		15.3
SE	LW3-G694	LW3-G694	N	11/16/2007	Surface Sediment	0	20	cm	7629340	700318	-23	--		***0.161***		85		--		122		1.69		70.6		6.7		63.9		0.5		32.0
SE	LW3-GSP07W	LW3-GSP07W	N	10/15/2007	Surface Sediment	0	19	cm	7628812	700370	-14	2	JT	0.038		2.6	U	0.086	U	65.9		0.83		68.4		15.6		52.8		5.3		15.9

Table A-1. RI/FS Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

CAS												LWG RA Total PCB Congener (Calculated U = 1/2)		Mercury		Naphthalene		Tributyltin ion		Zinc		Total Organic Carbon		Fines (silt + clay)		Total Clay		Total Silt		Total Gravel		Total Sand
Matrix	Location ID	Sample Name	Sample Type	Sample Date	Depth Class	Start Depth	End Depth	Depth Units	X Coordinate	Y coordinate	Elevation (NAVD88)	LRAtPCBCong_N		7439-97-6		91-20-3		36643-28-4		7440-66-6		TOC		GS-FINES		T-Clay		T-Silt		T-Gravel		T-Sand
												µg/kg		mg/kg		µg/kg		µg/kg		mg/kg		%		%		%		%		%		%
											Sediment/Bank Soil CUL	**9**		**0.085**		**NA**		**3,080**		**459**												
											Sitewide RAL	**75**		--		**NA**		--		--												
											PTW Threshold	**200**		--		**140,000**		--		--												
SE	LWG0108R002SDS015C00	LWG0108R002SDS015C00	N	10/17/2002	Surface Sediment	0	15	cm	7630259	699666	1	--		0.05	U	19	U	--		108		1.4		16.9		2.5		14.4		2.5		80.7
SE	TOSC099DMMU1	TOSC099DMMU1SDO1	N	1/21/1999	Subsurface Sediment	0	304	cm	7629016	700391	-24	--		0.2	U	30		--		164		2.16		85.1		68.2		17.0		0.1		14.9
SE	TOSC099DMMU1	TOSC099DMMU1SDO3	FR	1/21/1999	Subsurface Sediment	0	304	cm	7629016	700391	-24	--		0.2	U	34		--		177		2.25		84.0		65.9		18.1		0.1		15.9
SE	TOSC099DMMU2	TOSC099DMMU2SDO4	N	1/21/1999	Subsurface Sediment	0	164	cm	7629278	700566	-29	--		***0.2***		22		--		144		1.97		82.4		61.5		20.9		0.2		17.4
SE	W-09-A-PG	LWG2-PG-W9A	N	11/30/2005	Surface Sediment	0	23	cm	7628899	700250	3	--		0.03		0.51	U	--		68.7		0.4		79.0		14.2		64.8		0.8		20.7
SE	W-09-C-PG	LWG2-PG-W9C	N	11/30/2005	Surface Sediment	0	30	cm	7628988	700306	-20	--		***0.087***		10		--		133		2.32		76.0		10.7		65.4		0.6		21.9
SE	WLCASF97S001	WLCASF97S001W4159	N	6/12/1997	Surface Sediment	0	10	cm	7630379	699598	-9	--		--		17.5	T	--		--		1.8		84.4		11.7		72.7		--		15.6
SE	WLCASF97S002	WLCASF97S002W4158	N	6/12/1997	Surface Sediment	0	10	cm	7630047	700105	-32	--		--		13		--		--		2.3		87.3		13.2		74.1		0.1		12.6
SE	WLCASF97S003	WLCASF97S003W4157	N	6/12/1997	Surface Sediment	0	10	cm	7629493	700297	-17	--		--		36		--		--		2		87.4		16.3		71.1		0.1		12.5
SE	WLCASF97S004	WLCASF97S004W4156	N	6/12/1997	Surface Sediment	0	10	cm	7629322	700277	-23	--		--		127		--		--		1.9		65.7		7.3		58.4		0.1		34.2
SE	WLCASF97S005	WLCASF97S005W4155	N	6/12/1997	Surface Sediment	0	10	cm	7629019	700128	-5	--		--		74		--		--		1.8		36.6		4.4		32.2		10.7		52.7
SE	WLCASF97S006	WLCASF97S006W4154	N	6/12/1997	Surface Sediment	0	10	cm	7628791	700365	-2	--		--		21		--		--		1.9		76.3		10.1		66.2		1.5		22.2
SE	WLCASF97S008	WLCASF97S008W4152	N	6/12/1997	Surface Sediment	0	10	cm	7629266	700704	-35	--		--		28		--		--		2.1		65.0		14.4		50.6		--		35.0
SE	WLCASF97S009	WLCASF97S009W4172	N	6/11/1997	Surface Sediment	0	10	cm	7628648	700612	-32	--		--		17		--		--		2.3		86.8		16.4		70.4		0.1		13.1
SE	WLCASF97S010	WLCASF97S010W4171	N	6/11/1997	Surface Sediment	0	10	cm	7629081	700977	-34	--		--		32		--		--		2.4		87.8		18.4		69.4		--		12.2
SE	WLCASF97S011	WLCASF97S011W4170	N	6/11/1997	Surface Sediment	0	10	cm	7628514	700853	-19	--		--		15		--		--		2.3		88.5		16.8		71.7		0.1		11.4
SE	WLCASF97S012	WLCASF97S012W4168	N	6/11/1997	Surface Sediment	0	10	cm	7628858	701245	-20	--		--		15		--		--		2		85.7		13.2		72.5		0.1		14.2
SE	WLCASF97S012	WLCASF97S012W4169	FR	6/11/1997	Surface Sediment	0	10	cm	7628858	701245	-20	--		--		20		--		--		2.6		91.2		18.7		72.5		0.1		8.7
SE	WLCCWI08ANCTPD03	WLCCWI08ANCTPD03	N	5/29/2009	Surface Sediment	0	2	cm	7629152	700952	-39	--		--		--		--		--		2.96		--		--		--		--		--
SE	WLCCWI08ANCTPD06	WLCCWI08ANCTPD06	N	5/29/2009	Surface Sediment	0	2	cm	7629255	700767	-38	--		--		--		--		--		2.5		--		--		--		--		--		--
SE	WLCCWI08DMMU1A	WLCCWI08DMMU1A	N	5/29/2009	Surface Sediment	0	2	cm	7628747	700605	-35	--		--		--		--		--		2.53		--		--		--		--		--		--
SE	WLCCWI08DMMU1A	WLCCWI08DMMU1A4041	N	9/2/2008	Subsurface Sediment	91	121	cm	7628747	700605	-35	--		***0.141***	***J***	9.56		2.7	UJ	119	J	--		--		--		--		--		--		--
SE	WLCCWI08DMMU1A	WLCCWI08DMMU1A4142	N	9/2/2008	Subsurface Sediment	121	152	cm	7628747	700605	-35	--		--		24.6	UJ	2.2	UJ	115	J	2.7		--		--		--		--		--		--
SE	WLCCWI08DMMU1ABC	WLCCWI08DMMU1ABC	N	9/4/2008	Subsurface Sediment	91	182	cm	7628839	700680	-33	--		0.0669	JT	9.47	T	3	UJT	47.7	JT	--		--		--		--		--		--		--
SE	WLCCWI08DMMU1B	WLCCWI08DMMU1B	N	5/29/2009	Surface Sediment	0	2	cm	7628834	700677	-33	--		--		--		--		--		2.41		--		--		--		--		--		--
SE	WLCCWI08DMMU1B	WLCCWI08DMMU1B4041	N	9/3/2008	Subsurface Sediment	121	152	cm	7628834	700677	-33	--		***0.101***	***J***	8.87		2.6	UJ	98.6	J	--		--		--		--		--		--		--
SE	WLCCWI08DMMU1B	WLCCWI08DMMU1B4142	N	9/3/2008	Subsurface Sediment	152	182	cm	7628834	700677	-33	--		--		23.3	UJ	2.3	UJ	110	J	2.5		--		--		--		--		--		--
SE	WLCCWI08DMMU1C	WLCCWI08DMMU1C	N	5/29/2009	Surface Sediment	0	5	cm	7628936	700759	-33	--		--		--		--		--		2.45		--		--		--		--		--		--
SE	WLCCWI08DMMU1C	WLCCWI08DMMU1C4041	N	9/4/2008	Subsurface Sediment	91	121	cm	7628936	700759	-33	--		***0.105***	***J***	7.75		2.6	UJ	106	J	--		--		--		--		--		--		--
SE	WLCCWI08DMMU1C	WLCCWI08DMMU1C4142	N	9/4/2008	Subsurface Sediment	121	152	cm	7628936	700759	-33	--		--		23.4	UJ	2.5	UJ	105	J	2.5		--		--		--		--		--		--
SE	WLCCWI08DMMU2A	WLCCWI08DMMU2A	N	5/29/2009	Surface Sediment	0	2	cm	7628879	700461	-33	--		--		--		--		--		2.47		--		--		--		--		--		--
SE	WLCCWI08DMMU2A	WLCCWI08DMMU2A4041	N	9/5/2008	Subsurface Sediment	152	182	cm	7628879	700461	-33	--		***0.112***	***J***	9.19	J	2.5	U	107	J	--		--		--		--		--		--		--
SE	WLCCWI08DMMU2A	WLCCWI08DMMU2A4142	N	9/5/2008	Subsurface Sediment	182	213	cm	7628879	700461	-33	--		0.173	U	24.9	U	2.3	U	125		2.5		--		--		--		--		--		--
SE	WLCCWI08DMMU2B	WLCCWI08DMMU2B	N	5/29/2009	Surface Sediment	0	2	cm	7628979	700542	-33	--		--		--		--		--		2.72		--		--		--		--		--		--
SE	WLCCWI08DMMU2B	WLCCWI08DMMU2B4041	N	9/5/2008	Subsurface Sediment	91	121	cm	7628979	700542	-33	--		***0.0951***	***J***	10.1	J	2.4	U	112	J	--		--		--		--		--		--		--
SE	WLCCWI08DMMU2B	WLCCWI08DMMU2B4142	N	9/5/2008	Subsurface Sediment	121	152	cm	7628979	700542	-33	--		--		23.6	UJ	2.3	UJ	143	J	2.5		--		--		--		--		--		--
SE	WLCCWI08DMMU2C	WLCCWI08DMMU2C	N	5/29/2009	Surface Sediment	0	2	cm	7629098	700580	-33	--		--		--		--		--		2.16		--		--		--		--		--		--
SE	WLCCWI08DMMU2C	WLCCWI08DMMU2C4041	N	9/5/2008	Subsurface Sediment	60	91	cm	7629098	700580	-33	--		***0.126***	***J***	27.8	J	3	U	114	J	--		--		--		--		--		--		--
SE	WLCCWI08DMMU2C	WLCCWI08DMMU2C4142	N	9/5/2008	Subsurface Sediment	91	121	cm	7629098	700580	-33	--		--		23.3	UJ	2.3	UJ	118	J	2.5		--		--		--		--		--		--
SE	WLCCWI08DMMU3A	WLCCWI08DMMU3A	N	5/29/2009	Surface Sediment	0	2	cm	7628650	700651	-24	--		--		--		--		--		2.76		--		--		--		--		--		--
SE	WLCCWI08DMMU3A	WLCCWI08DMMU3A13	N	9/8/2008	Subsurface Sediment	30	91	cm	7628650	700651	-24	--		***0.191***	***J***	25.2	UJ	2.4	UJ	136	J	2.5		--		--		--		--		--		--
SE	WLCCWI08DMMU3B	WLCCWI08DMMU3B	N	5/29/2009	Surface Sediment	0	2	cm	7628779	700408	-18	--		--		--		--		--		1.95		--		--		--		--		--		--
SE	WLCCWI08DMMU3B	WLCCWI08DMMU3B13	N	9/8/2008	Subsurface Sediment	30	91	cm	7628779	700408	-18	--		***0.15***		165	U	1.8	UJ	90.2		0.96		--		--		--		--		--		--
SE	WLCCWI08DMMU3D	WLCCWI08DMMU3D	N	5/29/2009	Surface Sediment	0	2	cm	7628873	700325	-13	--		--		--		--		--		0.77		--		--		--		--		--		--
SE	WLCCWI08DMMU3D	WLCCWI08DMMU3D13	N	9/8/2008	Subsurface Sediment	30	91	cm	7628873	700325	-13	--		***0.102***	***J***	393	UJ	1.5	UJ	85.6	J	0.57		--		--		--		--		--		--
SE	WLCCWI08DMMU3E	WLCCWI08DMMU3E	N	5/29/2009	Surface Sediment	0	2	cm	7628981	700402	-24	--		--		--		--		--		2.48		--		--		--		--		--		--
SE	WLCCWI08DMMU3E	WLCCWI08DMMU3E13	N	9/5/2008	Subsurface Sediment	30	91	cm	7628981	700402	-24	--		0.14	U	23.4	U	2.3	U	101		2.2		--		--		--		--		--		--
SE	WLCDRD05PG074	WLCDRD05PG07474	N	5/25/2005	Surface Sediment	0	30	cm	7628952	701290	-24	--		0.067		0.9	U	--		87.1	J	2.9		88.7		14.0		74.7		0.0		9.2
SE	WLCOFJ022201	WLCOFJ02220122010	N	10/21/2002	Surface Sediment	0	10	cm	7629006	700111	-2	--		0.0184	J	16.4	U	--		102	J	0.0576		--		--		--		--		--		--
SE	WLCOFJ022202	WLCOFJ02220222020	N	10/21/2002	Surface Sediment	0	15	cm	7629048	700077	-3	--		0.0457		26.8	J	--		78.4	J	1.54		--		--		--		--		--		--
SE	WLCOFJ022203	WLCOFJ02220322030	N	10/21/2002	Surface Sediment	0	15	cm	7628994	700157	-4	--		***0.0949***		154		--		72.7	J	0.953		--		--		--		--		--		--
SE	WLCOFJ0222031	WLCOFJ022203122031	FR	10/21/2002	Surface Sediment	0	15	cm	7628991	700159	-4	--		0.0413		18.5	UJ	--		67.8	J	5.53		--		--		--		--		--		--
SE	WLCOFJ022204	WLCOFJ02220422040	N	10/21/2002	Surface Sediment	0	12	cm	7629090	700117	-16	--		0.0803		29.1	J	--		87.3	J	1.75		--		--		--		--		--		--
SE	WLCOFJ022205	WLCOFJ02220522050	N	10/21/2002	Surface Sediment	0	12	cm	7629070	700151	-18	--		0.0582		34.9	J	--		83	J	2.18		--		--		--		--		--		--
SE	WLCOFJ022206	WLCOFJ02220622060	N	10/21/2002	Surface Sediment	0	10	cm	7629040	700177	-19	--		0.0719		24.5	UJ	--		74.4	J	2		--		--		--		--		--		--
SE	WLCPWL09APS-1	WLCPWL09APS-1B	N	12/29/2009	Subsurface Sediment	60	121	cm	7628973	700319	-21	--		***0.148***	***J***	28	J	57	J	131		2.42		92.8		11.9		80.9		--		10.1
SE	WLCPWL09APS-1	WLCPWL09APS-1T	N	12/29/2009	Subsurface Sediment	0	60	cm	7628973	700319	-21	--		0.077	J	13	J	20	J	98.3		1.9		93.1		15.8		77.3		--		7.8
SE	WLCPWL09APS-2	WLCPWL09APS-2B	N	12/29/2009	Subsurface Sediment	60	121	cm	7629116	700296	-15	--		0.074	J	21	J	22	J	102		2.04		89.9		12.8		77.1		--		11.9
SE	WLCPWL09APS-2	WLCPWL09APS-2T	N	12/29/2009	Subsurface Sediment	0	60	cm	7629116	700296	-15	--		***0.086***	***J***	21	J	14	J	109		2.34		93.4		11.5		81.9		--		7.9
SE	WLCPWL09APS-3	WLCPWL09APS-3B	N	12/29/2009	Subsurface Sediment	60	121	cm	7629238	700264	-20	--		***0.226***	***J***	110	J	1.8	UJ	134		2.18		85.3		8.4		76.9		--		17.3
SE	WLCPWL09APS-3	WLCPWL09APS-3T	N	12/29/2009	Subsurface Sediment	0	60	cm	7629238	700264	-20	--		***0.195***	***J***	120	J	1.8	UJ	134		2.16		87.8		7.2		80.6		--		18.3
SE	WLCPWL09DRB-1	WLCPWL09DRB-1Z	N	12/29/2009	Subsurface Sediment	320	350	cm	7629031	700383	-22	--		***0.287***	***J***	35	J	37	J	154		2.15		93.7		18.0		75.7		--		6.9
SE	WLCPWL09DRB-2	WLCPWL09DRB-2Z	N	12/29/2009	Subsurface Sediment	307	338	cm	7629105	700447	-25	--		***0.122***	***J***	41	J	58	J	156		2.05		94.5		18.0		76.5		--		2.6
SE	WLCPWL09DRB-3	WLCPWL09DRB-3Z	N	12/29/2009	Subsurface Sediment	280	310	cm	7629193	700495	-25	--		***0.121***	***J***	50	J	55	J	143		2.21		99.2		15.8		83.4		--		6.3
SE	WLCPWL09DRB-D	WLCPWL09DRB-DP	N	12/29/2009	Subsurface Sediment	0	302	cm	7629110	700442	-24	--		***0.094***	***J***	40	J	51	J	131		2.29		88.7		0.9		87.8		--		6.3
SE	WLCPWL09URB-1	WLCPWL09URB-1Z	N	12/29/2009	Subsurface Sediment	295	326	cm	7629179	700364	-17	--		***0.319***	***J***	110	J	4	J	168		2.7		96.0		14.6		81.4		--		4.4
SE	WLCPWL09URB-2	WLCPWL09URB-2Z	N	12/29/2009	Subsurface Sediment	118	149	cm	7629295	700478	-23	--		***0.72***	***J***	95	J	1.8	UJ	208		2.72		98.6		24.2		74.4		--		5.0
SE	WLCPWL09URB-3	WLCPWL09URB-3Z	N	12/29/2009	Subsurface Sediment	167	198	cm	7629268	700417	-21	--		***0.297***	***J***	69	J	1.8	UJ	165		2.75		97.4		16.1		81.3		--		2.7
SE	WLCPWL09URB-D	WLCPWL09URB-DP	N	12/29/2009	Subsurface Sediment	0	194	cm	7629248	700420	-20	--		***0.161***	***JT***	40	JT	43.5	JT	139	T	2.22		96.3		7.4		88.9		--		6.9

Table A-1. RI/FS Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

CAS			Sample	Sample		Start	End	Depth			Elevation	LWG RA Total PCB Congener (Calculated U = 1/2)	Mercury		Naphthalene		Tributyltin ion		Zinc		Total Organic Carbon		Fines (silt + clay)		Total Clay		Total Silt		Total Gravel		Total Sand
Matrix	Location ID	Sample Name	Type	Date	Depth Class	Depth	Depth	Units	X Coordinate	Y coordinate	(NAVD88)	LRAtPCBCong_N	7439-97-6		91-20-3		36643-28-4		7440-66-6		TOC		GS-FINES		T-Clay		T-Silt		T-Gravel		T-Sand
												µg/kg	mg/kg		µg/kg		µg/kg		mg/kg		%		%		%		%		%		%
											Sediment/Bank Soil CUL	**9**	**0.085**		**NA**		**3,080**		**459**												
											Sitewide RAL	**75**	--		**NA**		--		--												
											PTW Threshold	**200**	--		**140,000**		--		--												
SE	WLCWTG02ANCTPD01	WLCWTG02ANCTPD01A	N	7/16/2002	Surface Sediment	0	2	cm	7628756	700622	-35	--	***0.09***		19		73		133		2.48		86.1		15.4		70.7		0.0		11.9
SE	WLCWTG02ANCTPD01	WLCWTG02ANCTPD01B	N	7/16/2002	Surface Sediment	0	15	cm	7628756	700622	-35	--	***0.09***		25		47		118		2.29		71.6		13.0		58.6		0.0		22.9
SE	WLCWTG02ANCTPD02	WLCWTG02ANCTPD02A	N	7/16/2002	Surface Sediment	0	2	cm	7628958	700792	-34	--	0.08		16		33		104		2.26		83.0		14.1		68.9		0.0		16.4
SE	WLCWTG02ANCTPD02	WLCWTG02ANCTPD02B	N	7/16/2002	Surface Sediment	0	15	cm	7628958	700792	-34	--	***0.09***		30		15		100		2.2		79.9		11.9		68.0		0.0		19.4
SE	WLCWTG02ANCTPD03	WLCWTG02ANCTPD03A	N	7/16/2002	Surface Sediment	0	2	cm	7629152	700954	-39	--	0.08		15		8.1		106		2.74		79.3		9.8		69.5		0.9		15.3
SE	WLCWTG02ANCTPD03	WLCWTG02ANCTPD03B	N	7/16/2002	Surface Sediment	0	15	cm	7629152	700954	-39	--	***0.1***		18		1.8		131		1.38		70.9		16.1		54.8		3.9		21.2
SE	WLCWTG02ANCTPD05	WLCWTG02ANCTPD09R	FR	7/16/2002	Surface Sediment	0	15	cm	7629438	700917	-40	--	0.06		6.4		2.3	U	89.6		2.65		88.9		14.2		74.7		0.1		14.4
SE	WLCWTG02ANCTPD06	WLCWTG02ANCTPD06A	N	7/16/2002	Surface Sediment	0	2	cm	7629255	700769	-38	--	0.08		15		56		116		2.61		83.8		15.3		68.5		0.6		18.0
SE	WLCWTG02ANCTPD06	WLCWTG02ANCTPD06B	N	7/16/2002	Surface Sediment	0	15	cm	7629255	700769	-38	--	***0.1***	***T***	27	T	83	T	141	T	2.47		84.2		19.0		65.3		0.0		18.2
SE	WLCWTG02ANCTPD07	WLCWTG02ANCTPD07A	N	7/16/2002	Surface Sediment	0	2	cm	7629084	700617	-34	--	0.07		19		29		111		2.54		88.1		14.3		73.8		0.2		14.5
SE	WLCWTG02ANCTPD07	WLCWTG02ANCTPD07B	N	7/16/2002	Surface Sediment	0	15	cm	7629084	700617	-34	--	***0.11***		7		4.5		87.8		2.64		93.6		16.8		76.8		0.0		10.1
SE	WLCWTG02ANCTPD08	WLCWTG02ANCTPD08A	N	7/16/2002	Surface Sediment	0	2	cm	7628892	700455	-33	--	0.07		20		11		121		2.88		92.1		10.9		81.2		0.0		9.9
SE	WLCWTG02ANCTPD08	WLCWTG02ANCTPD08B	N	7/16/2002	Surface Sediment	0	15	cm	7628892	700455	-33	--	***0.09***		13		19		121		2.81		88.5		12.6		75.9		0.0		13.4
SE	WLCWTI00N20	WLCWTI00N20N20	N	9/22/2000	Subsurface Sediment	15	30	cm	7628972	700109	9	--	--		--		--		--		--		--		--		--		--		--
SE	WLCWTI00N40	WLCWTI00N40N40	N	9/22/2000	Subsurface Sediment	15	30	cm	7628959	700126	8	--	--		--		--		--		--		--		--		--		--		--		--
SE	WLCWTI00S20	WLCWTI00S20S20	N	9/22/2000	Subsurface Sediment	15	30	cm	7628998	700078	8	--	--		--		--		--		--		--		--		--		--		--		--
SE	WLCWTI00S40	WLCWTI00S40S40	N	9/22/2000	Subsurface Sediment	15	30	cm	7629011	700062	9	--	--		--		--		--		--		--		--		--		--		--		--
SE	WLCWTI00TP11	WLCWTI00TP11TP11	N	9/21/2000	Subsurface Sediment	122	137	cm	7628955	700089	13	--	--		--		--		--		--		--		--		--		--		--		--
SE	WLRWTF98ATXSD3	WLRWTF98ATXSD3ATXSD3	N	12/18/1998	Surface Sediment	0	12	cm	7628416	701026	-8	--	--		--		--		--		0.0438		--		--		--		--		--		--
SE	WLRWTF98ATXSD4	WLRWTF98ATXSD4ATXSD4	N	12/18/1998	Surface Sediment	0	12	cm	7628460	700769	5	--	--		6.7	U	--		--		0.555		--		--		--		--		--		--
SE	WLRWTF98ATXSD5	WLRWTF98ATXSD5ATXSD5	N	12/18/1998	Surface Sediment	0	12	cm	7628584	700620	5	--	0.05	U	7	UJ	--		46	J	0.906		--		--		--		--		--		--
SE	WLRWTF98HEVSD1	WLRWTF98HEVSD1HEVSD1	N	12/17/1998	Surface Sediment	0	12	cm	7628832	700321	1	--	0.05	U	7	UJ	--		73.7	J	0.305		--		--		--		--		--		--
SE	WLRWTF98HEVSD2	WLRWTF98HEVSD2HEVSD2	N	12/17/1998	Surface Sediment	0	12	cm	7628783	700396	-15	--	0.05	U	7	UJ	--		40.2	J	0.324		--		--		--		--		--		--
SE	WLRWTF98HEVSD3	WLRWTF98HEVSD3HEVSD3	N	12/17/1998	Surface Sediment	0	12	cm	7628708	700471	0	--	0.05	U	7	UJ	--		61.7	J	0.954		--		--		--		--		--		--
SE	WLRWTF98TOSSD1	WLRWTF98TOSSD1TOSSD1	N	12/17/1998	Surface Sediment	0	12	cm	7628882	700247	6	--	0.05	U	7	UJ	--		70.3	J	0.378		--		--		--		--		--		--
SE	WLRWTF98TOSSD2	WLRWTF98TOSSD2TOSSD2	N	12/17/1998	Surface Sediment	0	12	cm	7628955	700135	7	--	--		13.4	U	--		--		0.0758		--		--		--		--		--		--
SE	WLRWTF98TOSSD3	WLRWTF98TOSSD3TOSSD3	N	12/17/1998	Surface Sediment	0	12	cm	7629084	700168	-12	--	0.12	U	168	UJ	--		17.3	J	1.51		--		--		--		--		--		--
SE	WLRWTF98TOSSD4	WLRWTF98TOSSD4TOSSD4	N	12/17/1998	Surface Sediment	0	12	cm	7629054	699987	13	--	--		6.7	U	--		--		0.236		--		--		--		--		--		--
SE	WLRWTF98TOSSD5	WLRWTF98TOSSD5TOSSD5	N	12/18/1998	Surface Sediment	0	12	cm	7629232	699945	9	--	0.05	U	7	UJ	--		64	J	0.0554		--		--		--		--		--		--
SE	WR-WSI98SD104	WR-WSI98SD1040	N	9/23/1997	Surface Sediment	0	10	cm	7628572	701218	-1	--	0.07		19	U	--		69.5		1.3		89.6		19.3		70.2		0.2		10.3
SE	WR-WSI98SD105	WR-WSI98SD1050	N	9/23/1997	Surface Sediment	0	10	cm	7628878	701254	-20	--	0.06		19	U	--		97.1		1.5		89.1		10.9		78.2		0.2		10.7
SE	WR-WSI98SD107	WR-WSI98SD1070	N	9/23/1997	Surface Sediment	0	10	cm	7628533	700849	-19	--	0.07		21		--		108		1.6		91.6		12.4		79.2		0.1		8.4
SE	WR-WSI98SD110	WR-WSI98SD1100	N	9/23/1997	Surface Sediment	0	10	cm	7629093	700983	-35	--	0.07		34		--		108		1.7		75.7		12.1		63.6		0.2		24.2
SE	WR-WSI98SD113	WR-WSI98SD1130	N	9/23/1997	Surface Sediment	0	10	cm	7629298	700704	-35	--	0.07		21		--		105		1.7		86.3		11.4		74.9		0.2		13.5
SE	WR-WSI98SD114	WR-WSI98SD1140	N	9/23/1997	Surface Sediment	0	10	cm	7629023	700135	-8	--	0.02		20	U	--		212		0.72		6.2		0.9		5.3		5.1		88.7
SE	WR-WSI98SD115	WR-WSI98SD1150	N	9/23/1997	Surface Sediment	0	10	cm	7629434	700515	-24	--	0.06		20	U	--		101		1.5		84.4		10.1		74.7		0.0		15.2
SE	WR-WSI98SD117	WR-WSI98SD1170000A	N	10/16/1997	Subsurface Sediment	0	90	cm	7629340	700260	-22	--	***0.17***		110		6	U	157		1.3		67.6		8.4		59.2		0.1		32.3
SE	WR-WSI98SD117	WR-WSI98SD1170000CC	N	9/23/1997	Surface Sediment	0	10	cm	7629340	700260	-22	--	***0.1***	***T***	29.5	T	5.7	UT	99.2	T	0.765		41.4		3.4		38.2		0.4		58.2
SE	WR-WSI98SD118	WR-WSI98SD1180	N	9/23/1997	Surface Sediment	0	10	cm	7629521	700296	-16	--	0.06		20	U	--		89.2		1.5		28.4		3.1		25.3		0.3		71.3
SE	WR-WSI98SD123	WR-WSI98SD1230	N	9/24/1997	Surface Sediment	0	10	cm	7630398	699583	-8	--	0.06		19	U	--		97.2		1.5		76.0		10.5		65.5		--		24.0

Table A-1. RI/FS Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Notes:

µg/kg = microgram(s) per kilogram

mg/kg = milligram(s) per kilogram

cm = centimeters

ft = feet

A = Summed value based on a limited number of analytes

N = normal/primary sample

NAVD88 = North American Vertical Datum of 1988

J = estimated result

FR = field replicate

T = Result calculated or selected from >1 reported value

U = non detect result

Bold Italic = detected result exceeds Sediment/Bank Soil CUL

Yellow = detected result exceeds applicable RAL (Sitewide)

Underline = detected result exceeds PTW Threshold

Table A-2. Pre-RD Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Matrix	Sys_Loc_Code	Sample Name	Sample Date	Sample Type	Depth Class	Start Depth	End Depth	Depth Unit	X Coordinate	Y Coordinate	Surface Elevation (ft NAVD 88)	1,2,3,4,7,8-HxCDF µg/kg		1,2,3,7,8-PeCDD µg/kg		2,3,4,7,8-PeCDF µg/kg		2,3,7,8-TCDD µg/kg		2,3,7,8-TCDF µg/kg		Aldrin µg/kg		Arsenic mg/kg		Bis(2-ethylhexyl)phthalate µg/kg		Cadmium mg/kg		Copper mg/kg		DDx – 6 Isomers µg/kg		Dieldrin µg/kg	
											Sediment/Bank Soil CUL	0.0004		0.0002		0.0003		0.0002		0.0004		2		3		135		0.51		359		6.1		0.07	
											Sitewide RAL	NA		0.0008		0.2		0.0006		NA		--		--		--		--		--		160		--	
											PTW Threshold	0.04		0.01		0.2		0.01		0.6		--		--		--		--		--		7,050		--	
SE	B236	PDI-SG-B236-BL1	20-Apr-18	N	Surface Sediment	0	30	cm	7628693.5	701205.1	-11.7	***0.0057***	J	***0.00036***	JN	***0.0012***	J	0.00018	JN	***0.0024***		0.59	U	***4.7***		91	J	0.16	J	36		***6.17***		1.2	U
SE	B237	PDI-SG-B237-BL1	20-Apr-18	N	Surface Sediment	0	30	cm	7628595.4	700932.5	-19.6	***0.0042***	J	***0.00035***	JN	***0.001***	J	0.00015	JN	***0.0025***		0.57	UJ	***4.3***		92	J	0.18	J	33		***9.85***		1.1	UJ
SE	B239	PDI-SG-B239-BL1	21-Apr-18	N	Surface Sediment	0	30	cm	7628724.1	700674.0	-32.8	***0.029***		***0.00053***	J	***0.07***		***0.00035***	JN	***0.16***		0.61	U	***5.1***		100	J	0.24	J	41		***8.57***		1.2	U
SE	B241	PDI-SG-B241-BL1	21-Apr-18	N	Surface Sediment	0	30	cm	7629093.9	700308.8	-14.0	***0.0034***	J	***0.0006***	JN	***0.00087***	J	***0.00031***	JN	***0.0021***		0.58	U	***5.3***		63	J	0.19	J	42		***6.74***		1.2	U
SE	B245	PDI-SG-B245-BL1	21-Apr-18	N	Surface Sediment	0	22	cm	7629622.9	700261.9	-6.1	***0.00099***	J	0.00012	J	0.00019	JN	0.000059	U	0.00033	J	0.4	U	***4.6***		15	J	0.098	J	16		2.18		1	U
SE	B246	PDI-SG-B246-BL1	22-May-18	N	Surface Sediment	0	30	cm	7629841.9	700111.0	-3.3	***0.0016***	JN	***0.0004***	J+	***0.00052***	JN	0.00015	U	***0.0009***		0.92	U	***6.7***		86	J	0.16	J	27		4.22		2	U
SE	B251	PDI-SG-B251-BL1	22-May-18	N	Surface Sediment	0	30	cm	7630185.1	699860.8	-18.5	***0.0028***	J	***0.00049***	J+	***0.00064***	J	0.00023	U	***0.0015***		1.2	U	***5.7***		***140***		0.28	J	48		***6.63***		2.5	U
SE	B253	PDI-SG-B253-BL1	23-May-18	N	Surface Sediment	0	24	cm	7630335.5	699548.4	3.5	***0.00079***	J+	***0.00024***	J+	0.0003	J+	0.000056	U	0.00063	U	0.79	U	***3.9***		75		0.27		25		0.94	U	2	U
SE	B257	PDI-SG-B257-BL1	22-May-18	N	Surface Sediment	0	24	cm	7630634.1	699464.6	-1.7	0.00015	J	0.000053	U	0.000074	U	0.000077	U	0.000044	U	0.79	U	1.1		59	U	0.054	J	10		0.94	U	2	U
SE	S157	PDI-SG-S157	14-May-18	N	Surface Sediment	0	30	cm	7628995.8	700977.0	-35.4	***0.015***		***0.00051***	J	***0.0061***		***0.0005***	J	***0.0097***		--		--		--		--		--		***13.3***		--	
SE	S158	PDI-SG-S158	14-May-18	N	Surface Sediment	0	25	cm	7629028.1	701044.6	-35.1	***0.0079***		***0.00056***	J	***0.0017***	J	***0.00064***	J	***0.0029***		--		--		--		--		--		***11.8***		--	
SE	S159	PDI-SG-S159	18-May-18	N	Surface Sediment	0	30	cm	7629035.3	700769.7	-33.9	***0.0064***		***0.00068***	J	***0.0029***	J	***0.00033***	JN	***0.006***		--		--		--		--		--		***25.6***		--	
SE	S160	PDI-SG-S160	18-May-18	N	Surface Sediment	0	30	cm	7628845.2	700431.9	-32.7	***0.0067***		***0.00055***	JN	***0.0023***	J	***0.00038***	JN	***0.0044***		--		--		--		--		--		***8.77***		--	
SE	S161	PDI-SG-S161	14-May-18	N	Surface Sediment	0	29	cm	7629277.9	700783.8	-38.3	***0.0026***	J	***0.00052***	J	***0.00076***	J	***0.00053***	J	***0.0014***		--		--		--		--		--		***7.56***		--	
SE	S162	PDI-SG-S162	18-May-18	N	Surface Sediment	0	30	cm	7629427.9	700572.1	-23.2	***0.0061***	J	***0.00053***	JN	***0.0023***	J	0.0002	U	***0.0041***		--		--		--		--		--		***7.05***		--	
SE	S163	PDI-SG-S163	18-May-18	N	Surface Sediment	0	30	cm	7629261.9	700355.8	-19.7	***0.017***		***0.00077***	J	***0.0032***	J	***0.00052***	J	***0.0042***		--		--		--		--		--		***8.61***		--	
SE	S164	PDI-SG-S164	20-Jun-18	N	Surface Sediment	0	27	cm	7629972.7	700029.5	-9.5	***0.0024***	J	***0.00069***	J	***0.00074***	J	***0.00037***	JN	***0.0012***		--		--		--		--		--		5.22		--	
SE	S165	PDI-SG-S165	16-May-18	N	Surface Sediment	0	27	cm	7630314.5	699724.3	-16.0	***0.0069***		0.00029	U	***0.0013***	J	***0.00057***	JN	***0.0012***	J	--		--		--		--		--		5.90		--	
SE	SC-S155	PDI-SC-S155-0to2.1	05-Sep-18	N	Subsurface Sediment	0	2.1	ft	7628606.6	701255.1	-1.3	***0.0047***		***0.00038***	J	***0.0012***	J	0.00018	JN	***0.003***		--		--		--		--		--		***18.4***		--	
SE	SC-S155	PDI-SC-S155-2.1to4.2	05-Sep-18	N	Subsurface Sediment	2.1	4.2	ft	7628606.6	701255.1	-1.3	***0.081***		***0.001***	J	***0.01***		***0.00062***	J	***0.015***		--		--		--		--		--		***89.1***		--	
SE	SC-S155	PDI-SC-S155-4.2to5.3	05-Sep-18	N	Subsurface Sediment	4.2	5.3	ft	7628606.6	701255.1	-1.3	0.00023	J	0.00006	J	0.000087	J+	0.000016	U	0.00016	J+	--		--		--		--		--		2.58		--	
SE	SC-S157	PDI-SC-S157-0to2	17-Aug-18	N	Subsurface Sediment	0	2	ft	7629020.5	700988.0	-37.3	***0.0045***	J	0.00022	U	***0.0012***	J	***0.00023***	JN	***0.0014***	J+	--		--		--		--		--		***7.07***		--	
SE	SC-S157	PDI-SC-S157-10to12.4	17-Aug-18	N	Subsurface Sediment	10	12.4	ft	7629020.5	700988.0	-37.3	0.000064	U	0.000091	U	0.000068	U	0.00008	U	0.000046	U	--		--		--		--		--		0.25	U	--	
SE	SC-S157	PDI-SC-S157-12.4to14	17-Aug-18	N	Subsurface Sediment	12.4	14	ft	7629020.5	700988.0	-37.3	0.000065	U	0.000098	U	0.000062	U	0.000085	U	0.00017	U	--		--		--		--		--		0.09		--	
SE	SC-S157	PDI-SC-S157-14to15.9	17-Aug-18	N	Subsurface Sediment	14	15.9	ft	7629020.5	700988.0	-37.3	0.000045	U	0.000077	U	0.00006	U	0.00008	U	0.000038	U	--		--		--		--		--		0.04	U	--	
SE	SC-S157	PDI-SC-S157-2to3.7	17-Aug-18	N	Subsurface Sediment	2	3.7	ft	7629020.5	700988.0	-37.3	***0.0017***	J	***0.00023***	J	***0.0008***	J	0.00012	JN	***0.0019***	J+	--		--		--		--		--		***6.41***		--	
SE	SC-S157	PDI-SC-S157-3.7to6	17-Aug-18	N	Subsurface Sediment	3.7	6	ft	7629020.5	700988.0	-37.3	0.00011	U	0.00012	U	0.000075	U	0.0001	U	0.000093	U	--		--		--		--		--		2.19		--	
SE	SC-S157	PDI-SC-S157-6to8	17-Aug-18	N	Subsurface Sediment	6	8	ft	7629020.5	700988.0	-37.3	0.000086	U	0.00012	U	0.000074	U	0.0001	U	0.00022	U	--		--		--		--		--		0.25		--	
SE	SC-S157	PDI-SC-S157-8to10	17-Aug-18	N	Subsurface Sediment	8	10	ft	7629020.5	700988.0	-37.3	0.000072	U	0.00013	U	0.000074	U	0.000097	U	0.00016	U	--		--		--		--		--		0.39		--	
SE	SC-S163	PDI-SC-S163-0to2	27-Jul-18	N	Subsurface Sediment	0	2	ft	7629284.3	700353.0	-20.8	***0.0035***	J	***0.00086***	J	***0.00083***	JN	***0.00073***	JN	***0.001***		--		--		--		--		--		***15.1***		--	
SE	SC-S163	PDI-SC-S163-10TO12.7	27-Jul-18	N	Subsurface Sediment	10	12.7	ft	7629284.3	700353.0	-20.8	***0.1***		***0.0025***	J	***0.027***		***0.0023***		***0.037***	J	--		--		--		--		--		***113***		--	
SE	SC-S163	PDI-SC-S163-12.7to13	27-Jul-18	N	Subsurface Sediment	12.7	13	ft	7629284.3	700353.0	-20.8	0.0001	U	0.000092	U	0.000052	U	0.000084	U	0.00016	JN	--		--		--		--		--		0.23		--	
SE	SC-S163	PDI-SC-S163-2to4	27-Jul-18	N	Subsurface Sediment	2	4	ft	7629284.3	700353.0	-20.8	***0.0046***	J	***0.0012***	J	***0.0016***	J	***0.0013***		***0.0029***		--		--		--		--		--		***25.7***		--	
SE	SC-S163	PDI-SC-S163-4to6	27-Jul-18	N	Subsurface Sediment	4	6	ft	7629284.3	700353.0	-20.8	***0.0035***	J	***0.00098***	J	***0.0012***	J	***0.0013***		***0.00087***	J	--		--		--		--		--		***17.2***		--	
SE	SC-S163	PDI-SC-S163-6to8	27-Jul-18	N	Subsurface Sediment	6	8	ft	7629284.3	700353.0	-20.8	***0.015***		***0.0016***	J	***0.0036***	J	***0.0017***		***0.0047***		--		--		--		--		--		***28.4***		--	
SE	SC-S163	PDI-SC-S163-8to10	27-Jul-18	N	Subsurface Sediment	8	10	ft	7629284.3	700353.0	-20.8	***0.014***		***0.0019***	JN	***0.0043***	J	***0.0011***		***0.004***		--		--		--		--		--		***17.6***		--	

Notes:
µg/kg = microgram(s) per kilogram
mg/kg = milligram(s) per kilogram
cm = centimeters
ft = feet
J = estimated result
N = normal/primary sample
NAVD88 = North American Vertical Datum of 1988
U = non detect result
Bold Italic = detected result exceeds Sediment/Bank Soil CUL
Yellow = detected result exceeds applicable RAL (Sitewide)
Underline = detected result exceeds PTW Threshold

Table A-2. Pre-RD Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Matrix	Sys_Loc_Code	Sample Name	Sample Date	Sample Type	Depth Class	Start Depth	End Depth	Depth Unit	X Coordinate	Y Coordinate	Surface Elevation (ft NAVD 88)	gamma-BHC (Lindane) µg/kg		Lead mg/kg		Mercury mg/kg		Naphthalene µg/kg		Total BaP TE µg/kg		Total Chlordanes µg/kg		Total Dioxin/Furan TEQ TCDD TEQ µg/kg		Total PAHs µg/kg		Total PCB Aroclors µg/kg		Total PCB Congeners µg/kg	
											Sediment/Bank Soil CUL	**5**		**196**		**0.085**		**NA**		**774**		**1.4**		**0.01**		**23,000**		**9**		**9**	
											Sitewide RAL	--		--		--		**NA**		**NA**		--		--		**30,000**		**75**		**75**	
											PTW Threshold	--		--		--		**140,000**		**774,000**		--		--		**NA**		**200**		**200**	
SE	B236	PDI-SG-B236-BL1	20-Apr-18	N	Surface Sediment	0	30	cm	7628693.5	701205.1	-11.7	0.59	U	9.9		0.065		3.2		40.7		***2.475***		0.0037		441.4		--		7.9	
SE	B237	PDI-SG-B237-BL1	20-Apr-18	N	Surface Sediment	0	30	cm	7628595.4	700932.5	-19.6	0.57	UJ	11		0.049	J	5.2		50.5		***2.335***		0.0031		647.4		--		***10.1***	
SE	B239	PDI-SG-B239-BL1	21-Apr-18	N	Surface Sediment	0	30	cm	7628724.1	700674.0	-32.8	0.61	U	14		0.056	J	7.5		79.6		1.2	U	***0.0475***		910.1		--		***14.4***	
SE	B241	PDI-SG-B241-BL1	21-Apr-18	N	Surface Sediment	0	30	cm	7629093.9	700308.8	-14.0	0.58	U	13		0.075		12		92.1		***2.51***		0.0056		1,378		--		***18.1***	
SE	B245	PDI-SG-B245-BL1	21-Apr-18	N	Surface Sediment	0	22	cm	7629622.9	700261.9	-6.1	0.36	U	5.5		0.019	J	2.4		45.0		1	U	0.0008		307.51		--		2.9	
SE	B246	PDI-SG-B246-BL1	22-May-18	N	Surface Sediment	0	30	cm	7629841.9	700111.0	-3.3	0.92	U	8		0.04		7.2		61.0		2	U	0.0029		672.1		--		7.3	
SE	B251	PDI-SG-B251-BL1	22-May-18	N	Surface Sediment	0	30	cm	7630185.1	699860.8	-18.5	1.2	U	14		0.07		7.2		71.6		2.5	U	0.0039		734.3		--		***11.5***	
SE	B253	PDI-SG-B253-BL1	23-May-18	N	Surface Sediment	0	24	cm	7630335.5	699548.4	3.5	0.71	U	13		0.022	J	1.7		62.1		2	U	0.0014		435.5		--		***15.7***	
SE	B257	PDI-SG-B257-BL1	22-May-18	N	Surface Sediment	0	24	cm	7630634.1	699464.6	-1.7	0.58	U	1.7		0.022	U	0.59	U	0.5		2	U	0.0001		3.38		--		0.1	
SE	S157	PDI-SG-S157	14-May-18	N	Surface Sediment	0	30	cm	7628995.8	700977.0	-35.4	--		--		--		8.8		28.2		--		0.0091		392.9		--		***23.9***	
SE	S158	PDI-SG-S158	14-May-18	N	Surface Sediment	0	25	cm	7629028.1	701044.6	-35.1	--		--		--		39	J	85.5		--		0.0059		1,339		--		***42.8***	
SE	S159	PDI-SG-S159	18-May-18	N	Surface Sediment	0	30	cm	7629035.3	700769.7	-33.9	--		--		--		28		139		--		0.0072		3,402		--		***18.6***	
SE	S160	PDI-SG-S160	18-May-18	N	Surface Sediment	0	30	cm	7628845.2	700431.9	-32.7	--		--		--		10		80.4		--		0.0063		1,286		--		***18.7***	
SE	S161	PDI-SG-S161	14-May-18	N	Surface Sediment	0	29	cm	7629277.9	700783.8	-38.3	--		--		--		12		54.0		--		0.0041		771.9		--		***9.8***	
SE	S162	PDI-SG-S162	18-May-18	N	Surface Sediment	0	30	cm	7629427.9	700572.1	-23.2	--		--		--		11		93.2		--		0.0049		1,154		--		***11.4***	
SE	S163	PDI-SG-S163	18-May-18	N	Surface Sediment	0	30	cm	7629261.9	700355.8	-19.7	--		--		--		16		86.6		--		0.0082		1,039		--		***15.3***	
SE	S164	PDI-SG-S164	20-Jun-18	N	Surface Sediment	0	27	cm	7629972.7	700029.5	-9.5	--		--		--		31		125		--		0.0058		2,898		--		***13.2***	
SE	S165	PDI-SG-S165	16-May-18	N	Surface Sediment	0	27	cm	7630314.5	699724.3	-16.0	--		--		--		5.7		41.5		--		0.0046		410.3		--		***14.3***	
SE	SC-S155	PDI-SC-S155-0to2.1	05-Sep-18	N	Subsurface Sediment	0	2.1	ft	7628606.6	701255.1	-1.3	--		--		--		14	J	51.8		--		0.0034		619.1		***12.1***		--	
SE	SC-S155	PDI-SC-S155-2.1to4.2	05-Sep-18	N	Subsurface Sediment	2.1	4.2	ft	7628606.6	701255.1	-1.3	--		--		--		12	J	47.4		--		***0.0241***		562.8		***27.1***		--	
SE	SC-S155	PDI-SC-S155-4.2to5.3	05-Sep-18	N	Subsurface Sediment	4.2	5.3	ft	7628606.6	701255.1	-1.3	--		--		--		8.2		21.7		--		0.0003		176.5		2.6	U	--	
SE	SC-S157	PDI-SC-S157-0to2	17-Aug-18	N	Subsurface Sediment	0	2	ft	7629020.5	700988.0	-37.3	--		--		--		50		46.9		--		0.0030		777		***13.2***		--	
SE	SC-S157	PDI-SC-S157-10to12.4	17-Aug-18	N	Subsurface Sediment	10	12.4	ft	7629020.5	700988.0	-37.3	--		--		--		0.67	J	1.4		--		0.0002		15.08		2.6	U	--	
SE	SC-S157	PDI-SC-S157-12.4to14	17-Aug-18	N	Subsurface Sediment	12.4	14	ft	7629020.5	700988.0	-37.3	--		--		--		0.86	J	36.9		--		0.0002		52.25		2.6	U	--	
SE	SC-S157	PDI-SC-S157-14to15.9	17-Aug-18	N	Subsurface Sediment	14	15.9	ft	7629020.5	700988.0	-37.3	--		--		--		1.3		1.6		--		0.0002		15.02		2.6	U	--	
SE	SC-S157	PDI-SC-S157-2to3.7	17-Aug-18	N	Subsurface Sediment	2	3.7	ft	7629020.5	700988.0	-37.3	--		--		--		120		55.1		--		0.0022		1,764		***9.4***		--	
SE	SC-S157	PDI-SC-S157-3.7to6	17-Aug-18	N	Subsurface Sediment	3.7	6	ft	7629020.5	700988.0	-37.3	--		--		--		1.9		1.8		--		0.0004		28.86		2.7	U	--	
SE	SC-S157	PDI-SC-S157-6to8	17-Aug-18	N	Subsurface Sediment	6	8	ft	7629020.5	700988.0	-37.3	--		--		--		1.5	J	3.4		--		0.0004		45.19		2.7	U	--	
SE	SC-S157	PDI-SC-S157-8to10	17-Aug-18	N	Subsurface Sediment	8	10	ft	7629020.5	700988.0	-37.3	--		--		--		0.8	J	1.8		--		0.0003		16.21		2.7	U	--	
SE	SC-S163	PDI-SC-S163-0to2	27-Jul-18	N	Subsurface Sediment	0	2	ft	7629284.3	700353.0	-20.8	--		--		--		170		158		--		0.0080		2,998		***27***		--	
SE	SC-S163	PDI-SC-S163-10TO12.7	27-Jul-18	N	Subsurface Sediment	10	12.7	ft	7629284.3	700353.0	-20.8	--		--		--		330		589		--		***0.0475***		14,264		***285***		--	
SE	SC-S163	PDI-SC-S163-12.7to13	27-Jul-18	N	Subsurface Sediment	12.7	13	ft	7629284.3	700353.0	-20.8	--		--		--		92		264		--		0.0002		2,722		2.7	U	--	
SE	SC-S163	PDI-SC-S163-2to4	27-Jul-18	N	Subsurface Sediment	2	4	ft	7629284.3	700353.0	-20.8	--		--		--		370		160		--		***0.0113***		3,373		***177***		--	
SE	SC-S163	PDI-SC-S163-4to6	27-Jul-18	N	Subsurface Sediment	4	6	ft	7629284.3	700353.0	-20.8	--		--		--		380		192		--		***0.0101***		3,842		***59.75***		--	
SE	SC-S163	PDI-SC-S163-6to8	27-Jul-18	N	Subsurface Sediment	6	8	ft	7629284.3	700353.0	-20.8	--		--		--		360		235		--		***0.0157***		3,966		***88.2***		--	
SE	SC-S163	PDI-SC-S163-8to10	27-Jul-18	N	Subsurface Sediment	8	10	ft	7629284.3	700353.0	-20.8	--		--		--		460		348		--		***0.0219***		5,981		***50.8***		--	

Notes:
µg/kg = microgram(s) per kilogram
mg/kg = milligram(s) per kilogram
cm = centimeters
ft = feet
J = estimated result
N = normal/primary sample
NAVD88 = North American Vertical Datum of 1988
U = non detect result
Bold Italic = detected result exceeds Sediment/Bank Soil CUL
Yellow = detected result exceeds applicable RAL (Sitewide)
Underline = detected result exceeds PTW Threshold

Table A-2. Pre-RD Sample Data for ROD Table 17 and ROD Table 21 Constituents and Physical Parameters

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Matrix	Sys_Loc_Code	Sample Name	Sample Date	Sample Type	Depth Class	Start Depth	End Depth	Depth Unit	X Coordinate	Y Coordinate	Surface Elevation (ft NAVD 88)	TPH-Diesel Range Organics		Tri-n-butyltin		Zinc		TOC		Gravel	Sand, Coarse	Sand, Medium	Sand, Fine (adjusted)	Silt (adjusted)	Clay	Fines (silt + clay)
												mg/kg		µg/kg		mg/kg		mg/kg		%	%	%	%	%	%	%
											Sediment/Bank Soil CUL	**91**		**3,080**		**459**		--								
											Sitewide RAL	--		--		--		--								
											PTW Threshold	--		--		--		--								
SE	B236	PDI-SG-B236-BL1	20-Apr-18	N	Surface Sediment	0	30	cm	7628693.5	701205.1	-11.7	80	J	1.1	J	91		23000		0.0	0.0	2.1	18.8	69.0	10.1	79.1
SE	B237	PDI-SG-B237-BL1	20-Apr-18	N	Surface Sediment	0	30	cm	7628595.4	700932.5	-19.6	52	J	1.7	J	83		23000		0.0	0.0	0.1	9.0	79.4	11.5	90.9
SE	B239	PDI-SG-B239-BL1	21-Apr-18	N	Surface Sediment	0	30	cm	7628724.1	700674.0	-32.8	83	J	3		100		26000		0.0	0.0	0.1	9.2	78.2	12.6	90.8
SE	B241	PDI-SG-B241-BL1	21-Apr-18	N	Surface Sediment	0	30	cm	7629093.9	700308.8	-14.0	82	J	4.4		120		25000		0.0	0.0	0.1	11.7	77.8	10.4	88.2
SE	B245	PDI-SG-B245-BL1	21-Apr-18	N	Surface Sediment	0	22	cm	7629622.9	700261.9	-6.1	69	U	2.1		64		2700		0.1	0.0	6.3	85.7	4.2	3.6	7.8
SE	B246	PDI-SG-B246-BL1	22-May-18	N	Surface Sediment	0	30	cm	7629841.9	700111.0	-3.3	38	J	19		83		11000		0.0	1.6	7.7	49.4	37.2	4.2	41.4
SE	B251	PDI-SG-B251-BL1	22-May-18	N	Surface Sediment	0	30	cm	7630185.1	699860.8	-18.5	55	J	2.4	U	120		27000		0.0	0.0	0.3	10.2	75.4	14.2	89.6
SE	B253	PDI-SG-B253-BL1	23-May-18	N	Surface Sediment	0	24	cm	7630335.5	699548.4	3.5	27	J	16		140		4700		0.0	3.6	28.1	56.4	11.2	0.8	12.0
SE	B257	PDI-SG-B257-BL1	22-May-18	N	Surface Sediment	0	24	cm	7630634.1	699464.6	-1.7	54	U	1.2	U	24		590	J	28.8	32.8	25.7	11.6	1.2	0.0	1.2
SE	S157	PDI-SG-S157	14-May-18	N	Surface Sediment	0	30	cm	7628995.8	700977.0	-35.4	--		--		--		15000		0.3	0.4	3.9	48.7	37.9	8.8	46.7
SE	S158	PDI-SG-S158	14-May-18	N	Surface Sediment	0	25	cm	7629028.1	701044.6	-35.1	--		--		--		16000		0.0	0.1	1.4	34.5	52.6	11.6	64.2
SE	S159	PDI-SG-S159	18-May-18	N	Surface Sediment	0	30	cm	7629035.3	700769.7	-33.9	--		--		--		31000		0.0	0.0	0.2	5.9	83.0	10.9	93.9
SE	S160	PDI-SG-S160	18-May-18	N	Surface Sediment	0	30	cm	7628845.2	700431.9	-32.7	--		--		--		29000		0.0	0.0	0.2	6.6	84.8	8.4	93.2
SE	S161	PDI-SG-S161	14-May-18	N	Surface Sediment	0	29	cm	7629277.9	700783.8	-38.3	--		--		--		28000		0.0	0.0	0.2	7.6	76.1	16.2	92.3
SE	S162	PDI-SG-S162	18-May-18	N	Surface Sediment	0	30	cm	7629427.9	700572.1	-23.2	--		--		--		30000		0.0	0.0	0.1	6.2	85.2	8.5	93.7
SE	S163	PDI-SG-S163	18-May-18	N	Surface Sediment	0	30	cm	7629261.9	700355.8	-19.7	--		--		--		26000		0.0	0.0	0.2	9.8	81.8	8.1	89.9
SE	S164	PDI-SG-S164	20-Jun-18	N	Surface Sediment	0	27	cm	7629972.7	700029.5	-9.5	--		--		--		27000		0.0	0.1	0.3	13.3	78.5	7.8	86.3
SE	S165	PDI-SG-S165	16-May-18	N	Surface Sediment	0	27	cm	7630314.5	699724.3	-16.0	--		--		--		22000		0.0	0.0	0.1	11.6	75.7	12.5	88.2
SE	SC-S155	PDI-SC-S155-0to2.1	05-Sep-18	N	Subsurface Sediment	0	2.1	ft	7628606.6	701255.1	-1.3	--		--		--		29000		2.6	0.5	5.5	30.6	48.7	12.1	60.8
SE	SC-S155	PDI-SC-S155-2.1to4.2	05-Sep-18	N	Subsurface Sediment	2.1	4.2	ft	7628606.6	701255.1	-1.3	--		--		--		8900		0.3	0.4	28.6	58.3	8.7	3.7	12.4
SE	SC-S155	PDI-SC-S155-4.2to5.3	05-Sep-18	N	Subsurface Sediment	4.2	5.3	ft	7628606.6	701255.1	-1.3	--		--		--		1200	J	3.4	1.1	10	42.3	37.8	5.4	43.2
SE	SC-S157	PDI-SC-S157-0to2	17-Aug-18	N	Subsurface Sediment	0	2	ft	7629020.5	700988.0	-37.3	--		--		--		59000		0.0	0.8	0.8	23.2	61.0	14.2	75.2
SE	SC-S157	PDI-SC-S157-10to12.4	17-Aug-18	N	Subsurface Sediment	10	12.4	ft	7629020.5	700988.0	-37.3	--		--		--		8700		0.0	0.0	0	27.3	67.4	5.3	72.7
SE	SC-S157	PDI-SC-S157-12.4to14	17-Aug-18	N	Subsurface Sediment	12.4	14	ft	7629020.5	700988.0	-37.3	--		--		--		11000		0.0	0.0	0.1	7.1	87.0	5.9	92.9
SE	SC-S157	PDI-SC-S157-14to15.9	17-Aug-18	N	Subsurface Sediment	14	15.9	ft	7629020.5	700988.0	-37.3	--		--		--		11000		0.0	0.0	0	8.0	84.3	7.7	92.0
SE	SC-S157	PDI-SC-S157-2to3.7	17-Aug-18	N	Subsurface Sediment	2	3.7	ft	7629020.5	700988.0	-37.3	--		--		--		15000		0.0	0.2	1.8	72.1	22.3	3.7	26.0
SE	SC-S157	PDI-SC-S157-3.7to6	17-Aug-18	N	Subsurface Sediment	3.7	6	ft	7629020.5	700988.0	-37.3	--		--		--		1200	J	1.7	0.7	0.5	29.1	66.1	1.9	68.0
SE	SC-S157	PDI-SC-S157-6to8	17-Aug-18	N	Subsurface Sediment	6	8	ft	7629020.5	700988.0	-37.3	--		--		--		1400	J	0.0	0.2	0	45.0	51.1	3.8	54.9
SE	SC-S157	PDI-SC-S157-8to10	17-Aug-18	N	Subsurface Sediment	8	10	ft	7629020.5	700988.0	-37.3	--		--		--		6200		0.0	0.0	0	34.1	61.5	4.4	65.9
SE	SC-S163	PDI-SC-S163-0to2	27-Jul-18	N	Subsurface Sediment	0	2	ft	7629284.3	700353.0	-20.8	--		--		--		25000		0.0	0.1	0.5	13.0	74.3	12.2	86.5
SE	SC-S163	PDI-SC-S163-10TO12.7	27-Jul-18	N	Subsurface Sediment	10	12.7	ft	7629284.3	700353.0	-20.8	--		--		--		32000		0.5	0.9	0.2	5.3	72.2	20.8	93.0
SE	SC-S163	PDI-SC-S163-12.7to13	27-Jul-18	N	Subsurface Sediment	12.7	13	ft	7629284.3	700353.0	-20.8	--		--		--		8500		0.0	0.1	0.1	13.8	78.4	7.7	86.1
SE	SC-S163	PDI-SC-S163-2to4	27-Jul-18	N	Subsurface Sediment	2	4	ft	7629284.3	700353.0	-20.8	--		--		--		34000		0.1	0.0	0.2	9.3	76.2	14.3	90.5
SE	SC-S163	PDI-SC-S163-4to6	27-Jul-18	N	Subsurface Sediment	4	6	ft	7629284.3	700353.0	-20.8	--		--		--		30000		0.1	0.0	0.2	11.9	74.1	13.7	87.8
SE	SC-S163	PDI-SC-S163-6to8	27-Jul-18	N	Subsurface Sediment	6	8	ft	7629284.3	700353.0	-20.8	--		--		--		34000		0.0	0.0	0.1	7.0	74.7	18.2	92.9
SE	SC-S163	PDI-SC-S163-8to10	27-Jul-18	N	Subsurface Sediment	8	10	ft	7629284.3	700353.0	-20.8	--		--		--		38000		0.5	0.4	0.2	4.9	80.6	13.4	94.0

Notes:
µg/kg = microgram(s) per kilogram
mg/kg = milligram(s) per kilogram
cm = centimeters
ft = feet
J = estimated result
N = normal/primary sample
NAVD88 = North American Vertical Datum of 1988
U = non detect result
Bold Italic = detected result exceeds Sediment/Bank Soil CUL
Yellow = detected result exceeds applicable RAL (Sitewide)
Underline = detected result exceeds PTW Threshold

Table A-3A. Bank Soil Sampling Results – KMLT Willbridge Terminal

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Method Group	Analyte	Units	Portland Harbor CUL[a]	Portland Harbor RAL[a]	JSCS SLVs[b]		Location ID:	KM-S-3		KM-S-4		KM-S-4 DUP		KM-S-5		S-1		S-2		SB-N1							
					MacDonald PECs and other SQVs	DEQ 2007 Bioaccumulative Sediments SLVs	Sample ID:	KM-S-3		KM-S-4		KM-S-4 DUP		KM-S-5		S-1		S-2		SB-N1-092018-0-1		SB-N1-092018-12-13		SB-N1-092018-19.5-20		SB-N4-092118-0-1	
							Sample Date:	11/13/2012		11/13/2012		11/13/2012		11/13/2012		1/17/2018		1/17/2018		9/20/2018		9/20/2018		9/20/2018		9/21/2018	
							Sample Depth (feet):													0 to 1		12 to 13		19.5 to 20		0 to 1	
							Background[c]	Sampling Results																			
NWTPH-Dx	#2 Diesel (C10-C24)	mg/kg	91	--	--	--	--									**2.33**		**149**		40	U	**1,000**		21	U	410	UJ
	Motor oil (>C24-C36)	mg/kg	--	--	--	--	--									**9.49**		**423**		**210**		**190**		**110**		**700**	
	Gasoline	mg/kg	--	--	--	--	--									0.610	U	0.623	U								
VOCs	Toluene	µg/kg	--	--			--									**10.2**		**10.4**									
	Xylenes (total)	µg/kg	--	--			--									26	U	**40.4**									
	1,2,4-Trimethylbenzene	µg/kg	--	--			--									16.7	U	9.26	U								
	1,2,5-Trimethylbenzene	µg/kg	--	--			--									17.1	U	**20.8**									
Metals	Aluminum	mg/kg	--	--	--	--	--									**1,670**		**2,440**									
	Antimony	mg/kg	--	--	--	--	--									0.078	U	**4.76**									
	Arsenic	mg/kg	3	--	33	**7**	8.8	**6.34**		**88.7**		**81.6**		**1.83**		**4.42**		**129**		**13**		**3.4**		**50-**		**2.9**	
	Barium	mg/kg	--	--	--	--	--									**67.8**		**147**									
	Beryillium	mg/kg	--	--	--	--	--									0.249	U	**0.305**									
	Cadmium	mg/kg	0.51	--	4.98	**1**	0.63	**0.066**		**0.392**		**0.335**		**0.108**		0.238	U	**1.15**		**0.18**	J	**0.12**	J	0.098	U	0.062	U
	Chromium	mg/kg	--	--	--	--	--									**11.2**		**20.7**									
	Copper	mg/kg	359	--	**149**	--	34	**13.8**		**41.2**		**34.2**		**14.1**		**10.3**		**160**		**26**	J	**22**		**30**		**14**	
	Lead	mg/kg	196	--	128	**17**	79	**13.0**		**116**		**102**		**6.07**		**9.44**		**377**		**43**	J	**6.7**		**6.3**		**41**	
	Magnesium	mg/kg	--	--	--	--	--									**2,630**		**4,020**									
	Manganese	mg/kg	--	--	**1,100**	--	--	**283**		**366**		**327**		**1260**	J	**236**		**339**		**460**		**720**		**370**		**190**	
	Mercury	mg/kg	0.085	--	1.06	**0.07**	0.23													**0.019**	J	**0.026**	J	**0.038**	J	**0.013**	J
	Nickel	mg/kg	--	--	--	--	--									**11.9**		**18**									
	Selenium	mg/kg	--	--	--	--	--																				
	Silver	mg/kg	--	--	--	--	--									0.0438	U	**0.196**									
	Zinc	mg/kg	459	--	**459**	--	180	**62**		**179**		**138**		**47.4**	J	**42.6**		**1,000**		**98**	J	**55**		**60**		**60**	
SW8081B	alpha-chlordane	µg/kg	--	--			--	0.062	U	**0.618**	J	**0.671**	J	0.0620	U												
	gamma-chlordane	µg/kg	--	--			--	0.064	U	**0.727**	J	**0.787**	J	0.0640	U												
	Chlordane[b]	µg/kg	--	--			--	0.064	U	**1.345**	J	**1.458**	J	0.0640	U												
	2,4'-DDD	µg/kg	114	--	28	0.33	--	0.063	U	**4.04**	J	**3.48**	J	0.063	U					0.62	U	0.17	U	0.17	UJ	**0.88**	J
	4,4'-DDD	µg/kg	114	--	28	0.33	--	**0.141**		**15.8**	J	**15.7**	J	**0.21**		0.384	U	**60.4**		**2.3**		0.059	U	0.059	UJ	**0.86**	J
	2,4'-DDD and 4,4'-DDD	µg/kg	114	--			--	**0.141**		**19.84**	J	**19.18**	J	**0.21**													
	2,4'-DDE	µg/kg	50	--	31.3	0.33	--	0.079	U	**0.392**	J	**0.387**	J	0.079	U					0.51	U	0.14	U	0.14	UJ	0.3	UJ
	4,4'-DDE	µg/kg	50	--	31.3	0.33	--	0.079	U	**2.20**	J	**2.93**	J	**0.253**		0.384	U	**13.5**		**0.58**	J	0.08	U	0.08	UJ	0.18	UJ
	2,4'-DDE and 4,4'-DDE	µg/kg	50	--			--	0.079	U	**2.592**	J	**3.317**	J	**0.253**													
	2,4'-DDT	µg/kg	246	--	62.9	0.33	--	0.094	U	**5.56**	J	**6.36**	J	**0.366**						1.1	U	0.29	U	0.29	U	**1.3**	J
	4,4'-DDT	µg/kg	246	--	62.9	0.33	--	**0.795**		**45.9**	J	**34.6**	J	**2.79**		**0.603**		**107**		**8.7**		0.059	U	0.059	UJ	**6.1**	J
	2,4'-DDT and 4,4'-DDT	µg/kg	246	--			--	**0.795**		**51.46**	J	**40.96**	J	**3.156**													
	Total DDx[d]	µg/kg	6.1	160	--	0.33	--	0.2	U	0.788	U	**1.380**	J	0.2	U					**12.7**		0.399	U	0.399	UJ	**9.38**	J
	Endosulfan II	µg/kg	--	--			--	0.061	U	0.235	U	**0.164**	J	0.061	U												
	Endosulfan Sulfate	µg/kg	--	--			--	0.092	U	**1.45**		**1.310**	J	0.09200	U												
	Hexachlorobenzene	µg/kg	--	--			--	0.097	U	0.235	U	**0.152**	J	0.09700	U												
	cis-Nonachlor	µg/kg	--	--			--	0.058	U	**0.4390**	J	**0.499**	J	0.0590	U												
	trans-Nonachlor	µg/kg	--	--			--																				
Phthalates	bis(2-ethylhexyl)phthalate	µg/kg	135	--			--									**17.9**		**108**									
SW8082A	PCB-1016	mg/kg	--	--	**0.53**	--	--													0.00038	U	0.00051	U	0.00051	UJ	0.00038	U
	PCB-1221	mg/kg	--	--	--	--	--													0.0011	U	0.0014	U	0.0014	UJ	0.0011	U
	PCB-1232	mg/kg	--	--	--	--	--													0.00053	U	0.00071	U	0.00071	UJ	0.00052	U
	PCB-1242	mg/kg	--	--	--	--	--													0.00055	U	0.00074	U	0.00074	UJ	0.00054	U
	PCB-1248	mg/kg	--	--	**1.5**	--	--													0.00018	U	0.00024	U	0.00024	UJ	0.00018	U
	PCB-1254	mg/kg	--	--	**0.3**	--	--													0.00089	U	0.0012	U	0.0012	UJ	0.00088	U
	PCB-1260	mg/kg	--	--	**0.2**	--	--													**0.00091**	J	0.00051	U	0.00051	UJ	**0.0021**	J
	Polychlorinated biphenyls, total	mg/kg	0.009	0.075	0.676	**0.00039**	--													0.0011	U	0.0014	U	0.0014	UJ	**0.0021**	J

Table A-3A. Bank Soil Sampling Results – KMLT Willbridge Terminal

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Method Group	Analyte	Units	Portland Harbor CUL[a]	Portland Harbor RAL[a]	JSCS SLVs[b]		Location ID:	KM-S-3		KM-S-4		KM-S-4 DUP		KM-S-5		S-1		S-2		SB-N1							
					MacDonald PECs and other SQVs	DEQ 2007 Bioaccumulative Sediments SLVs	Sample ID:	KM-S-3		KM-S-4		KM-S-4 DUP		KM-S-5		S-1		S-2		SB-N1-092018-0-1		SB-N1-092018-12-13		SB-N1-092018-19.5-20		SB-N4-092118-0-1	
							Sample Date:	11/13/2012		11/13/2012		11/13/2012		11/13/2012		1/17/2018		1/17/2018		9/20/2018		9/20/2018		9/20/2018		9/21/2018	
							Sample Depth (feet):													0 to 1		12 to 13		19.5 to 20		0 to 1	
							Background[c]	Sampling Results																			
SW8270-SIM-PAH	1-Methylnaphthalene	µg/kg	--	--	--	--	--													5.8	J	0.94	U	0.93	U	7	U
	2-Methylnaphthalene	µg/kg	--	--	**200**	--	--													9.5	J	0.67	U	0.67	U	5.5	J
	Acenaphthene	µg/kg	--	--	**300**	--	--													4.1	J	39		0.89	U	6.7	U
	Acenaphthylene	µg/kg	--	--	**200**	--	--													6.3	J	19		0.74	U	5.6	U
	Anthracene	µg/kg	--	--	**845**	--	--									3.83	U	37.8		7.8	J	45		0.89	U	6.7	U
	Benzo(a)anthracene	µg/kg	--	--	**1,050**	--	--									12		82.6		67		5.4	J	1.1	U	15	J
	Benzo(a)pyrene	µg/kg	--	--	**1,450**	--	--									10.8		175		80		1.7	J	0.59	U	52	J
	Benzo(b)fluoranthene	µg/kg	--	--	--	--	--									7.21		131		80		0.88	U	2.6	J	33	J
	Benzo(g,h,i)perylene	µg/kg	--	--	**300**	--	--									6.7		192		52		2.2	J	0.74	U	80	
	Benzo(k)fluoranthene	µg/kg	--	--	--	--	--									8.89		94.3		40		0.89	U	0.89	U	11	J
	Chrysene	µg/kg	--	--	**1,290**	--	--									12.3		240		110		7.5		2.2	U	61	
	Dibenz(a,h)anthracene	µg/kg	--	--	**1,300**	--	--									3.83	U	41.8		4	U	1.1	U	1.1	U	19	J
	Fluoranthene	µg/kg	--	--	**2,230**	37,000	--									15		305		110		16		6.1	U	22	U
	Fluorene	µg/kg	--	--	**536**	--	--													2.8	J	90		0.74	U	5.6	U
	Indeno(1,2,3-cd)pyrene	µg/kg	--	--	**100**	--	--									5.33		92.8		40		0.89	U	0.89	U	36	J
	Naphthalene	µg/kg	--	--	**561**	--	--													9.8	J	35		1.2	U	9.6	J
	Phenanthrene	µg/kg	--	--	**1,170**	--	--									7.19		175		42		200		2.5	J	7.7	U
	Pyrene	µg/kg	--	--	**1,520**	1,900	--									19.1		228		120		16		3.8	J	30	J
	TPAHs	µg/kg	23,000	30,000	--	--	--													770		480		15		370	
	cPAHs (BaP eq)[e]	µg/kg	85	--	--	--	--									15		249		101		2.89		1.21		79.57	
SW8290	1,2,3,4,7,8-HxCDF	ng/kg	0.4	--	--	2.7	--													5	J	0.16	U	0.69	U	8.1	
	1,2,3,7,8-PeCDD	ng/kg	0.2	0.8	--	2.6	--													0.9	J	0.15	U	0.51	U	1.6	J
	2,3,4,7,8-PeCDF	ng/kg	0.3	200	--	0.03	--													0.83	J	0.1	U	0.32	U	2	J
	2,3,7,8-TCDD	ng/kg	0.2	0.6	9	0.0091	--													0.2	U	0.11	U	0.47	U	0.4	J
	2,3,7,8-TCDF	ng/kg	0.40658	--	--	0.77	--													0.64	J	0.049	U	0.22	U	1.6	
SW9060A	Total organic carbon - average dup	mg/kg	--	--	--	--	--	**480**	J	**5,240**	J	--		**1,600**	J					9,800		4,300		4,700		5,800	

Notes:

a The source of the CUL or RAL is the Portland Harbor Cleanup Level (CUL) or Remedial Action Level (RAL) of the Portland Harbor Record of Decision, dated 2017 and subsequent Explanation of Significant differences and errata. If the CUL or RAL is not available, the bolded SLV is used for screening.

[b] Chlordane is equal to the sum of all chlordanes (e.g. alpha and gamma-chorldane). Non-detects are not included in total. If both non-detects, use higher of the two detection limits for total.

[c] Development of Oregon Background Metals Concentrations in Soil – Table 4 – Regional 95% Upper Prediction Limit Default Background Concentrations for Metals in Soil – State of Oregon – Portland Basin, March 2013.

[d] Total DDx = The sum of detected 2,4' and 4,4' compounds. If non-detect, half the detection limit was used.

[e] cPAHs = Benzo(a)pyrene equivalent (BaP eq) values used to represent carcinogenic PAHs (cPAHs). cPAHs were calculated by multiplying the cPAHs by their respective equivalency factors (PEFs) and summing the resulting concentrations. If non-detect, half the detection limit was used.

Bold = value detected

Bold and shaded = detected result exceeds CUL.

Bold and Yellow = detected result exceeds applicable RAL (Sitewide)

µg/kg = micrograms per kilogram

mg/kg = milligrams per kilogram

ng/kg = nanograms per kilogram

NWTPH-Dx = Northwest TPH-diesel range

PCB = polychlorinated biphenyl

PAH = polycyclic aromatic hydrocarbon

DDx = dichlorodiphenyldichloroethane (DDD), dichlorodiphenyldichloroethene (DDE), and dichlorodiphenyltrichloroethane (DDT) compounds

TPAH = total polycyclic aromatic hydrocarbons

TPH = total petroleum hydrocarbons

Table A-3A. Bank Soil Sampling Results – KMLT Willbridge Terminal

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Method Group	Analyte	Units	Portland Harbor CUL[a]	Portland Harbor RAL[a]	JSCS SLVs[b]		Location ID:	SB-N4			
					MacDonald PECs and other SQVs	DEQ 2007 Bioaccumulative Sediments SLVs	Sample ID:	SB-N4-092118-4.5-5.5		SB-N4-092118-14-15	
							Sample Date:	9/21/2018		9/21/2018	
							Sample Depth (feet):	4.5 to 5.5		14 to 15	
							Background[c]				
NWTPH-Dx	#2 Diesel (C10-C24)	mg/kg	91	--	--	--	--	**2,100**		18	U
	Motor oil (>C24-C36)	mg/kg	--	--	--	--	--	**370**		67	U
	Gasoline	mg/kg	--	--	--	--	--				
VOCs	Toluene	µg/kg	--	--			--				
	Xylenes (total)	µg/kg	--	--			--				
	1,2,4-Trimethylbenzene	µg/kg	--	--			--				
	1,2,5-Trimethylbenzene	µg/kg	--	--			--				
Metals	Aluminum	mg/kg	--	--	--	--	--				
	Antimony	mg/kg	--	--	--	--	--				
	Arsenic	mg/kg	3	--	33	**7**	8.8	**4.2**		**4.5**	
	Barium	mg/kg	--	--	--	--	--				
	Beryillium	mg/kg	--	--	--	--	--				
	Cadmium	mg/kg	0.51	--	4.98	**1**	0.63	0.088	U	**0.12**	J
	Chromium	mg/kg	--	--	--	--	--				
	Copper	mg/kg	359	--	**149**	--	34	**19**		**33**	
	Lead	mg/kg	196	--	128	**17**	79	**4.8**		**7.3**	
	Magnesium	mg/kg	--	--	--	--	--				
	Manganese	mg/kg	--	--	**1,100**	--	--	**260**		**1,200**	
	Mercury	mg/kg	0.085	--	1.06	**0.07**	0.23	**0.035**		**0.055**	
	Nickel	mg/kg	--	--	--	--	--				
	Selenium	mg/kg	--	--	--	--	--				
	Silver	mg/kg	--	--	--	--	--				
	Zinc	mg/kg	459	--	**459**	--	180	**50**		**73**	
SW8081B	alpha-chlordane	µg/kg	--	--			--				
	gamma-chlordane	µg/kg	--	--			--				
	Chlordane[b]	µg/kg	--	--			--				
	2,4'-DDD	µg/kg	114	--	28	0.33	--	0.14	U	0.16	UJ
	4,4'-DDD	µg/kg	114	--	28	0.33	--	0.049	U	0.057	UJ
	2,4'-DDD and 4,4'-DDD	µg/kg	114	--			--				
	2,4'-DDE	µg/kg	50	--	31.3	0.33	--	0.11	U	0.13	UJ
	4,4'-DDE	µg/kg	50	--	31.3	0.33	--	0.067	U	0.077	UJ
	2,4'-DDE and 4,4'-DDE	µg/kg	50	--			--				
	2,4'-DDT	µg/kg	246	--	62.9	0.33	--	0.24	U	0.28	UJ
	4,4'-DDT	µg/kg	246	--	62.9	0.33	--	**0.32**		0.057	UJ
	2,4'-DDT and 4,4'-DDT	µg/kg	246	--			--				
	Total DDx[d]	µg/kg	6.1	160	--	0.33	--	**0.503**		0.3805	UJ
	Endosulfan II	µg/kg	--	--			--				
	Endosulfan Sulfate	µg/kg	--	--			--				
	Hexachlorobenzene	µg/kg	--	--			--				
	cis-Nonachlor	µg/kg	--	--			--				
	trans-Nonachlor	µg/kg	--	--			--				
Phthalates	bis(2-ethylhexyl)phthalate	µg/kg	135	--			--				
SW8082A	PCB-1016	mg/kg	--	--	**0.53**	--	--	0.00043	U	0.0005	UJ
	PCB-1221	mg/kg	--	--	--	--	--	0.0012	U	0.0014	UJ
	PCB-1232	mg/kg	--	--	--	--	--	0.00059	U	0.00069	UJ
	PCB-1242	mg/kg	--	--	--	--	--	0.00062	U	0.00072	UJ
	PCB-1248	mg/kg	--	--	**1.5**	--	--	0.0002	U	0.00023	UJ
	PCB-1254	mg/kg	--	--	**0.3**	--	--	0.001	U	0.0012	UJ
	PCB-1260	mg/kg	--	--	**0.2**	--	--	0.00043	U	0.0005	UJ
	Polychlorinated biphenyls, total	mg/kg	0.009	0.075	0.676	**0.00039**	--	0.0012	U	0.0014	UJ

Table A-3A. Bank Soil Sampling Results – KMLT Willbridge Terminal

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Method Group	Analyte	Units	Portland Harbor CUL[a]	Portland Harbor RAL[a]	JSCS SLVs[b]		Location ID:	SB-N4			
					MacDonald PECs and other SQVs	DEQ 2007 Bioaccumulative Sediments SLVs	Sample ID:	SB-N4-092118-4.5-5.5		SB-N4-092118-14-15	
							Sample Date:	9/21/2018		9/21/2018	
							Sample Depth (feet):	4.5 to 5.5		14 to 15	
							Background[c]				
SW8270-SIM-PAH	1-Methylnaphthalene	µg/kg	--	--	--	--	--	0.78	U	0.94	U
	2-Methylnaphthalene	µg/kg	--	--	**200**	--	--	0.56	U	0.67	U
	Acenaphthene	µg/kg	--	--	**300**	--	--	0.74	U	0.89	U
	Acenaphthylene	µg/kg	--	--	**200**	--	--	0.62	U	0.74	U
	Anthracene	µg/kg	--	--	**845**	--	--	0.74	U	0.89	U
	Benzo(a)anthracene	µg/kg	--	--	**1,050**	--	--	**21**		1.1	U
	Benzo(a)pyrene	µg/kg	--	--	**1,450**	--	--	**15**		0.6	U
	Benzo(b)fluoranthene	µg/kg	--	--	--	--	--	**17**		0.88	U
	Benzo(g,h,i)perylene	µg/kg	--	--	**300**	--	--	**13**		0.74	U
	Benzo(k)fluoranthene	µg/kg	--	--	--	--	--	**13**		0.89	U
	Chrysene	µg/kg	--	--	**1,290**	--	--	**48**		2.2	U
	Dibenz(a,h)anthracene	µg/kg	--	--	**1,300**	--	--	0.89	U	1.1	U
	Fluoranthene	µg/kg	--	--	**2,230**	37,000	--	1.7	U	2.1	U
	Fluorene	µg/kg	--	--	**536**	--	--	0.62	U	0.74	U
	Indeno(1,2,3-cd)pyrene	µg/kg	--	--	**100**	--	--	**14**		0.89	U
	Naphthalene	µg/kg	--	--	**561**	--	--	**20**		1.2	U
	Phenanthrene	µg/kg	--	--	**1,170**	--	--	0.85	U	1	U
	Pyrene	µg/kg	--	--	**1,520**	1,900	--	**84**		1.4	U
	TPAHs	µg/kg	23,000	30,000	--	--	--	**250**		2.2	U
	cPAHs (BaP eq)[e]	µg/kg	85	--	--	--	--	**20.82**		**1.00**	
SW8290	1,2,3,4,7,8-HxCDF	ng/kg	0.4	--	--	2.7	--	0.24	U	0.15	U
	1,2,3,7,8-PeCDD	ng/kg	0.2	0.8	--	2.6	--	0.18	UJ	0.15	U
	2,3,4,7,8-PeCDF	ng/kg	0.3	200	--	0.03	--	**0.21**	J	0.1	U
	2,3,7,8-TCDD	ng/kg	0.2	0.6	9	0.0091	--	**0.38**	J	0.09	U
	2,3,7,8-TCDF	ng/kg	0.40658	--	--	0.77	--	0.069	UJ	0.2	U
SW9060A	Total organic carbon - average dup	mg/kg	--	--	--	--	--	5,700		6,200	

Notes:

a The source of the CUL or RAL is the Portland Harbor Cleanup Level (CUL) or Remedial Action Level (RAL) of the Portland Harbor Record of Decision, dated 2017 and subsequent Explanation of Significant differences and errata. If the CUL or RAL is not available, the bolded SLV is used for screening.

[b] Chlordane is equal to the sum of all chlordanes (e.g. alpha and gamma-chorldane). Non-detects are not included in total. If both non-detects, use higher of the two detection limits for total.

[c] Development of Oregon Background Metals Concentrations in Soil – Table 4 – Regional 95% Upper Prediction Limit Default Background Concentrations for Metals in Soil – State of Oregon – Portland Basin, March 2013.

[d] Total DDx = The sum of detected 2,4' and 4,4' compounds. If non-detect, half the detection limit was used.

[e] cPAHs = Benzo(a)pyrene equivalent (BaP eq) values used to represent carcinogenic PAHs (cPAHs). cPAHs were calculated by multiplying the cPAHs by their respective equivalency factors (PEFs) and summing the resulting concentrations. If non-detect, half the detection limit was used.

Bold = value detected

Bold and shaded = detected result exceeds CUL.

Bold and Yellow = detected result exceeds applicable RAL (Sitewide)

µg/kg = micrograms per kilogram

mg/kg = milligrams per kilogram

ng/kg = nanograms per kilogram

NWTPH-Dx = Northwest TPH-diesel range

PCB = polychlorinated biphenyl

PAH = polycyclic aromatic hydrocarbon

DDx = dichlorodiphenyldichloroethane (DDD), dichlorodiphenyldichloroethene (DDE), and dichlorodiphenyltrichloroethane (DDT) compounds

TPAH = total polycyclic aromatic hydrocarbons

TPH = total petroleum hydrocarbons

Table A-3A. Bank Soil Sampling Results – KMLT Willbridge Terminal

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Method Group	Analyte	Units	Portland Harbor CUL[a]	Portland Harbor RAL[a]	JSCS SLVs[b]		Location ID:	SB-N5						SB-N6						SB-S1				SB-S4			
					MacDonald PECs and other SQVs	DEQ 2007 Bioaccumulative Sediments SLVs	Sample ID:	SB-N5-092118-0-1		SB-N5-092118-5-6		SB-N5-092118-14-15		SB-N6-092118-0-1		SB-N6-092118-7-9.5		SB-N6-092118-19-20		SB-S1-092118-0-1		SB-S1-092118-12-13		SB-S4-092118-0-1		SB-S4-092118-4.5-7	
							Sample Date:	9/21/2018		9/21/2018		9/21/2018		9/21/2018		9/21/2018		9/21/2018		9/21/2018		9/21/2018		9/21/2018		9/21/2018	
							Sample Depth (feet):	0 to 1		5 to 6		14 to 15		0 to 1		7 to 9.5		19 to 20		0 to 1		12 to 13		0 to 1		4.5 to 7	
							Background[c]	Sampling Results																			
NWTPH-Dx	#2 Diesel (C10-C24)	mg/kg	91	--	--	--	--	170	U	**450**		18	U	**1,100**	J	**2,800**		27	U	15	U	17	U	59	UJ	15	U
	Motor oil (>C24-C36)	mg/kg	--	--	--	--	--	530	UJ	**180**		51	U	**2,600**		**290**		**120**		33	U	**29**	J	**220**		59	U
	Gasoline	mg/kg	--	--	--	--	--																				
VOCs	Toluene	µg/kg	--	--			--																				
	Xylenes (total)	µg/kg	--	--			--																				
	1,2,4-Trimethylbenzene	µg/kg	--	--			--																				
	1,2,5-Trimethylbenzene	µg/kg	--	--			--																				
Metals	Aluminum	mg/kg	--	--	--	--	--																				
	Antimony	mg/kg	--	--	--	--	--																				
	Arsenic	mg/kg	3	--	33	**7**	8.8	**23**		**9.3**		**5**		**6**		**4**		**3.4**		**3.5**		**2.8**		**3.6**		**5**	
	Barium	mg/kg	--	--	--	--	--																				
	Beryillium	mg/kg	--	--	--	--	--																				
	Cadmium	mg/kg	0.51	--	4.98	**1**	0.63	**0.43**		**0.68**		**0.16**	J	**0.22**	J	**0.11**	J	**0.4**	J	0.075	U	0.067	U	**0.07**	J	**0.074**	J
	Chromium	mg/kg	--	--	--	--	--																				
	Copper	mg/kg	359	--	**149**	--	34	**42**		**44**		**42**		**21**		**36**		**17**		**27**		**23**		**17**		**18**	
	Lead	mg/kg	196	--	128	**17**	79	**110**		**220**		**11**		**100**		**9.7**		**5.5**		**4.7**		**5.5**		**7**		**6.3**	
	Magnesium	mg/kg	--	--	--	--	--																				
	Manganese	mg/kg	--	--	**1,100**	--	--	**390**		**400**		**660**		**260**		**400**		**240**		**290**		**520**		**390**		**270**	
	Mercury	mg/kg	0.085	--	1.06	**0.07**	0.23	**0.061**		**0.068**		**0.045**		**0.051**		**0.072**		**0.04**		**0.016**	J	**0.02**	J	**0.042**		0.011	U
	Nickel	mg/kg	--	--	--	--	--																				
	Selenium	mg/kg	--	--	--	--	--																				
	Silver	mg/kg	--	--	--	--	--																				
	Zinc	mg/kg	459	--	**459**	--	180	**250**		**200**		**85**		**140**		**67**		**54**		**56**		**41**		**67**		**67**	
SW8081B	alpha-chlordane	µg/kg	--	--			--																				
	gamma-chlordane	µg/kg	--	--			--																				
	Chlordane[b]	µg/kg	--	--			--																				
	2,4'-DDD	µg/kg	114	--	28	0.33	--	0.64	U	0.16	UJ	0.16	U	**84**	J	0.89	U	0.17	U	**0.77**		0.15	U	**4.8**		0.14	U
	4,4'-DDD	µg/kg	114	--	28	0.33	--	**2.8**		0.058	UJ	0.058	U	**200**	J	0.32	U	0.061	U	**0.6**		0.053	U	**17**	J	**0.61**	
	2,4'-DDD and 4,4'-DDD	µg/kg	114	--			--																				
	2,4'-DDE	µg/kg	50	--	31.3	0.33	--	0.53	U	0.14	UJ	0.13	U	**7.5**	J	0.74	U	0.14	U	0.11	U	0.12	U	0.5	U	0.11	U
	4,4'-DDE	µg/kg	50	--	31.3	0.33	--	**1**	J	0.079	UJ	0.079	U	**14**	J	0.43	U	0.082	U	0.067	U	0.073	U	**7.5**		**0.31**	
	2,4'-DDE and 4,4'-DDE	µg/kg	50	--			--																				
	2,4'-DDT	µg/kg	246	--	62.9	0.33	--	**1.1**	J	0.28	UJ	0.28	U	12	U	1.5	U	0.3	U	0.24	U	0.26	U	**15**		0.23	U
	4,4'-DDT	µg/kg	246	--	62.9	0.33	--	**12**	J	0.058	UJ	0.058	U	2.5	U	0.32	U	0.061	U	0.049	U	0.053	U	**72**		**0.85**	J
	2,4'-DDT and 4,4'-DDT	µg/kg	246	--			--																				
	Total DDx[d]	µg/kg	6.1	160	--	0.33	--	**19.385**		0.3875	UJ	0.3825	U	**312.75**		2.1	U	0.407	U	**1.603**		0.3545	U	**116.55**		**2.47**	
	Endosulfan II	µg/kg	--	--			--																				
	Endosulfan Sulfate	µg/kg	--	--			--																				
	Hexachlorobenzene	µg/kg	--	--			--																				
	cis-Nonachlor	µg/kg	--	--			--																				
	trans-Nonachlor	µg/kg	--	--			--																				
Phthalates	bis(2-ethylhexyl)phthalate	µg/kg	135	--			--																				
SW8082A	PCB-1016	mg/kg	--	--	**0.53**	--	--	0.00039	U	0.00051	U	0.0005	U	0.0043	UJ	0.00055	UJ	0.00053	UJ	0.00043	U	0.00047	U	0.00037	U	0.00042	U
	PCB-1221	mg/kg	--	--	--	--	--	0.0011	U	0.0014	U	0.0014	U	0.012	UJ	0.0015	UJ	0.0015	UJ	0.0012	U	0.0013	U	0.001	U	0.0012	U
	PCB-1232	mg/kg	--	--	--	--	--	0.00054	U	0.0007	U	0.0007	U	0.006	UJ	0.00076	UJ	0.00073	UJ	0.00059	U	0.00064	U	0.00052	U	0.00058	U
	PCB-1242	mg/kg	--	--	--	--	--	0.00057	U	0.00073	U	0.00073	U	0.0062	UJ	0.00079	UJ	0.00076	UJ	0.00062	U	0.00067	U	0.00054	U	0.0006	U
	PCB-1248	mg/kg	--	--	**1.5**	--	--	0.00018	U	0.00024	U	0.00024	U	0.002	UJ	0.00026	UJ	0.00025	UJ	0.0002	U	0.00022	U	0.00018	U	0.0002	U
	PCB-1254	mg/kg	--	--	**0.3**	--	--	0.00091	U	0.0012	U	0.0012	U	0.01	UJ	0.0013	UJ	0.0012	UJ	0.00099	U	0.0011	U	0.00087	U	0.00097	U
	PCB-1260	mg/kg	--	--	**0.2**	--	--	**0.0067**		0.00051	U	0.0005	U	**0.0066**	J	0.00055	UJ	0.00053	UJ	0.00043	U	0.00047	U	**0.017**		0.00042	U
	Polychlorinated biphenyls, total	mg/kg	0.009	0.075	0.676	**0.00039**	--	**0.0067**		0.0014	U	0.0014	U	0.012	UJ	0.0015	UJ	0.0015	UJ	0.0012	U	0.0013	U	**0.017**		0.0012	U

Table A-3A. Bank Soil Sampling Results – KMLT Willbridge Terminal

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Method Group	Analyte	Units	Portland Harbor CUL[a]	Portland Harbor RAL[a]	JSCS SLVs[b]		Location ID:	SB-N5						SB-N6						SB-S1				SB-S4			
					MacDonald PECs and other SQVs	DEQ 2007 Bioaccumulative Sediments SLVs	Sample ID:	SB-N5-092118-0-1		SB-N5-092118-5-6		SB-N5-092118-14-15		SB-N6-092118-0-1		SB-N6-092118-7-9.5		SB-N6-092118-19-20		SB-S1-092118-0-1		SB-S1-092118-12-13		SB-S4-092118-0-1		SB-S4-092118-4.5-7	
							Sample Date:	9/21/2018		9/21/2018		9/21/2018		9/21/2018		9/21/2018		9/21/2018		9/21/2018		9/21/2018		9/21/2018		9/21/2018	
							Sample Depth (feet):	0 to 1		5 to 6		14 to 15		0 to 1		7 to 9.5		19 to 20		0 to 1		12 to 13		0 to 1		4.5 to 7	
							Background[c]	Sampling Results																			
SW8270-SIM-PAH	1-Methylnaphthalene	µg/kg	--	--	--	--	--	**7.5**	J	**170**		0.9	U	**32**	J	**8,300**		**3.3**	J	**1.2**	J	0.82	U	**5.3**	J	**0.97**	J
	2-Methylnaphthalene	µg/kg	--	--	**200**	--	--	**12**	J	**340**		0.65	U	**83**	J	**220**		0.7	U	0.56	U	0.59	U	**11**	J	**1.3**	J
	Acenaphthene	µg/kg	--	--	**300**	--	--	**11**	J	**66**		0.86	U	19	U	**550**		0.93	U	**1.8**	J	**1.9**	J	**4.2**	J	**0.82**	J
	Acenaphthylene	µg/kg	--	--	**200**	--	--	**9.2**	J	**100**		0.72	U	**49**	J	**370**		0.78	U	0.62	U	0.65	U	**8.8**	J	**1.4**	J
	Anthracene	µg/kg	--	--	**845**	--	--	**22**	J	**200**		0.86	U	**51**	J	**310**		0.93	U	**2.6**	J	0.79	U	**18**	J	**1.9**	J
	Benzo(a)anthracene	µg/kg	--	--	**1,050**	--	--	**110**		**490**		**1.1**	J	**97**	J	**40**		1.2	U	**7**		0.99	U	**41**		**8.9**	
	Benzo(a)pyrene	µg/kg	--	--	**1,450**	--	--	**170**		**590**		0.57	U	**270**		**15**		0.62	U	**3.9**	J	0.52	U	**57**		**11**	
	Benzo(b)fluoranthene	µg/kg	--	--	--	--	--	**180**		**500**		**2.9**	J	**150**	J	**27**		0.92	U	**4.5**	J	0.77	U	**66**		**12**	
	Benzo(g,h,i)perylene	µg/kg	--	--	**300**	--	--	**130**		**340**		0.72	U	**360**		**6.5**	J	0.78	U	**3.2**	J	0.65	U	**57**		**13**	
	Benzo(k)fluoranthene	µg/kg	--	--	--	--	--	**82**		**220**		**0.98**	J	**31**	J	**14**		0.93	U	**1.9**	J	0.79	U	**30**		**5.5**	J
	Chrysene	µg/kg	--	--	**1,290**	--	--	160		**620**		**2.2**	J	**470**		**74**		2.3	U	**7.9**		2	U	**61**		**17**	
	Dibenz(a,h)anthracene	µg/kg	--	--	**1,300**	--	--	4.1	U	**80**		1	U	23	U	1.2	U	1.1	U	0.9	U	0.94	U	3.8	U	0.82	U
	Fluoranthene	µg/kg	--	--	**2,230**	37,000	--	**200**		**610**		2	U	**220**		**76**		2.2	U	6.4	U	1.8	U	**90**		**19**	
	Fluorene	µg/kg	--	--	**536**	--	--	**9.6**	J	**65**		**0.89**	J	16	U	**1,300**		**1**	J	**1.2**	J	**5.1**	J	**3.7**	J	**0.57**	J
	Indeno(1,2,3-cd)pyrene	µg/kg	--	--	**100**	--	--	**150**		**440**		0.86	U	**220**		**8.3**		0.93	U	**3.1**	J	0.79	U	**57**		**14**	
	Naphthalene	µg/kg	--	--	**561**	--	--	**20**	J	**330**		1.1	U	**150**	J	**780**		1.2	U	**1.6**	J	1	U	**19**	J	**2.6**	J
	Phenanthrene	µg/kg	--	--	**1,170**	--	--	**110**		**350**		**1.6**	J	**100**	J	**1,800**		**2.4**	J	**4.3**	J	**3.4**	J	**59**		**7.7**	
	Pyrene	µg/kg	--	--	**1,520**	1,900	--	**230**		**910**		**1.8**	J	**540**		**130**		**1.9**	J	**20**		1.3	U	**91**		**24**	
	TPAHs	µg/kg	23,000	30,000	--	--	--	**1,600**		**5,900**		**11**		**2,700**		**5,500**		5.3	U	**69**		**10**		**660**		**140**	
	cPAHs (BaP eq)[e]	µg/kg	85	--	--	--	--	**217.03**		**815.82**		**1.24**		**328.98**		**23.34**		**1.02**		**5.84**		**0.86**		**75.66**		**14.97**	
SW8290	1,2,3,4,7,8-HxCDF	ng/kg	0.4	--	--	2.7	--	**12**		0.14	U	0.85	UJ	**43**		0.46	U	0.56	U	0.64	UJ	0.37	U	0.64	U	0.69	UJ
	1,2,3,7,8-PeCDD	ng/kg	0.2	0.8	--	2.6	--	**1.8**	J	0.15	UJ	0.5	U	**5.9**	J	0.4	U	0.57	U	0.39	UJ	0.41	U	**1.1**	J	0.46	UJ
	2,3,4,7,8-PeCDF	ng/kg	0.3	200	--	0.03	--	**2.6**	J	0.11	U	0.33	U	4.6	U	0.26	U	0.37	U	0.31	UJ	0.27	U	0.44	UJ	0.34	UJ
	2,3,7,8-TCDD	ng/kg	0.2	0.6	9	0.0091	--	**0.52**	J	0.076	U	0.42	U	**1.8**	J	**1.7**		0.43	U	0.46	U	0.33	U	**3.3**		0.43	U
	2,3,7,8-TCDF	ng/kg	0.40658	--	--	0.77	--	**1.8**		0.032	U	0.28	U	**10**		0.28	U	0.37	U	0.24	U	0.19	U	0.19	U	0.25	U
SW9060A	Total organic carbon - average dup	mg/kg	--	--	--	--	--	6,000		10,000		5,800		39,000		14,000		13,000		2,500		4,100		5,500		1,100	J

Notes:

a The source of the CUL or RAL is the Portland Harbor Cleanup Level (CUL) or Remedial Action Level (RAL) of the Portland Harbor Record of Decision, dated 2017 and subsequent Explanation of Significant differences and errata. If the CUL or RAL is not available, the bolded SLV is used for screening.

[b] Chlordane is equal to the sum of all chlordanes (e.g. alpha and gamma-chorldane). Non-detects are not included in total. If both non-detects, use higher of the two detection limits for total.

[c] Development of Oregon Background Metals Concentrations in Soil – Table 4 – Regional 95% Upper Prediction Limit Default Background Concentrations for Metals in Soil – State of Oregon – Portland Basin, March 2013.

[d] Total DDx = The sum of detected 2,4' and 4,4' compounds. If non-detect, half the detection limit was used.

[e] cPAHs = Benzo(a)pyrene equivalent (BaP eq) values used to represent carcinogenic PAHs (cPAHs). cPAHs were calculated by multiplying the cPAHs by their respective equivalency factors (PEFs) and summing the resulting concentrations. If non-detect, half the detection limit was used.

Bold = value detected

Bold and shaded = detected result exceeds CUL.

Bold and Yellow = detected result exceeds applicable RAL (Sitewide)

µg/kg = micrograms per kilogram

mg/kg = milligrams per kilogram

ng/kg = nanograms per kilogram

NWTPH-Dx = Northwest TPH-diesel range

PCB = polychlorinated biphenyl

PAH = polycyclic aromatic hydrocarbon

DDx = dichlorodiphenyldichloroethane (DDD), dichlorodiphenyldichloroethene (DDE), and dichlorodiphenyltrichloroethane (DDT) compounds

TPAH = total polycyclic aromatic hydrocarbons

TPH = total petroleum hydrocarbons

Table A-3B. Bank Soil Sampling Results – Chevron Willbridge Terminal

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Method Group	Analyte	Units	Portland Harbor CUL[a]	*Location ID:*	S-3		S-4		S-5	
				Sample ID:	S-3		S-4		S-5	
				Sample Date:	12/17/2007		12/17/2007		10/8/2007	
				Background[b]	Sampling Results					
TPH	#2 Diesel (C10-C24)	mg/kg	91	--	1.57	U	1.81	U	14.8	U
	Motor oil (>C24-C36)	mg/kg	--	--	3.13	U	**6.26**		29.6	U
	Gasoline	mg/kg	--	--	**0.758**		**0.827**		4.46	U
VOCs	Toluene	µg/kg	--	--	**11.9**		**13.8**		5.0	U
	Xylenes (total)	µg/kg	--	--	24.9	U	28.8	U	5.0	U
	1,2,4-Trimethylbenzene	µg/kg	--	--	16	U	**18.8**		5.0	U
	1,2,5-Trimethylbenzene	µg/kg	--	--	8.86	U	10.3	U	5.0	U
Metals	Aluminum	mg/kg	--	--					**10,100**	
	Antimony	mg/kg	--	--						
	Arsenic	mg/kg	3	8.8	**1.18**		**5.97**		**4.66**	
	Barium	mg/kg	--	--	**41**		**117**			
	Beryillium	mg/kg	--	--						
	Cadmium	mg/kg	0.51	0.63	**0.187**		**0.168**		**0.609**	
	Chromium	mg/kg	--	--	**4.74**		**20.1**		**9.16**	
	Copper	mg/kg	359	34	**13.2**		**50.3**		**24.4**	
	Lead	mg/kg	196	79	**3.06**		**15.9**		**19.8**	
	Magnesium	mg/kg	--	--						
	Manganese	mg/kg	--	--	**216**		**412**		**417**	
	Mercury	mg/kg	0.085	0.23	0.0155	U	0.0187	U		
	Nickel	mg/kg	--	--					**9.74**	
	Selenium	mg/kg	--	--	0.404	U	0.494	U		
	Silver	mg/kg	--	--	0.0617	U	**0.114**		0.609	U
	Zinc	mg/kg	459	180	**28.8**		**82.7**		**83**	
Pesticides	4,4'-DDD	µg/kg	114	--	0.363	U	0.434	U	15.7	U
	4,4'-DDE	µg/kg	50	--	0.363	U	0.434	U	15.7	U
	4,4'-DDT	µg/kg	246	--	**0.616**		**1.99**		39.3	U
Phthalates	bis(2-ethylhexyl)phthalate	µg/kg	135	--	**38.7**		**145**		**149**	
PAHs	Anthracene	µg/kg	--	--	3.6	U	**4.64**		158	U
	Benzo(a)anthracene	µg/kg	--	--	**10.7**		**40.9**		158	U
	Benzo(a)pyrene	µg/kg	--	--	**12.4**		**42.8**		158	U
	Benzo(b)fluoranthene	µg/kg	--	--	**14.1**		**49.5**		158	U
	Benzo(g,h,i)perylene	µg/kg	--	--	**8.68**		**30.5**		158	U
	Benzo(k)fluoranthene	µg/kg	--	--	**11.3**		**42.4**		158	U
	Chrysene	µg/kg	--	--	**14.5**		**50.7**		158	U
	Dibenz(a,h)anthracene	µg/kg	--	--	3.6	U	**9.99**		158	U
	Fluoranthene	µg/kg	--	--	**25.6**		**83.3**		158	U
	Fluorene	µg/kg	--	--						
	Indeno(1,2,3-cd)pyrene	µg/kg	--	--	**8.52**		**31.1**		158	U
	Naphthalene	µg/kg	--	--						
	Phenanthrene	µg/kg	--	--	**8.65**		**30.9**		158	U
	Pyrene	µg/kg	--	--	**17**		**65.5**		158	U
	cPAHs (BaP eq)[c]	µg/kg	85	--	**18**		**65**		183	U

Notes:

[a] The source of the CUL is the Portland Harbor Cleanup Level (CUL) of the Portland Harbor Record of Decision, dated 2017. If the CUL is not available, the bolded SLV is used for screening.

[b] Development of Oregon Background Metals Concentrations in Soil – Table 4 – Regional 95% Upper Prediction Limit Default Background Concentrations for Metals in Soil – State of Oregon – Portland Basin, March 2013.

[c] cPAHs = Benzo(a)pyrene equivalent (BaP eq) values used to represent carcinogenic PAHs (cPAHs). cPAHs were calculated by multiplying the cPAHs by their respective equivalency factors (PEFs) and summing the resulting concentrations. If non-detect, half the detection limit was used.

Bold = value detected

Bold and shaded = detected result exceeds CUL.

Bold and Yellow = detected result exceeds applicable RAL (Sitewide)

Table A-3C. Bank Soil Sampling Results – Phillips 66 Willbridge Terminal

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Method Group	Analyte	Units	Portland Harbor CUL[a]	Location ID:	S-6		S-7		S-8		S-9		S-10	
				Sample ID:	S-6		S-7		S-8		S-9		S-10	
				Sample Date:	12/13/2007		12/13/2007		12/13/2007		12/13/2007		12/13/2007	
				Background[b]	Sampling Results									
TPH	#2 Diesel (C10-C24)	mg/kg	91	--	14.1	U	14.3	U	13.4	U	13.1	U	14	U
	Motor oil (>C24-C36)	mg/kg	--	--	28.2	U	28.6	U	26.7	U	26.2	U	28	U
	Gasoline	mg/kg	--	--	4.13	U	4.34	U	3.98	U	4.18	U	4.39	U
VOCs	Toluene	µg/kg	--	--	51.6	U	54.3	U	49.7	U	52.2	U	54.9	U
	Xylenes (total)	µg/kg	--	--	103	U	109	U	99.4	U	104	U	110	U
	1,2,4-Trimethylbenzene	µg/kg	--	--	103	U	109	U	99.4	U	104	U	110	U
	1,2,5-Trimethylbenzene	µg/kg	--	--	51.6	U	54.3	U	49.7	U	52.2	U	54.9	U
Metals	Aluminum	mg/kg	--	--	--		--		--		--		--	
	Antimony	mg/kg	--	--	--		--		--		--		--	
	Arsenic	mg/kg	3	8.8	**2.1**		**4.77**		**6.87**		**3.44**		**3.65**	
	Barium	mg/kg	--	--	**64.9**		**78.7**		**44.9**		**54**		**76.6**	
	Beryillium	mg/kg	--	--	--		--		--		--		--	
	Cadmium	mg/kg	0.51	0.63	**0.53**		**0.584**		**0.511**		**0.530**		**0.555**	
	Chromium	mg/kg	--	--	**11.8**		**11.2**		**11.3**		**11.7**		**10.8**	
	Copper	mg/kg	359	34	**18**		**21.1**		**14.2**		**13.1**		**14**	
	Lead	mg/kg	196	79	**8.75**		**16**		**17.3**		**10.9**		**14.5**	
	Magnesium	mg/kg	--	--	--		--		--		--		--	
	Manganese	mg/kg	--	--	--		--		--		--		--	
	Mercury	mg/kg	0.085	0.23	0.102	U	0.0839	U	0.0857	U	0.0803	U	0.0925	U
	Nickel	mg/kg	--	--	--		--		--		--		--	
	Selenium	mg/kg	--	--	0.530	U	0.584	U	0.511	U	0.530	U	0.555	U
	Silver	mg/kg	--	--	0.530	U	0.584	U	0.511	U	0.530	U	0.555	U
	Zinc	mg/kg	459	180	**39.7**		**62.5**		**74.2**		**74.6**		**70.6**	
Pesticides	4,4'-DDD	µg/kg	114	--	7.44	U	7.62	U	7.07	U	7.05	U	7.41	U
	4,4'-DDE	µg/kg	50	--	7.44	U	7.62	U	7.07	U	7.05	U	7.41	U
	4,4'-DDT	µg/kg	246	--	**9.44**		**26.4**		**14.5**		**12.9**		**24.5**	
Phthalates	bis(2-ethylhexyl)phthalate	µg/kg	135	--	**29.6**		**30.6**		**28.4**		**28.2**		**29.6**	
PAHs	Anthracene	µg/kg	--	--	14.8	U	15.3	U	14.2	U	14.1	U	**15**	
	Benzo(a)anthracene	µg/kg	--	--	14.8	U	**23.3**		14.2	U	14.1	U	**58.6**	
	Benzo(a)pyrene	µg/kg	--	--	14.8	U	**22**		14.2	U	14.1	U	**38.3**	
	Benzo(b)fluoranthene	µg/kg	--	--	14.8	U	**27.2**		14.2	U	14.1	U	**24.8**	
	Benzo(g,h,i)perylene	µg/kg	--	--	14.8	U	**20.3**		14.2	U	14.1	U	**19.9**	
	Benzo(k)fluoranthene	µg/kg	--	--	14.8	U	**20.3**		14.2	U	14.1	U	**31.4**	
	Chrysene	µg/kg	--	--	14.8	U	**35.6**		14.2	U	14.1	U	**31.4**	
	Dibenz(a,h)anthracene	µg/kg	--	--	14.8	U	**35.6**		14.2	U	14.1	U	**61.1**	
	Fluoranthene	µg/kg	--	--	14.8	U	**83.1**		**21.4**		14.1	U	**86.9**	
	Fluorene	µg/kg	--	--	--		--		--		--		--	
	Indeno(1,2,3-cd)pyrene	µg/kg	--	--	14.8	U	**17.7**		14.2	U	14.1	U	**19.3**	
	Naphthalene	µg/kg	--	--	--		--		--		--		--	
	Phenanthrene	µg/kg	--	--	14.8	U	**28.5**		14.2	U	14.1	U	**89.35**	
	Pyrene	µg/kg	--	--	14.8	U	**50.9**		14.2	U	14.1	U	**106**	
	cPAHs (BaP eq)[c]	µg/kg	85	--	17	U	**65**		16	U	16	U	**110**	

Notes:

[a] The source of the CUL is the Portland Harbor Cleanup Level (CUL) of the Portland Harbor Record of Decision, dated 2017. If the CUL is not available, the bolded SLV is used for screening.

[b] Development of Oregon Background Metals Concentrations in Soil – Table 4 – Regional 95% Upper Prediction Limit Default Background Concentrations for Metals in Soil – State of Oregon – Portland Basin, March 2013.

[c] cPAHs = Benzo(a)pyrene equivalent (BaP eq) values used to represent carcinogenic PAHs (cPAHs). cPAHs were calculated by multiplying the cPAHs by their respective equivalency factors (PEFs) and summing the resulting concentrations. If non-detect, half the detection limit was used.

Bold = value detected

Bold and shaded = detected result exceeds CUL.

Bold and Yellow = detected result exceeds applicable RAL (Sitewide)

Table A-3D. Bank Soil Sampling Results – McCall Terminal

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Group	Analyte	Units	Portland Harbor CUL[a]	Location ID:	GP-14		GP-15		GP-16		GP-17		GP-18		GP-19		RBC-1		RBC-2		RBC-3	
				Sample ID:	GP-14 0-2		GP-15 0-2		GP-16 0-2		GP-17 0-2		GP-18 0-2		GP-19 0-2		--		--		--	
				Sample Date:	12/13/2000		12/13/2000		12/13/2000		12/13/2000		12/14/2000		12/14/2000		11/10/2010		11/10/2010		11/10/2010	
				Sample Depth (feet):	0 to 2		0 to 2		0 to 2		0 to 2		0 to 2		0 to 2		--		--		--	
				Background[c]																		
TPH	Gasoline	mg/kg	--	--	10	U	10	U	10	U	10	U	10	U	10	U	5.59	U	5.33	U	5.02	U
	Diesel	mg/kg	91	--	**14**	F	10	U	10	U	**13**	H	**21**	H	20	U	9.33	F-0	39.6	F-0	**41.7**	U
	Residual Oil	mg/kg	--	--	55	F	**30**	Z	**49**	F	**84**	F	**210**	F	25	U	**195**		**653**		**549**	
	Total Petroleum	mg/kg	--	--	**69**		**30**		**49**		**97**		**231**				**204**		**693**		**549**	
Metals	Arsenic	mg/kg	3	8.8	**2.2**		**1.7**		**1.6**		**1.5**		**1.3**		**1.6**		**1.74**		**2.85**		**1.56**	
	Cadmium	mg/kg	0.51	0.63			NA		NA		NA		NA		NA		**0.206**		**0.629**		**0.276**	
	Chromium	mg/kg	--	--	**13**		**11**		**11**		**10**		**9**		**10**		**11.1**		**17.5**		**11.9**	
	Copper	mg/kg	359	34	**17**		**18**		**15**		**13**		**14**		**12**		**21.5**		**36.9**		**17.0**	
	Lead	mg/kg	196	79													**7.49**		**16.7**		**5.15**	
	Zinc	mg/kg	459	180													**61.7**		**190**		**68.1**	
PAHs	1-Methylnaphthalene	µg/kg	--	--	**5.8**		**7**		**7.5**		**32**		**1.2**									
	2-Methylnaphthalene	µg/kg	--	--	**0.6**	J	**1**	J	**1**	J	7.4	U	**0.5**	J	7.3	U	127	U	325	U	303	U
	Acenaphthene	µg/kg	--	--	**0.7**	J	**0.5**		7.6	U	7.4	U	7.6	U	7.3	U	63.5	U	162	U	152	U
	Acenaphthylene	µg/kg	--	--	**0.7**	J	**0.5**		7.6	U	7.4	U	7.6	U	7.3	U	63.5	U	162	U	152	U
	Anthracene	µg/kg	--	--	**0.9**	J	**2**	J	7.6	U	7.4	U	7.6	U	7.3	U	63.5	U	162	U	152	U
	Benzo(a)anthracene	µg/kg	--	--	**4**	J	**17**		**5**	J	**3**	J	**3**	J	**2**	J	**47.6**	J	162	U	152	U
	Benzo(a)pyrene	µg/kg	--	--	**6**	J	**24**		**5**	J	**4**	J	**4**	J	**2**	J	**55.5**	J	243	U	227	U
	Benzo(b)fluoranthene	µg/kg	--	--	**5**	J	**25**		**6**	J	**4**	J	**5**	J	**2**	J	**108**		**131**	J	**152**	J
	Benzo(g,h,i)perylene	µg/kg	--	--	**8**	J	**23**		**8**	J	**6**	J	**5**	J	**2**	J	**64.3**		**111**	J	758	U
	Benzo(k)fluoranthene	µg/kg	--	--	**5**	J	**22**		**6**	J	**3**	J	**4**	J	**2**	J	63.5	U	162	U	152	U
	Chrysene	µg/kg	--	--	**7**	J	**28**		**7**	J	**5**	J	**6**	J	**2**	J	**73.6**		162	U	152	U
	Dibenz(a,h)anthracene	µg/kg	--	--	**1**	J	**5**	J	**1**	J	**1**	J	**1**	J	**1**	J	63.5	U	162	U	758	U
	Fluoranthene	µg/kg	--	--	**6**	J	**34**		**8**	J	**5**	J	**6**	J	**2**	J	**57.8**	J	**89**	J	152	U
	Fluorene	µg/kg	--	--	7.5	U	**0.8**		7.6	U	7.4	U	7.6	U	7.3	U	63.5	U	162	U	152	U
	Indeno(1,2,3-cd)pyrene	µg/kg	--	--	**6**	J	**24**		**7**	J	**5**	J	**5**	J	**2**	J	**69.2**		**84.1**		758	U
	Naphthalene	µg/kg	--	--	7.5	U	**1**		**1**		7.4	U	7.6	U	7.3	U	127	U	325	U	303	U
	Phenanthrene	µg/kg	--	--	7.5	U	**13**		**3**	J	7.4	U	7.6	U	7.3	U	63.5	U	162	U	152	U
	Pyrene	µg/kg	--	--	**7**	J	**29**		**7**	J	**4**	J	**6**	J	**2**	J	**53.8**	J	162	U	152	U
	cPAHs (BaP eq)[d]	µg/kg	85	--	**8.56**		**35.85**		**7.87**		**6.24**		**6.35**				**142**		**233**		**539**	

Table A-3D. Bank Soil Sampling Results – McCall Terminal

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Group	Analyte	Units	Portland Harbor CUL[a]	*Location ID:*	GP-14		GP-15		GP-16		GP-17		GP-18		GP-19		RBC-1		RBC-2		RBC-3	
				Sample ID:	GP-14 0-2		GP-15 0-2		GP-16 0-2		GP-17 0-2		GP-18 0-2		GP-19 0-2		--		--		--	
				Sample Date:	12/13/2000		12/13/2000		12/13/2000		12/13/2000		12/14/2000		12/14/2000		11/10/2010		11/10/2010		11/10/2010	
				Sample Depth (feet):	0 to 2		0 to 2		0 to 2		0 to 2		0 to 2		0 to 2		--		--		--	
				Background[c]																		
PCBs	PCB-1016	µg/kg	--	--													8.02	U	7.77	U	7.71	U
	PCB-1221	µg/kg	--	--													8.02	U	7.77	U	7.71	U
	PCB-1232	µg/kg	--	--													8.02	U	7.77	U	7.71	U
	PCB-1242	µg/kg	--	--													8.02	U	7.77	U	7.71	U
	PCB-1248	µg/kg	--	--													8.02	U	7.77	U	7.71	U
	PCB-1254	µg/kg	--	--													**6.48**		**7.44**		7.71	U
	PCB-1260	µg/kg	--	--													8.02	U	7.77	U	7.71	U
	Polychlorinated biphenyls, total	µg/kg	9	--													**6.48**		**7.44**		7.71	U
Organochlorine Pesticides	Aldrin	µg/kg	2	--													8.86	U	17.5	U	9.22	U
	α - BHC	µg/kg	--	--													8.86	U	17.5	U	9.22	U
	β - BHC	µg/kg	--	--													8.86	U	17.5	U	9.22	U
	δ - BHC	µg/kg	--	--													8.86	U	17.5	U	9.22	U
	γ - BHC (Lindane)	µg/kg	5	--													8.86	U	17.5	U	9.22	U
	alpha - Chlordane	µg/kg	--	--													8.86	U	17.5	U	9.22	U
	gamma - Chlordane	µg/kg	--	--													8.86	U	17.5	U	9.22	U
	4,4'-DDD	µg/kg	--	--													8.86	U	17.5	U	9.22	U
	4,4'-DDE	µg/kg	--	--													8.86	U	17.5	U	9.22	U
	4,4'-DDT	µg/kg	--	--													8.86	U	17.5	U	9.22	U
	Dieldrin	µg/kg	0.07	--													8.86	U	17.5	U	9.22	U
	Endosulfan I	µg/kg	--	--													8.86	U	17.5	U	9.22	U
	Endosulfan II	µg/kg	--	--													8.86	U	17.5	U	9.22	U
	Endosulfan sulfate	µg/kg	--	--													8.86	U	17.5	U	9.22	U
	Heptachlor	µg/kg	--	--													8.86	U	17.5	U	9.22	U
	Heptachlor epoxide	µg/kg	--	--													8.86	U	17.5	U	9.22	U
	Methoxychlor	µg/kg	--	--													26.00	U	51.4	U	27.10	U
	Toxaphene (Total)	µg/kg	--	--													104.00	U	205.0	U	108.00	U
	2,4'-DDD	µg/kg	114	--													8.86	U	17.5	U	9.22	U
	2,4'-DDE	µg/kg	50	--													8.86	U	17.5	U	9.22	U
	2,4'-DDT	µg/kg	246	--													8.86	U	17.5	U	9.22	U

Table A-3D. Bank Soil Sampling Results – McCall Terminal

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Group	Analyte	Units	Portland Harbor CUL[a]	Location ID:	GP-14		GP-15		GP-16		GP-17		GP-18		GP-19		RBC-1		RBC-2		RBC-3	
				Sample ID:	GP-14 0-2		GP-15 0-2		GP-16 0-2		GP-17 0-2		GP-18 0-2		GP-19 0-2		--		--		--	
				Sample Date:	12/13/2000		12/13/2000		12/13/2000		12/13/2000		12/14/2000		12/14/2000		11/10/2010		11/10/2010		11/10/2010	
				Sample Depth (feet):	0 to 2		0 to 2		0 to 2		0 to 2		0 to 2		0 to 2		--		--		--	
				Background[c]																		
Miscellaneous Semivolatiles	3- and 4-Methylphenol	µg/kg	--	--	150	U	150	U	150	U	150	U	150	U	150	U	159	U	406	U	379	U
	Dibenzofuran	µg/kg	--	--	**0.6**	**J**	**0.8**	**J**	7.6	U	7.4	U	7.6	U	7.3	U	635	U	162	U	152	U
	Dimethyl Phthalate	µg/kg	--	--	15	U	**4**	J	**0.7**	**J**	**1**	**J**	**1**	**J**	**1**	**J**	635	U	1620	U	1520	U
	Diethyl Phthalate	µg/kg	--	--													635	U	1620	U	1520	U
	Di-n-butyl Phthalate	µg/kg	--	--													635	U	1620	U	1520	U
	Butyl Benzyl Phthalate	µg/kg	--	--													635	U	1620	U	1520	U
	Bis(2-ethylhexyl) Phth.	µg/kg	135	--													635	U	1620	U	1520	U
	Di-n-octyl Phthalate	µg/kg	--	--	150	U	150	U	150	U	150	U	150	U	0.8	J	1270	U	3250	U	3030	U

Notes:

[a] The source of the CUL is the Portland Harbor Cleanup Level (CUL) of the Portland Harbor Record of Decision, dated 2017, updated in 2018 and 2020.

[c] Development of Oregon Background Metals Concentrations in Soil – Table 4 – Regional 95% Upper Prediction Limit Default Background Concentrations for Metals in Soil – State of Oregon – Portland Basin, March 2013.

[d] cPAHs = Benzo(a)pyrene equivalent (BaP eq) values used to represent carcinogenic PAHs (cPAHs). cPAHs were calculated by multiplying the cPAHs by their respective equivalency factors (PEFs) and summing the resulting concentrations. If non-detect, half the detection limit was used.

Bold and shaded = detected result exceeds CUL.

Bold and Yellow = detected result exceeds applicable RAL (Sitewide)

Bold = value detected

µg/kg = micrograms per kilogram

DEQ = Oregon Department of Environmental Quality

F = Fingerprint of the sample matches elution pattern of calibration standard

H = Elution pattern indicates the presence of heavier weigh constituents

J = Estimated Concentration

mg/kg = milligrams per kilogram

NA = not analyzed

PAH = polycyclic aromatic hydrocarbon

PCB = polychlorinated biphenyl

PEC = probable effect concentration

SQV = sediment quality value

TPH = total petroleum hydrocarbons

U = Not detected at indicated quantitation limit

Z = Fingerprint does not resemble a petroleum product

Appendix B
Field Sampling Plan

Jacobs

Portland Harbor Superfund Site
Willbridge Cove Project Area Remedial Design

Pre-Design Investigation Field Sampling Plan

Final

June 10, 2021

Prepared for the Willbridge Cove Group

Prepared by Jacobs (Willbridge Cove RD Supervising Contractor)
Reviewed by EHS Support (Willbridge Cove RD Project Coordinator)
For Submittal to EPA Region 10

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Acronyms and Abbreviations

Acronym	Definition
°C	degree(s) Celsius
ASAOC	Administrative Settlement Agreement and Order on Consent
ASTM	ASTM International
bgs	below ground surface
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
Chevron	Chevron U.S.A. Inc.
dGPS	differential Global Positioning System
DOT	U.S. Department of Transportation
EPA	U.S. Environmental Protection Agency
ESD	Explanation of Significant Difference
FD	field duplicate
FSP	Field Sampling Plan
GIS	Geographic Information System
GPS	Global Positioning System
Jacobs	Jacobs Engineering Group Inc.
KMLT	Kinder Morgan Liquids Terminals, LLC.
McCall	McCall Oil and Chemical Corporation
MS	matrix spike
MSD	matrix spike duplicate
NAPL	non-aqueous phase liquid
NAVD88	North American Vertical Datum 1988
NGVD29	National Geodetic Vertical Datum of 1929

OHW	ordinary high water
PDI	Pre-Design Investigation
Phillips 66	Phillips 66 Company
PHSS	Portland Harbor Superfund Site
PID	photoionization detector
PPE	personal protective equipment
Pre-RD	Pre-Remedial Design
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
RM	river mile
ROD	Record of Decision
Shell	Shell Oil Company
SMA	sediment management area
SOP	Standard Operating Procedure
USACE	U.S. Army Corps of Engineers
USCS	Unified Soil Classification System
USGS	U.S. Geological Survey
WCPA	Willbridge Cove Project Area
WP	Work Plan

Note: If using a screen reader, adjustment to your default settings may be required.

1. Introduction

On behalf of the Willbridge Cove Group (Chevron U.S.A. Inc. [Chevron], Kinder Morgan Liquids Terminals, LLC [KMLT], McCall Oil and Chemical Corporation [McCall], Phillips 66 Company [Phillips 66], and Shell Oil Company [Shell]), Jacobs Engineering Group Inc. (Jacobs) has prepared this Field Sampling Plan (FSP) for the Pre-Design Investigation (PDI) Work Plan (WP) for the Willbridge Cove Project Area (WCPA) located within the Portland Harbor Superfund Site (PHSS) in Portland, Oregon. This FSP fulfills a portion of the requirements set forth by the U.S. Environmental Protection Agency (EPA) in the Administrative Settlement Agreement and Order on Consent (ASAOC) for Remedial Design for the WCPA (Comprehensive Environmental Response, Compensation, and Liability Act of 1980 [CERCLA] Docket No. 10-2020-0053), signed on January 31, 2020.

The Record of Decision (ROD) was issued by EPA in 2017 for the PHSS located in Portland, Oregon (EPA 2017). The PHSS covers an area along the Willamette River from approximately river mile (RM) 1.9 to RM 11.8.

1.1 Project Area Description

The WCPA borders the western shore of the Willamette River between RM 7.5 and 8.1 and is located in Portland's Northwest Industrial District (Figure B-1-1). Four adjacent and currently operating petroleum storage and transfer terminals are directly upland to the WCPA: KMLT Willbridge Terminal, Chevron Willbridge Terminal, Phillips 66 Willbridge Terminal, and McCall Terminal. Each facility has a dock for petroleum product transfer to/from marine transport vessels within the WCPA. In addition, Tidewater Barge Lines, Inc. leases the turning basin from McCall and parks spud barges in the area upstream side of the Phillips 66 dock. The properties where KMLT Willbridge, Chevron Willbridge, and Phillips 66 Willbridge Terminals (known collectively as Willbridge Terminals in some documents) operate are bisected by NW Front Avenue. The bulk storage facilities are inland of NW Front Avenue and are between NW Front Avenue and Highway 30.

Five preliminary sediment management areas (SMAs)[1] were identified for the WCPA in the ROD (Figure B-1-2). The river banks within the WCPA consists of a sloping bank with a beach area present below the ordinary high water (OHW) elevation in some locations. Large riprap is present along the majority of the river bank. The bank is steeper along the McCall shoreline. There are no ROD river banks identified in the WCPA.

1.2 Project Organization and Communication

Key project staff, their titles and responsibilities, and communication pathways are provided on Figure B-1-3. The Willbridge Cove Project Coordinator will notify the EPA Project Coordinator at least 28 days prior to sample collection activities per the ASAOC Statement of Work Section 5.6 (d)(5) so that EPA can schedule any planned oversight. The Project Manager will be responsible for coordinating EPA approval of deviations in the field through field change requests.

[1] The SMAs are described as "Explanation of Significant Difference (ESD) Areas Jan 2018" in the source files; map legends denote these as (ROD ESD 2018) accordingly.

2. Data Quality Objectives and Sampling Approach

The sampling design and data quality objective for the PDI are established in Section 3 of the WCPA PDI WP, to which this FSP is Appendix B. This section provides a summary of the samples that will be collected, the target locations, and the analytical schedule for each media. The sampling plan data quality objectives (DQOs) are presented in the PDI WP Tables 1-2, 1-3, and 1-4.

2.1 Sediment Sampling

The approach for sediment sampling is described in Section 3 the PDI WP and focuses on characterization of current conditions and refinement of selected preliminary SMAs within the WCPA. The sampling program includes two types of sediment samples: primary samples that will be analyzed immediately and step-out samples will be archived and then submitted for analytical testing pending the results of the primary samples.

Figures B-2-1 through B-2-4 provide the layout of the proposed sediment sampling locations and Table B-2-1 presents the target coordinates and associated analytical and archival schedule for the sediment samples. A total of 53 stations (including both primary and step-out locations) will be sampled to support further refinement of the SMA footprints. Surface grabs comprised of 3-point composites will be collected from all stations; sediment cores will be collected from 40 locations. Six of the surface grabs will be collected from under the docks using divers. Sediment sample collection is detailed in Section 3 of this FSP.

2.2 River Bank Soil Sampling and Field Data Collection

The approach for the physical characterization of the river bank is consistent with the guidelines provided in Section 2.3 of *Guidance for River Bank Characterizations and Evaluations* (EPA 2019b, Appendix D). Bank surface soil will be collected at locations along the bank adjacent to SMA 4. Sample locations are shown on Figure B-2-5. Table B-2-2 presents the target coordinates and associated analytical and archival schedule for the river bank soil samples. Bank soil samples will be collected continuously on 1-foot (30-centimeter) sampling intervals to a depth of 5 feet. The surface intervals will be analyzed immediately, and subsurface samples will be archived. A total of 12 locations will be attempted during the PDI.

3. Sediment Sampling and Sample Collection Procedures

This section describes the proposed sediment sampling collection procedures. The data will be collected using field sampling and analytical methods considered with previous EPA-approved sampling investigations at the PHSS such as the remedial investigation phases of sampling and 2018 Pre-Remedial Design (Pre-RD) sampling plans. The procedures established during the Pre-RD sampling have been incorporated into the following sections (AECOM and Geosyntec 2018a, 2018b). The Standard Operating Procedures (SOPs) referenced herein are broad in scope to address multiple types of projects; only the procedures identified in this project-specific FSP are applicable.

3.1 Sampling Vessels and Equipment

It is expected that most sediment sampling will be conducted from an appropriately sized and outfitted research marine vessel, or equivalent, operated by a qualified subcontractor.

Surface sediment samples will be collected using a power-grab sampler, Van Veen sampler or similar mechanical grab, or with hand equipment (if low water level). Subsurface sediment samples will be collected using vibracoring methods. Some samples nearest the shoreline may also be collected on foot using a “backpack” vibracore unit, or equivalent. The approach for collecting the samples will be made by the field team and selected subcontractor based on field conditions and health and safety considerations.

In addition to the vessel, the following sampling equipment may be required for collection of sediment samples:

- Surface sediment grab sampler capable of collecting sediment to a depth of 1 foot
- Sediment vibracore sampler capable of recovering 15-foot core samples, tubing, and core catchers
- Large plastic tub
- Differential Global Positioning System (dGPS) unit
- Weighted tape measure and/or depth sounder
- Plastic sheeting and duct tape
- Field table
- Electric double cut shears or circular saw for cutting open the core tubes
- Straight bladed knife and cut resistant gloves
- Measuring tape (engineering scale)
- Dry erase board and marker
- Digital camera
- Munsell color chart
- Sample containers
- Cooler with ice
- Personal protective equipment (PPE) (e.g., gloves, safety glasses, and face shields)
- Disposable, dedicated single-use pans and scoops (either stainless-steel bowls and utensils, or aluminum pans and high-density polyethylene scoops)
- Photoionization detector (PID)

- Ziploc-type bags
- Deionized water
- Paper towels
- Duct tape
- Large garbage bags
- Water-resistant field notebooks
- Sample data sheets (core logging forms) and field log sheets
- Chain-of-custody forms and seals
- Sample labels
- Lithology identification guide
- Pens (water-resistant pen, permanent pens, paint marker, etc.)
- Toolbox (wrench, field knife, etc.)
- Ice

3.2 Positioning, Horizontal and Vertical Control

Planned sampling location coordinates will be entered into the sampling vessel's onboard Global Positioning System (GPS) unit. Sediment sampling locations will be adjusted in the field if riprap or other debris is encountered that will preclude core collection. Side-scan sonar and/or an underwater camera may be used to locate structures or surface materials that may impede sediment sampling.

Sampling stations will be located in the field using a dGPS unit with submeter accuracy. As-sampled locations will be measured at the time of collection (SOP-4). Sampling station coordinates (X, Y) will be reported in Oregon State Plane (feet) to the nearest foot relative to the North American Datum of 1983 and will be entered into the project database. The accuracy of the dGPS will be verified daily against a surveyed benchmark. The PH-1 or PH-2 benchmark installed as part of the Pre-RD investigation in Swan Island Lagoon close to the boat launch ramps (AECOM and Geosyntec 2018a, 2018b) may be used if they are conducive to the daily operations. If benchmarks within the project work area are not readily available, additional benchmarks may be placed by a licensed surveyor in the vicinity. Approval from EPA will be obtained prior to using any new benchmarks.

Vertical control will be established using an onboard fathometer or lead line (or weighted tape) to measure depth to mudline (water surface to sediment surface) at sample locations at the time of collection. The fathometer accuracy will be checked regularly by vessel contractor and calibrated when necessary following ASTM International (ASTM) D6318 Standard Practice for Calibrating a Fathometer Using a Bar Check Method or other similar practice. Vertical position control at each location will be evaluated by using a lead line (or weighted tape) to measure from the water surface to the sediment surface if a fathometer is not available or is not positioned on the vessel to provide an accurate measurement from where the sample is collected. Depths will be recorded to the nearest one-tenth of a foot. Mudline elevations will be recorded based on North American Vertical Datum of 1988 (NAVD88). If available, real-time kinematic dGPS will be used to record elevations. The measurement time and tide stage from the U.S. Geological Survey (USGS) Willamette River Gauge located at the Morrison Bridge in

Portland Oregon (USGS Gauge 14211720)[2] will also be utilized to determine river elevation. This gauge presents data in North Geodetic Vertical Datum of 1929 (NGVD29). The tide stage will be recorded when the depths are measured. If mudline elevations are calculated based on water depth and tidal corrections, the mudline elevation will be converted to NAVD88. The conversion factor is approximately NAVD88 = NGVD29 +3.5 feet (USACE 2014).

3.3 Sediment Grab Sampling

Grab samples located under docks will be collected as three-point composites using divers and hand collection equipment (scoops, trowels, or other means). Surface samples in areas not under the docks will be collected after the coring has been performed in order to center surface samples around locations where acceptable cores were able to be collected. Surface sediment samples that are not under docks will be collected as three-point composites within a 25-foot target radius of the accepted core or target location, if practicable. In instances where specific locations are to be reoccupied, all reasonable efforts will be made to get as close as practicable to the target location (this is applicable to target locations PD03, PD14, and PD18).

The surface interval will be collected using a power-grab sampler deployed from a marine vessel (SOP-8). The target depth for sample collection is from 0 to 1 foot (0 to 30 centimeters) below the mudline. If this cannot be achieved, a minimum penetration of 20 centimeters below the mudline will be acceptable in areas with soft substrate material.

Before collecting sediment samples, a clean plastic-covered work space will be established on the work platform. Sample bottles will remain covered in plastic bags inside the plastic-lined ice chests until ready to be filled.

Sediment probing with a rigid steel-rod or weighted tape may be conducted prior to sampling to assess substrate conditions. Sediment samples collected with the grab sampler will be acceptable if the following criteria are met (EPA 2001):

1) The sampler is not over-filled, so the sediment surface is not pressed against the top of the sampler.
2) Overlying water is present (indicates minimal leakage). This can be very difficult to achieve in areas where gravel or larger material are present, in which case, the field team leader will need to use professional judgement to determine acceptability of the sample. Decisions will be documented in the field notes.
3) The overlying water is not excessively turbid (indicates minimal sample disturbance).
4) The jaws are closed and there are no visual signs of winnowing or leaking from the sample device.
5) The necessary penetration depth is achieved (for example, several centimeters more than the targeted sample depth).

Surface samples will be collected from 0 to 30 centimeters, depending on sediment substrate conditions. Multiple attempts may be necessary if the three grab subsamples contain an insufficient volume of sediment for all required analyses.

Sediment from the central portion (i.e., not in contact with the sides of the sampler) of each of the subsample grabs will be collected to a depth of 30 centimeters (if available) using a clean stainless-steel spoon or scoop and transferred to a decontaminated, clean stainless-steel compositing bowls (or

[2] U.S. Geological Survey (USGS). 2020. National Water Information System: Web Interface. August 27. https://waterdata.usgs.gov/or/nwis/uv/?site_no=14211720&PARAmeter_cd=00065,00060.

single-use aluminum foil pans). If necessary, material from multiple grabs at the same location can be combined to obtain sufficient volume for all required analyses. The sediment from each sub-sampled grab will be covered with clean foil and placed out of direct sunlight until the composite can be generated.

Once sufficient sediment has been obtained for all scheduled analyses, equal volumes of sediment from each of the three subsamples will be combined and homogenized until a uniform color and texture is achieved. This composite material will be containerized and submitted for analysis.

If probing or sampling activities encounter conditions that impede surface sediment grab sampling, the contingency plan presented below for such cases has been adapted from the Pre-RD FSPs developed by AECOM and Geosyntec (2018a). The following conditions are potentially present:

- Soft sediment—Minimum of 15 centimeters recovery depth in each accepted grab.
- Soft sediment with debris—Debris may include wood, metal, trash, or concrete and it may be challenging to consistently achieve the minimum recovery depth.
- Soft sediment on slope—Steeply sloped areas (such as near the navigation channel boundary) can impede grab sample collection.
- Natural hard sediment—Dense sands and gravels, cobbles, and stiff silts or clays can result in minimal grab penetration and recovery.
- No recoverable sediment—Areas covered by riprap or dense cobble, or in areas that cannot be sampled. The field team will make three grab attempts at such locations and confirm the bottom conditions using a probe rod. The conditions will be documented in the field records.

If conditions described in the second, third, and/or fourth bullets are encountered, the field team will perform no more than six total attempts within 25 feet of the primary target to obtain composite samples; the three best (meeting acceptance criteria) or deepest penetration depths will be accepted for compositing. If after six attempts only two acceptable grabs are collected, the two grabs will be composited and the conditions impeding sample collection will be documented in the field notes. If fewer than two acceptable grabs are obtained from a location, the location will not be sampled.

Sediment will be visually classified according to the ASTM Visual-Soil Classification Method and Sediment Sampling Logging Key. A summary of the method is included in SOP-8. Sediment grab sampling activities will be recorded on the Surface Sediment Sampling Field Form (SOP-8).

Samples will then be thoroughly homogenized to a uniform appearance using a decontaminated, clean, stainless-steel spoon, spooned into appropriate clean containers as identified in the Quality Assurance Project Plan (QAPP) (Appendix C of the PDI WP). Samples will be transported in coolers on ice to the field lab for packaging and shipment to the analytical laboratory for analysis or archiving. Any remaining excess samples will be disposed of and the sampling equipment decontaminated before reuse.

3.4 Sediment Core Collection

Sediment cores will be collected with a vibracore or similar method that is able to meet the acceptance criteria and target depth outlined in this section. The target core depth is up to 15 feet or core refusal (target core depths are included in Table B-2-1). Vessel maneuvering, positioning, and the sediment core collection will be performed by the qualified subcontractor in accordance with their standard procedures and the specifications detailed in the scope of work.

In general, sediment coring will follow these steps:

1) Where possible, the target sampling area will be probed with a thin metal rod to determine if debris or rocks are present before attempting to collect a core. If the probing suggests the bottom type will preclude core collection, the station will initially be offset up to 25 feet; however, if obstructions are present, coring locations may be further offset as long as they remain within the grid cell, to attempt to find an area that can be effectively sampled. In instances where specific locations are to be reoccupied, all reasonable efforts will be made to get as close as practicable to the target location (this is applicable to target locations PD03, PD14, and PD18).
2) Core tube caps will be removed immediately before placement into coring device to minimize potential core contamination.
3) GPS position will be recorded when the vibracore first rests on the sediment surface.
4) The vibracore will be advanced without power (under its own weight), then vibration will be applied until the core tube is advanced to the target depth or refusal.
5) After a brief pause, the core tube will be extracted from the sediment using only the minimum vibratory power needed for extraction.
6) As soon as the core tube daylights to the surface water/air interface, a bottom cap may be placed over the tube to prevent material loss out of the core catcher.
7) Exterior sidewalls of the core tube will be inspected for signs of potential non-aqueous phase liquid (NAPL) and scrapes or scoring of the core tube walls from contact with dense gravel.
8) The core will be accepted, rejected, or stored on the vessel based on the acceptance criteria described in Section 3.4.1. Depending on the result, additional drives may be attempted, as described in Section 3.4.1.
9) After core acceptance, water will be carefully decanted or siphoned from the top of the core tube to minimize sediment disturbance.
10) Sediment cores will be capped and labeled to clearly indicate the core location and top/bottom of the core. To the extent possible, manipulation and handling of the core will be minimized. If vibracores longer than 5 feet are collected, they may be segmented into manageable lengths (e.g., 4 to 5 feet) capped, tapped, and labeled (location ID; a top/bottom designator; and indication of upper, middle, or lower core segment). Cores will be stored as upright as possible on the vessel until they are transferred to the processing team. If the cores are not able to be opened and processed shortly after they are transferred to the processing area, the cores will be held chilled (for example, using ice, refrigeration, or other means).
11) Core processing will be as described in Section 3.4.2.

The following information at a minimum will be recorded in the field logbook or on project-specific field forms by the project team representative on the vessel:

- Time and date of core collection
- Weather conditions
- Location coordinates (as sampled)
- Surface water elevation (NAVD88)
- Water depth
- Calculated sediment surface (mudline) elevation (NAVD88)
- Core recovery

- Note any problems with core collection (e.g., number of attempts needed)
- Note any debris in area
- Note where signs of NAPL begin and end (including olfactory, visual, and MultiRAE/PID observations)
- Description of coring performance (e.g., free fall, refusal, etc.)
- Inspection of the core catcher for signs of refusal and blocking
- Any other pertinent information

3.4.1 Core Acceptance Criteria and Contingency Plans if Field Conditions Impede Core Collection

Sediment cores collected will be acceptable if the following criteria are met:

- Overlying water is present, and the sediment surface is intact.
- Core has at least 80 percent recovery versus penetration. Percent recovery is recovered thickness/penetration.
- Core tube is in good condition (not excessively bent).
- Target penetration depth has been achieved unless difficult site conditions are encountered.

During the coring efforts, the field crew may encounter field conditions that preclude collection of acceptable cores at the target location (for example, limited access, poor recovery, safety concerns, and debris/rock/bedrock causing refusal). No more than three attempts will be made to relocate the core within a 25-foot radius of the planned location, if accessible. If not accessible (i.e., under a dock/pier/vessel or shallow water depth), the target radius will be increased for sample collection. If the first core attempt meets the acceptance criteria, no additional cores will be collected at that station. If the first attempt does not meet the criteria, up to two additional cores will be attempted and retained (stored on vessel deck). The best (deepest or longest core meeting acceptance criteria) of three attempts will be retained and processed. The conditions preventing a minimum of 80 percent recovery will be documented.

If utilities run within 50 feet of a proposed coring location based on review of Geographic Information System (GIS) maps and confirmed in the field with "utility crossing" signage, the coring location will be adjusted to a suitable location (using best professional judgement and consultation with the PDI Project Manager) so that the location is at least 50 feet away from the presumed utility crossing and noted in the field notebook.

3.4.2 Core Processing and Subsurface Sample Collection

It is expected that cores will be transferred to a shore-based, or possibly a barge-based, location for characterization and sub-sampling. Cores will be stored as upright as possible and chilled until processing. To the extent practicable, cores will be processed and sampled within 48-hours of collection. Briefly, cores will first be field screened (visual, olfactory, and MultiRAE or PID) and logged (SOP-5a). Samples will be collected from the sediment cores on continuous 1-foot intervals using a decontaminated, clean, stainless-steel spoon; transferred into the appropriate containers as identified in Table B-5-1; and sent to the laboratory for analysis. The surface (0 to 30 centimeter) sampling interval is represented by the co-located sediment grab samples.

Subsurface sediment core processing at the shore-based field laboratory will follow these steps:

1) The processing tables will be prepared by covering with plastic sheeting and securing with heavy duty tape (e.g., duct tape). All core segments from a location will be placed on the table so that the top of the core is to the left and the bottom is to the right and the entire core is represented.
2) The core tube will be split open to preserve the material stratigraphy inside the core tube using a table saw, hand-held circular saw, radial saw, shearing tool, or similar device. The core material will also be split so that a fresh sediment surface that has not been in contact with the core liner will be screened, logged, and sampled.
3) PID readings will be collected from each 1-foot interval (SOP-6). The readings will be collected from in the ambient air immediately above the mid-point of the sampling interval unless there is an area within the interval with a notable odor or staining. The interval from which the reading was collected will be noted on the core log. If elevated PID readings are noted during core processing, then an additional reading may be taken from the headspace of that interval; material will be placed in a sealed plastic bag that is allowed to equilibrate for several minutes before obtaining the reading.
4) Cores will be photographed before sampling. Digital photographs of each core segment will be taken to visually document the undisturbed core structure.
5) The sediment cores will be visually characterized for sediment type, color, moisture content, texture, grain size and shape, sheen/odor, consistency, visible evidence of staining, and any other observations. Sediment cores will be visually described following Unified Soil Classification System (USCS) (modified slightly for sediment characterization) based visual-manual identification in accordance with the ASTM D2488-17e1 standard practice. A logging key of the visual classification method is provided in SOP-5a, following ASTM 2017. The colors will be designated using a Munsell color chart. Sediment logs will be recorded on the Core Log Field Form (SOP-5b).
6) Olfactory observations (odors) or positive responses to an organic vapor detector will be recorded on the sediment core log (SOP-5b). Examples of odors may include, but are not limited to, sulfur/sulfide, and petroleum-type odors.
7) If potential sheen or NAPL is observed, descriptions detailed in SOP-5 must be used to record field observations.
8) Subsurface sample intervals will be 1-foot (30 centimeter) intervals based on the recovered measured material in the core tube. Corrections for recovery will be applied during data post-processing.

After the cores have been described and sample intervals determined, sediment from each interval will be scooped from the core interval into a decontaminated stainless-steel bowl (or single-use aluminum foil pan if metals are not a target analyte), homogenized within each sample interval until uniform in color and texture, and then placed into appropriate sample containers for laboratory analysis (SOP-5 and SOP-7). The samples will be field screened to remove gravel-sized material and unrepresentative material (e.g., woody debris) before field processing. Samples will then be thoroughly homogenized to a uniform appearance using a decontaminated, clean, stainless-steel spoon and then spooned into appropriate clean containers as identified in the QAPP (Appendix C of the PDI WP). Sample jars will be labeled, packed, and then transported in coolers on ice to the field lab for packaging and shipment. Any remaining excess samples will be properly disposed of in refuse containers and the sampling equipment decontaminated before reuse.

4. River Bank Field Data and Sample Collection Procedures

This section describes the proposed field observational data and river bank soil sampling collection procedures. The field teams will carefully and completely document the as-sampled location information for incorporation into the project GIS database.

4.1 Field Observations

The erodibility of river banks within the WCPA will be evaluated qualitatively.

The following physical and material characteristics of the river bank to be sampled will be collected by the field team (see SOP 3a and SOP13b) to support a quantitative evaluation if needed:

- **Height of the bank**—River bank height is measured from the top of the bank, defined as the point where the slope of the land surface changes from toward the river to toward the uplands, to the toe of the slope, defined as the first significant break in slope that is below OHW but above mean high water. Bank height can be difficult to determine in the field and should be measured using survey methods. Bank height may instead be determined from topographic and bathymetric maps.
- **Bankfull level**—Bankfull level is the point on the river bank that contains normal non-flood-level flows of the river throughout the year and is typically identifiable by visible changes in topography, vegetation type, or sediment grain size (EPA 2019a). For the Willamette River, the bankfull level is approximated by the OHW elevation, which is equivalent to 20.08 feet NAVD88 (USACE 2014).
- **Bank angle**—The maximum bank angle between the toe of the slope and the top of the bank will be determined from topographic or bathymetric maps or measured in the field using a clinometer or other suitable direct field measurement technique.
- **Surface protection**—Surface protection is the amount of the river bank that is covered and protected by woody debris, rooted vegetation, embedded boulders, revetment, bedrock, or other embedded materials that protect the bank from erosion (EPA 2019a). River bank surface protection will be assessed in the field and visual observations recorded, including:
  - Type and size of protection
  - Location relative to the toe of the slope, top of the bank, and OHW elevation
  - Percent of the river bank surface protected, general condition of protection, if the protection is afforded by armoring
  - General condition of the armoring should be noted to determine whether the material is stable, unstable, or sloughing into the river
  - If the river bank is vegetated, include the general vegetation type and the percent of the river bank surface that is covered by vegetation. The vegetation types will be used to infer rooting depth and density.
- **Bank composition/soil type**—Bank composition is the soil types that comprise the river bank. River bank soil types will be determined in the field through visual inspection and by field classification methods. The USCS determined by ASTM Standard D2487-17 will be used to describe the soil types. Field classifications will be performed using the procedures described in ASTM Standard D2488-00, Standard Practice for Description and Identification of Soils (ASTM D2488). Soil samples may be collected for sieve analysis to supplement visual classification and verify grain size distribution.

4.2 River Bank Soil Sampling Chemical Characterization

The WCPA river banks will be accessed by field staff via upland access. All areas are accessible through the facility properties and crews will follow the sign-in/sign-out protocols for the respective facilities.

A total of 12 locations are targeted for sample collection at low water level using hand augering field techniques. If all locations can be sampled successfully to the planned depth, a total of 12 surface soil samples and 48 subsurface soil samples will be collected.

Before collecting soil samples, a clean plastic-covered work space will be established on the ground surface. Sample bottles will remain covered in plastic bags inside the plastic-lined ice chests until ready to be filled. Shallow soil borings will be advanced to a depth of 5 feet vertically below ground surface (bgs) into the bank, perpendicular to the ground surface, and samples will be collected continuously on 1-foot (30-centimeter) sampling intervals (SOP-11). Surface soil samples will be collected using decontaminated (or single-use) hand tools. Decontamination procedures are provided in SOP-9. The top layer of vegetation and gravel (if any) will be temporarily removed in an area at least 6 inches in diameter. If riprap or other surface obstructions are present at a target location, the location will be moved to the nearest accessible location within 50 feet in either direction, along the target elevation contour.

PID readings (SOP-6), photographs (SOP-3), and lithologic descriptions (SOP-12) will be recorded for each sample and boring location. This process will be continued bgs. Soil samples will be visually characterized for sediment type, color, moisture content, texture, grain size and shape, sheen/odor, consistency, visible evidence of staining, and any other observations. Samples will be visually described following USCS based visual-manual identification in accordance with the ASTM D2488-17e1 standard practice. A logging key of the visual classification method is provided in SOP-12c, following ASTM 2017. The colors will be designated using a Munsell color chart. Borehole logs will be recorded on the Soil Boring Log Field Form (SOP-12).

Olfactory observations (odors) or positive responses to an organic vapor detector will be recorded on the soil boring log (SOP-12). Examples of odors may include, but are not limited to, sulfur/sulfide, and petroleum-type odors. If potential sheen or NAPL is observed, descriptions detailed in SOP-12 must be used to record field observations. The depth where signs of NAPL begin and end (including olfactory, visual, and MultiRAE/PID observations) will be noted in the boring log.

Hand augers will be decontaminated between sampling intervals. Upon completion of sampling at each boring location, the hole will be backfilled with native material and topsoil, vegetation, or armoring will be replaced to the extent possible.

If refusal is met, sample locations will be relocated. Up to three attempts will be made to relocate soil borings.

The following sampling equipment may be required for collection of river bank soil samples:

- Hand auger with extensions capable of reaching a depth of 5 feet bgs
- Large plastic tub
- Stainless-steel pan
- Stainless-steel spatula
- dGPS unit
- Plastic sheeting and duct tape
- Field table

- Measuring tape (engineering scale)
- Survey stakes
- Dry erase board and marker
- Digital camera
- Soil boring log
- Munsell color chart
- Sample containers
- Cooler with ice
- PPE (e.g., gloves, safety glasses, and face shields)
- Disposable, dedicated, single-use pans and scoops (either stainless-steel bowls and utensils, or aluminum pans and high-density polyethylene scoops)
- MultiRAE/PID
- Ziploc-type bags
- Decontamination equipment, including deionized water, alconox, brushes, buckets
- Paper towels
- Duct tape
- Large garbage bags
- Water-resistant field notebooks
- Sample data sheets and field log sheets
- Chain-of-custody forms and seals
- Sample labels
- Lithology identification guide
- Pens (water-resistant pen, permanent pens, paint marker, etc.)
- Toolbox (wrench, field knife, etc.)
- Ice

5. Analytical Requirements and Field Quality Control

The sediment and river bank samples anticipated to be submitted for analysis according to the schedules presented in Tables B-2-1 and B-2-2, respectively. Tables B-5-1 and B-5-2 also present the analytical methods, holding times, sample volumes, sample containers, and preservatives required by the QAPP (Appendix C of the PDI WP). Additional details related to the laboratory quality assurance/quality control (QA/QC) requirements are provided in the QAPP.

QC samples will be collected to assist in determining data quality and reliability. QC samples will be collected using the same procedures and immediately following collection of the target samples. Field QA and QC samples for chemical and physical analyses on environmental media will be collected at the following frequencies:

- **Field duplicates:** Five percent (1 per 20 primary field samples). A field duplicate is an independent sample collected as close as possible to the original sample from the same source and is used to assess sampling precision. Field duplicates will be labeled in accordance with the procedures outlined in Section 6.2 and packaged in the same manner as normal samples. Each duplicate will be taken using the same sampling and preservation method as other samples.
- **Matrix spike/matrix spike duplicate (MS/MSD) (collect triple sample volume):** Five percent (1 per 20 primary field samples for bulk sediment). An MS is an aliquot of a sample spiked with a known concentration of a target analyte(s). An MS analysis provides a measure of the method accuracy. An MSD is a laboratory split sample of the MS and is used to determine the precision of the method. Twice the normal sample volume will be collected for these QC samples. Laboratory QC samples will be labeled as such (i.e., MS/MSD) on sample bottles and chain-of-custody forms.
- **Temperature blanks:** One per cooler (temperature blanks should be provided by laboratory). Temperature blanks provide a means of verifying that samples have been maintained at the proper temperature (0 to 6 degrees Celsius [°C]) following collection and during transport to the laboratory. The laboratory will supply the temperature blanks as part of each bottle order request (to be returned with the batch of samples).
- **Equipment blanks:** Blanks will be collected for single use and reusable sampling equipment that is in direct contact with environmental media for each matrix sampled and will be collected at a rate of one per week per matrix per equipment. Equipment blanks are collected by passing Type II (deionized) water over sampling equipment and collecting the rinsate into laboratory provided containers. Container and preservation requirements for blanks are provided in Table B-5-2.

6. General Field Procedures

This section describes the general field procedures to be followed during the PDI field activities. The SOPs and field forms are provided in Attachment 1.

6.1 Record Keeping

A written record of sampling activities and field observations will be maintained in a bound, water-resistant field notebook with consecutively numbered pages. Entries will be legibly written in black or blue, indelible ink. Entry errors will be corrected by drawing one solid line through the incorrect entry, followed by the user's initials and date. The end of each workday or task will be signed and dated by the individual making the entries. Factual and objective language will be used. Entries will be complete and accurate enough to allow reconstruction of each field activity. Activities should be recorded contemporaneously. When not in use, the logbook will be stored in the permanent project file. After completion of the sampling activities, the field notebooks will be kept in the custody of the Jacobs Project Manager. Additional guidance is provided in SOP-2.

Daily entries of the following information will be recorded in the logbook when applicable:

- Date and time, expressed in 24-hour format
- Time of arrival and departure from the site
- Meteorological and water conditions (including tidal conditions)
- Project personnel and subcontractor personnel onsite
- Any visitors onsite, their representative company, and their level of protection
- Health and safety hazards and precautions
- Level of personal protection
- Field observations
- Task start/stop times
- Time of each entry
- Daily survey control checks
- Duration of sampling activities
- Site identification (visual sketches where appropriate)
- Location of sampling points (visual sketches where appropriate)
- Description of sample
- Sample ID and analyses to be completed
- Number of samples taken
- Time of sample collection
- QA and QC samples taken
- Type of field instrumentation (if any)
- Names of people collecting samples
- Water depth per station location
- Comments on sampling (e.g., equipment or sampling difficulties)
- Volume of sample return
- Decontamination procedures
- Equipment calibration records and all calibrations conducted
- Any other field instruments, general observations, or notes
- Any deviations from the sampling plan or sampling protocol, if any
- Health and safety observations
- Signature of recorder

6.1.1 Photographic Logs

Digital photographs will be taken in the field to document representative sampling locations, collected samples, site conditions, and any other salient site-related observations. A photographic log will be kept in which the date, location, photo ID number, brief photographic description, direction the photographer is facing (if appropriate), and if sheen was produced during collection of sediment samples will be recorded.

Photographs and relevant log information will be downloaded onto a field computer on a regular basis. Additional guidance is included in SOP-3.

6.1.2 River Bank Soil Sample Logs

River bank soil sampling information will be recorded on project-specific forms, which include the following:

- Locations of all sample attempts
- Sample depth
- Field screening results
- Description of material encountered, including texture, color, presence of debris, presence sheens or odors
- Documentation of samples submitted to the laboratory

Additional guidance is included in SOP-12a and SOP-12b.

6.1.3 Sediment Grab Sample Logs

Sampling information for sediment surface grab samples will be recorded on a project-specific form, which will include the following details:

- Location of all grab sample attempts
- Sediment thickness recovered within each grab
- Gross characteristics of the sediment, such as texture, color, biological structures, presence of debris, presence of sheens or odors
- Comments on sample cohesiveness
- Documentation of grab acceptance criteria and which grab samples are included in the station composite that is submitted to the analytical laboratory

  Additional guidance is included in SOP-8.

6.1.4 Sediment Core Logs

Sediment core logs will be recorded using project-specific forms in the field for the following observations and information:

- Sampling location, water depth, tide stage (or water surface elevation)
- Sediment sampling depth (recovered depth and driven length) for each sample and the associated percent recovery
- Gross characteristics of the sediment, such as texture, color, biological structures, presence of debris, presence of sheens or odors

- Comments on sample cohesiveness
- A summary of the sample collected from each core and analyses for which they were submitted

Additional guidance is included in SOP-5a and SOP-5b.

6.2 Sampling Management

Sample management will be performed in accordance with SOP-7 and the procedures are detailed in the following subsections.

6.2.1 Sample Identification

Field sample identifier nomenclature will include the following information:

- Project Area ID = WCPA (Willbridge Cove Project Area)
- Matrix ID =
  - SG for sediment grab sample
  - SC for sediment core sample
  - SB for soil boring sample
- Station ID (defined in Tables B-2-1 and B-2-2) = station IDs preceded by "PD" to designate PDI activities
- Subsurface samples depth interval = up to seven digits (X.X-X.X) for depth in feet (for example, a sample collected from 2 to 3 feet would be denoted 2.0-3.0)

An example ID is WC-SCPD-105—3.0-4.02, which notes a sediment core sample collected from coring location 105, from 3 to 4 feet below the mudline. Sediment sample depths do not need to be included in the sample ID for surface grab samples.

QC sample nomenclature is as follows:

- Field duplicate (FD) = WCPA-SDPD105—3.0-4.02FD
- MS = WCPA-SDPD105—3.0-4.02MS
- MSD = WCPA-SDPD105—3.0-4.02MSD
- Trip blank (TB) = TB-Date-01
- Equipment blank (EB) = EB-Date-01

If more than one duplicate, trip blank, or equipment blank is collected in a given day, sequential numbering will be used for the suffix of the ID (for example, EB-081020-01, EB-081020-02). There is a 30-character limit on sample identifiers.

All samples will be placed on ice immediately and will be packaged and shipped in a manner consistent with SOP-7.

6.2.2 Sample Shipment

Soil and sediment samples, and all associated QA/QC samples will be shipped to the yet-to-be determined analytical laboratories. To minimize the potential for sample degradation and to maintain a temperature from 0 to 6°C, sediment samples will be chilled in a cooler with ice in resealable plastic bags. The chain-of-custody form and a QA sample form, if required, will be filled out in indelible ink, placed in a

resealable plastic bag, and taped to the inside lid of the shipping cooler. The following procedures will be implemented in packing environmental samples:

- Check the sample container caps to make sure they are tightened properly. Samples for volatile components must be discarded and recollected if the cap is loose.
- Tape over the drain hole on the inside of the cooler.
- Place a layer of cushioning material in the bottom of the cooler.
- Enclose each bottle in a separate, clear, plastic bag and seal each bag. Place the bottles upright in the cooler and separate with packing materials so that they will not touch against each other during shipment. Place additional cushioning material around sample bottles, and fill voids between bottles.
- Place ice substitute between samples and over the containers to preserve them at or below 4°C. (Note: Loose bagged ice is not acceptable if coolers are transported by commercial aircraft.)
- Fill the cooler with cushioning material.
- Tape the cooler drain shut from the outside of the cooler.
- Place completed chain-of-custody form inside a resealable bag and tape the bag to the inside lid of the cooler.
- Close and latch the cooler. Wrap a strong adhesive tape around the ends of the cooler to secure it, making sure to cover the spigots at the bottom and any open space between the lid and the cooler. Tape the cooler latch closed with strapping tape.
- Seal the cooler with custody seals on the front and the sides and seal the cooler with strapping tape. The signature on the custody seals should match the signature on the chain-of-custody form.
- Attach the completed shipping label to the top of the cooler, print “Laboratory Samples” and “This End Up” on the top of the cooler, with upward-pointing arrows on all four sides. Place “Fragile” and “Chill, Do Not Freeze” labels on at least one side.

Samples will be packaged for shipment according to U.S. Department of Transportation (DOT) regulations. Marking and labeling procedures will be consistent with DOT regulations. The method of shipment, courier name(s), and other pertinent information will be entered on the chain-of-custody form. Air bills will be properly completed, and copies will be retained and placed in the project file.

For environmental samples, no DOT marking, labeling, or shipping papers are required, and there are no DOT restrictions on the mode of transportation. DOT regulations do not apply to transport by government owned vehicles, including aircraft.

6.3 Equipment Calibration

All equipment used at the site will be calibrated according to the manufacturer specifications, if necessary. Field equipment will be calibrated before the start of work each day. Any instrument drift from prior calibration will be recorded in the field notebook. Procedures for PID calibration are provided in SOP-6. Equipment found to be damaged, inoperable, or out of calibration will not be used until the discrepancy is corrected and verified by the field task manager or designee. As necessary, a detector sensitivity or test grid may be used to test the functionality before daily use, and results documented accordingly. Once equipment has been used, it will be maintained following manufacturer recommendations, and at intervals recommended by the manufacturer. Equipment calibration data will be recorded on an Equipment Calibration Form.

6.4 Equipment Decontamination

Decontamination of sampling equipment will be conducted consistently to minimize the potential for cross-contamination. Decontamination of large sampling equipment (for example the coring or grab sampling equipment) will be performed using the following decontamination procedure:

- Physically remove visible debris, to the extent practicable
- Nonionic detergent wash
- Site water rinse

Small, reusable equipment will be decontaminated following a similar procedure

- Physically remove visible debris, to the extent practicable
- Nonionic detergent wash
- Distilled/deionized water rinse
- Wrap in foil or place into clean plastic bags if not being used in the near term

This decontamination procedure will be used between sampling locations. Additional guidance is included in SOP-9. If sheens or oily residue is present, the equipment may be spray rinsed with either methanol or hexane, followed by a water rinse.

Disposable equipment intended for one-time use that is factory wrapped generally does not need to be decontaminated before use (if evidence of contamination is present, the disposable equipment will be discarded and not used). One-time use, disposable sampling equipment and accessories will be discarded after use and a new set of equipment will be used for each subsequent sample. Disposable sampling equipment will be used as much as practical, including nitrile gloves. Decontamination liquids and solids will be collected and disposed on-land in an approved wastewater receptacle.

6.5 Waste Management and Disposal

Waste generated during this investigation will consist primarily of decontamination water, PPE, disposable materials used for sample collection and processing, and leftover sediment from core processing. Containerized solid wastes will be placed in appropriately sized and rated containers, characterized, and disposed of at an appropriate EPA-approved waste facility per the Waste Management Plan (Appendix E of the PDI WP) and SOP-10. To the extent possible, any excess water or sediment remaining after sediment core collection and sectioning on the vessel, or generated during hand augering along the river banks will be returned to the collection site. The coring and grab sampling equipment, as well as the deck of the sampling vessel will be washed down using site water before moving to the next station; this water will not be containerized. Excess sediment containing significant sheen or NAPL, if encountered, will be containerized and managed as waste. Containerized liquid wastes, including decontamination water from the processing area, will be accumulated at the onsite designated waste accumulation area in secondary containment. Solvent rinse waste and NAPL, if generated, will be stored at the onsite designated waste accumulation area and managed as investigation-derived waste before offsite disposal. PPE and disposable materials associated with nonhazardous wastes will be put into black trash bags and disposed of in a municipal waste bin onsite.

The drum/container log will be completed, and photo documentation of the secure onsite waste accumulation area will be completed in accordance with SOP-3. Waste sampling will be performed in accordance with SOP-10 and managed in accordance with applicable local, state, and federal regulations and the Waste Management Plan (Appendix E of the PDI WP).

The waste disposal subcontractor will provide services including, but not limited to, preparation of profiles and manifests, transportation, and waste disposal. Waste management activities will be coordinated with Jacobs waste management specialists. Treatment, storage, and disposal facilities review and approval in accordance with the CERCLA Off-Site Rule (Title 40 of the *Code of Federal Regulations* Part 400.330) and appropriate out-of-state notification will be made in accordance with the requirements of the ASAOC.

7. Field Data Management

Field observations and measurement data will be recorded on dedicated field forms or field notebooks to create a permanent record of field activities. All hand-entered data will be reviewed by a second person to minimize data entry errors. A check for completeness of field records (e.g., logbooks, field forms, databases, and electronic spreadsheets) will ensure that all requirements for field activities have been fulfilled, complete records exist for each activity, and the procedures specified in this FSP have been implemented. Field documentation will ensure sample integrity and provide enough technical information to re-create each field event using the guidelines in Section 6.

Field data collected will be reviewed by the respective field sampling leaders to determine whether the qualitative parameters of representativeness and comparability have been achieved. In general, the review will be accomplished by comparing the chain-of-custody and field notebook entries with the sampling requirements. Any deficiencies will be communicated to the Jacobs PDI Project Manager immediately to determine what corrective action, if any, should be implemented.

The analytical data from the laboratory will be reviewed and validated in accordance with the QAPP (Appendix C of the PDI WP).

8. Permits

The investigation area is within the Willamette River, and the proposed sampling locations are below the OHW line in this area and require Division of State Lands permission for access. Per EPA, permits for sediment sampling are not required.

9. Health and Safety

All field survey and sediment sampling activities will be conducted according to Jacobs' Health, Safety, and Environment program requirements, which include project-specific field safety instructions. In accordance with Jacobs policy, all Jacobs field team members and subcontractors must successfully satisfy all Jacobs and site-specific health and safety requirements before working on the site, including Drug-Free Workplace training, wearing required PPE, and other requirements of the field safety instructions. Employees working over or near water will be provided with U.S. Coast Guard-approved life jackets or buoyant work vests.

A project-specific Health and Safety Plan (which includes an emergency response plan) is provided in Appendix D to the PDI WP.

10. References

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Tables

Table B-5-1. Sample Containers, Preservation, and Holding Times—Solid

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Analyte	Method	Container and Minimum Quantity	Preservation	Holding Time	Laboratory
Metals	SW6010B or C/ SW7471A or B	4-oz., G	None	28 days for mercury; 180 days for all other metals	ALS
TPH-DRO	NWTPH-Dx	4-oz., G	Cool ≤ 6°C	14 days to extraction; 40 days to analysis	ALS
Low-level bis(2-ethylhexyl)phthalate	SW8270C or D-LL	4-oz., G	Cool ≤ 6°C	14 days to extraction; 40 days to analysis	ALS
Polycyclic Aromatic Hydrocarbons	EPA SW8270C or D-SIM	4-oz., G 100 grams	Cool ≤ 6°C	14 days to extraction; 40 days to analysis	ALS
DDx isomers	E1699M	4-oz., G 100 grams	Cool ≤ 6°C	14 days to extraction; 40 days to analysis	ALS
Low Level Pesticides	E1699	4-oz., AG 100 grams	Cool ≤ 6°C	14 days to extraction; 40 days to analysis	ALS
Polychlorinated Biphenyl Aroclors	EPA SW8082	4-oz., G 100 grams	Cool ≤ 6°C	1 year to extraction; 1 year to analysis	ALS
Polychlorinated Biphenyl Congeners	E1668	4-oz., AG 100 grams	Cool ≤ 6°C	1 year to extraction; 1 year to analysis	ALS
Dioxins/Furans	E1613B	4-oz., AG 100 grams	Cool ≤ 6°C; store in dark	1 year to extraction; 1 year to analysis	ALS
Total Organic Carbon	EPA SW9060	4-oz., G 25 grams	Cool ≤ 6°C; store in dark	28 days	ALS
Grain Size	ASTM D7928/D6913	16-oz., G 150 grams	None	NA	ALS

Notes:

Bottle sizes may vary based on laboratory selection and potential sample consolidation.

°C = degree(s) Celsius
≤ = less than or equal to
AG = amber glass
ASTM = ASTM International
DDx = sum of six isomers associated with dichlorodiphenyltrichloroethane (DDT), dichlorodiphenyldichloroethane (DDD), dichlorodiphenyldichloroethene (DDE)
EPA = U.S. Environmental Protection Agency
G = glass
NA = not applicable
oz. = ounce(s)
TBD = to be determined

Table B-5-2. Sample Containers, Preservation, and Holding Times—Aqueous (Blanks)

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Analyte	Method	Container and Minimum Quantity	Preservation	Holding Time	Laboratory
Metals	SW6010B or C/ SW7470A	1 × 500 mL, P	Nitric acid to pH < 2; chill to ≤ 6°C (field filter prior to acid preservation if dissolved metals required)	28 days for mercury; 180 days for all other metals	ALS
TPH-DRO	NWTPH-Dx	2 × 250-mL, G-TLC	Hydrochloric acid to pH < 2; chill to ≤ 6°C	14 days to extraction; 40 days to analysis	ALS
Low-level bis(2-ethylhexyl)phthalate	SW8270C or D-LL	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	7 days to extraction; 40 days to analysis	ALS
Polycyclic aromatic hydrocarbons	SW8270C or D-SIM	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	7 days to extraction; 40 days to analysis	ALS
DDx isomers	E1699M	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	7 days to extraction; 40 days to analysis	ALS
Low-level Pesticides	E1699	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	7 days to extraction; 40 days to analysis	ALS
Polychlorinated Biphenyl Aroclors	EPA SW8082	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	1 year to extraction; 1 year to analysis	ALS
Polychlorinated Biphenyl Congeners	E1668	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	1 year to extraction; 1 year to analysis	ALS
Dioxins/Furans	E1613	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C; store in dark	1 year to extraction; 1 year to analysis	ALS

Table B-5-2. Sample Containers, Preservation, and Holding Times—Aqueous (Blanks)

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Analyte	Method	Container and Minimum Quantity	Preservation	Holding Time	Laboratory

Notes:

Bottle types may vary based on availability or sharing.

°C = degree(s) Celsius

≤ = less than or equal to

AG-TLC = amber glass with Teflon-lined cap

DDx = sum of six isomers associated with dichlorodiphenyltrichloroethane (DDT), dichlorodiphenyldichloroethane (DDD), dichlorodiphenyldichloroethene (DDE)

EPA = U.S. Environmental Protection Agency

mL = milliliter(s)

TBD = to be determined

Table B-2-1 - Proposed Sediment Sample Locations and Associated Analytical Schedule

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Location ID	Primary or Stepout	Sample Type (Grab/Core)	Target Core Depth (ft)	Target X	Target Y	Elevation Portland Harbor 2018 Bathymetry (NAVD88)	Within FMD Area	FMD Operational Elevation (NAVD88)	Immediate Analysis and Archiving Schedule
Preliminary SMA 1									
PD01	Stepout	Surface Grab & Core	10	7630034	699944	-6.2	No	NA	A
PD02	Primary	Surface Grab	NA	7630123	699852	-8.6	No	NA	X
PD03	Primary	Surface Grab & Core	10	7630139	699833	-9.5	No	NA	B
PD04	Primary	Surface Grab	NA	7630155	699813	-10.9	No	NA	X
PD05	Stepout	Surface Grab & Core	10	7630111	699799	0.0	No	NA	A
PD06	Stepout	Surface Grab & Core	10	7630218	699706	-0.2	No	NA	A
PD07	Stepout	Surface Grab & Core	10	7630326	699601	-1.8	No	NA	A
PD08	Stepout	Surface Grab & Core	10	7630454	699489	-4.5	No	NA	A
Preliminary SMA 2									
PD09	Stepout	Surface Grab & Core	10	7629110	701165	-32.5	No - edge of channel	-34.7	A
PD10	Stepout	**Surface Grab & Core**	10	7629417	700791	-40.4	No - edge of channel	-34.7	A
PD11	Stepout	Surface Grab & Core	10	7629606	700558	-28.2	No - edge of channel	-34.7	A
PD12	Primary - Under Dock	Surface Grab	NA	7629005	701116	-20.7	No	NA	X
PD13	Primary	Surface Grab	NA	7629121	700972	-37.2	Yes - recently dredged	-34.7	X
PD14	Primary	Surface Grab & Core	10	7629137	700953	-37.0	Yes - recently dredged	-34.7	C
PD15	Primary	Surface Grab	NA	7629153	700933	-36.7	Yes - recently dredged	-34.7	X
PD16	Primary - Under Dock	Surface Grab	NA	7629210	700872	-24.3	No	NA	X
PD17	Primary	Surface Grab	NA	7629240	700793	-37.5	Yes - recently dredged	-34.7	X
PD18	Primary	Surface Grab & Core	10	7629240	700768	-38.0	Yes - recently dredged	-34.7	B
PD19	Primary	Surface Grab & Core	10	7629356	700675	-34.9	Yes - not recently dredged	-19.7*	B
PD20	Primary - Under Dock	Surface Grab	NA	7629410	700638	-25.5	No	NA	X
PD21	Primary	Surface Grab & Core	10	7629478	700546	-23.9	Yes - not recently dredged	-14.7**	B
PD22	Stepout	Surface Grab & Core	10	7629579	700428	-20.6	No	NA	A
PD23	Primary	Surface Grab & Core	10	7629027	700859	-33.9	Yes - recently dredged	-34.7	C
PD24	Stepout	Surface Grab & Core	10	7629167	700689	-35.0	Yes - recently dredged	-34.7	A
PD25	Stepout	Surface Grab & Core	10	7628907	700907	-34.7	Yes - recently dredged	-33.2	A
PD26	Primary - Under Dock	Surface Grab	NA	7629037	700731	-24.0	No	NA	X
PD27	Stepout	Surface Grab & Core	10	7628906	700718	-33.2	Yes - recently dredged	-34.7	A
Preliminary SMA 3									
PD28	Primary	Surface Grab & Core	10	7629070	700632	-34.7	Yes - recently dredged	-34.7	C
PD29	Stepout	Surface Grab & Core	10	7629391	700519	-23.8	Yes - not recently dredged	-14.7	A
PD30	Stepout	Surface Grab & Core	10	7629483	700407	-22.4	No	NA	A
PD31	Primary	Surface Grab & Core	15	7629282	700433	-22.6	Yes - not recently dredged	-14.7	B
PD32	Primary	Surface Grab & Core	15	7629377	700309	-22.3	No	NA	B
PD33	Stepout	Surface Grab & Core	15	7629425	700192	-16.6	No	NA	A
PD34	Primary - Under Dock	Surface Grab	NA	7629126	700405	-20.1	No	NA	X
PD35	Primary	Surface Grab & Core	15	7629152	700330	-14.6	Yes - not recently dredged	-14.7	B
PD36	Primary	Surface Grab & Core	15	7629255	700209	-17.4	No	NA	B
PD37	Stepout	Surface Grab & Core	15	7629318	700113	0.0	No	NA	A
PD38	Stepout	Surface Grab & Core	15	7629053	700233	-13.1	Yes - not recently dredged	-14.7	A
PD39	Stepout	Surface Grab & Core	15	7629140	700120	-11.8	No	NA	A
Preliminary SMA 4									
PD40	Stepout	Surface Grab & Core	10	7628637	700704	-33.2	Yes - recently dredged	-33.2	A
PD42	Primary	Surface Grab & Core	10	7628755	700547	-32.2	Yes - recently dredged	-34.7	C
PD43	Primary - Under Dock	Surface Grab	NA	7628791	700526	-20.0	No	NA	X

Table B-2-1 - Proposed Sediment Sample Locations and Associated Analytical Schedule

Pre-Design Investigation Work Plan, Willbridge Cove Project Area

Location ID	Primary or Stepout	Sample Type (Grab/Core)	Target Core Depth (ft)	Target X	Target Y	Elevation Portland Harbor 2018 Bathymetry (NAVD88)	Within FMD Area	FMD Operational Elevation (NAVD88)	Immediate Analysis and Archiving Schedule
PD44	Primary	Surface Grab & Core	10	7628854	700450	-33.1	Yes - recently dredged	-34.7	C
PD45	Stepout	Surface Grab & Core	10	7629003	700514	-34.0	Yes - recently dredged	-34.7	A
PD46	Primary	Surface Grab & Core	15	7628968	700363	-21.2	Yes - not recently dredged	-19.7*	B
PD47	Primary	Surface Grab & Core	10	7628607	700614	-14.5	No	NA	B
PD48	Primary	Surface Grab & Core	10	7628696	700502	-18.2	No	NA	B
PD49	Stepout	Surface Grab	NA	7628741	700426	-9.7	No	NA	A
PD50	Primary	Surface Grab & Core	10	7628773	700394	-14.2	No	NA	B
PD51	Stepout	Surface Grab	NA	7628799	700360	-10.5	No	NA	A
PD52	Primary	Surface Grab & Core	10	7628834	700355	-23.9	No	NA	B
PD53	Primary	Surface Grab & Core	10	7628912	700298	-17.4	No	NA	B
Preliminary SMA 5									
PD41	Primary	Surface Grab & Core	10	7628744	700632	-32.8	Yes - recently dredged	-34.7	C

Notes:

*Permitted elevation is -39 CRD or -33.7 NAVD88, but operational elevation is much shallower

**Permitted elevation is -32 CRD or -26.7 NAVD88, but operational elevation is much shallower

A = Archive all sampling intervals for all chemical analyses

B = Analyze surface grab and core intervals 1-2 ft, 2-3 ft, 3-4 ft, and 4-5 ft immediately. Archive core samples 0-1 foot and below 5 feet.

C = Analyze surface grab immediately and the four sediment core intervals at and below the the operational elevation immediately. Archive all other core sample intervals.

X = Analyze surface grab immediately.

DDD = dichlorodiphenyldichloroethane

DDE = dichlorodiphenyldichloroethene

DDT = dichlorodiphenyltrichloroethane

DDx = DDD, DDE, and DDT

PAH = polycyclic aromatic hydrocarbon

PCB = polychlorinated biphenyl

TOC = total organic carbon

Coordinates are in NAD 1983 StatePlane Oregon North FIPS 3601 Feet Intl

Samples for the following analyses will be collected from each sampling interval.

- PAHs by SW8270 C or D-SIM
- PCB Aroclors by SW8082
- DDX isomers by 1699M
- Dioxins/Furans by E1613
- Grainsize by D7928, D6913
- TOC by 9060
- Archive aliquot

Table B-2-2 - Proposed River Bank Soil Sample Locations and Associated Analytical Schedule

Pre-Design Investigation Work Plan Addendum, Willbridge Cove Group Project Area

Location ID	Sample Description	Target X	Target Y	Target Boring Depth	Analysis and Archiving Scheme
WC-S01	Soil Boring	7628501	700669	5 ft	All surface soil samples will be analyzed immediately for Table 17 analyses listed below. All subsurface soil samples will be held frozen pending analysis of the surface interval. Subsurface soil intervals released for analysis will be analyzed for Table 21 analyses listed below.
WC-S02	Soil Boring	7628534	700624	5 ft	
WC-S03	Soil Boring	7628576	700549	5 ft	
WC-S04	Soil Boring	7628590	700560	5 ft	
WC-S05	Soil Boring	7628652	700470	5 ft	
WC-S06	Soil Boring	7628661	700480	5 ft	
WC-S07	Soil Boring	7628719	700401	5 ft	
WC-S08	Soil Boring	7628728	700407	5 ft	
WC-S09	Soil Boring	7628802	700273	5 ft	
WC-S10	Soil Boring	7628798	700303	5 ft	
WC-S11	Soil Boring	7628835	700236	5 ft	
WC-S12	Soil Boring	7628856	700256	5 ft	

Notes:

Surface soils will be analyzed for the following:

- Metals, including mercury by SW6010B or C and SW7471A or B
- TPH-DRO by NWTPH-Dx
- PAHs by SW8270 C or D-SIM
- Bis(2-ethylhexyl)phthalate (BEHP) by low level SW8270C or D-SIM
- Pesticides by E1699
- Dioxins/Furans by E1613
- PCB congeners by E1668

Analysis of subsurface soils, if needed, will be performed using the following methods:

- PAHs by SW8270 C or D-SIM
- PCB Aroclors by SW8082
- DDT isomers by 1699M
- Dioxins/Furans by E1613

Coordinates are in NAD 1983 StatePlane Oregon North FIPS 3601 Feet Intl

Figures

Willbridge Cove Project Area

Smith and Bybee Lakes Park
Forest Park
Heron Lakes Golf Course
Portland International Raceway
Portland International Airport
Columbia River
Willamette River
Columbia Slough
Portland
Multnomah
Forest Park
Bethany
Oak Hills
Cedar Mill
Washington
Germantown
Springville
Thompson
Clark
Columbia House
Pearson Field
Cornfoot
McCarthy Creek
Miller Creek
Newberry
Skyline
Burgard
Columbia
Lombard
Saint Louis
Smith
Fessenden
Richmond
Oswego
Wall
Portsmouth
Willis
Chautauqua
Peninsular
Delaware
Kilpatrick
Denver
Marine
Portland
Vancouver
Whitaker
Victory 5 Ramp
Schmeer
Walker
6th
3rd
33rd
Dekum
Ainsworth
Killingsworth
Jessup
Albina
Minnesota
Alberta
Going
Blandena Going
Prescott
Fremont
Knott
Tillamook
Broadway
Halsey
Multnomah
Irving
Glisan
Burnside
Lovejoy
Everett
Couch
Broadway Bridge
Burnside Bridge
Vaughn
Nicolai
Ward
Russell
Weidler
Cook
Williams
Front
Saint Helens
Willamette
Doane Creek
Saltzman Creek
Abbey Creek
Rock Creek
Cornelius Pass
Old Skyline
Kaiser
Bronson Creek
Willow Creek
Cedar Mill Creek
Evergreen
Bethany
West Union
185th
143rd
158th
112th
Cornell
Barnes
Murray
Walker
Cedar Hills
Johnson Creek
Skyline
Thompson
Orchard
Vancouver
Hillsboro
Beaverton
Project Location
26
30
99
5
405
84
14
Evergreen
Columbia House
Columbia
Marine
Portland
Vancouver
Walker
6th
3rd
33rd
Dekum
15th
Ainsworth
Killingsworth
Albina
Jessup
Minnesota
Alberta
Going
Blandena Going
Prescott
Fremont
Knott
Tillamook
Broadway
Halsey
Multnomah
Irving
Glisan
Burnside
Lovejoy
Everett
Couch
Broadway Bridge
Burnside Bridge
Vaughn
Nicolai
Ward
Russell
Weidler
Cook
Williams
Front
Saint Helens
Willamette
Doane Creek
Saltzman Creek
Abbey Creek
Rock Creek
Cornelius Pass
Old Skyline
Kaiser
Bronson Creek
Willow Creek
Cedar Mill Creek
Evergreen
Bethany
West Union
185th
143rd
158th
112th
Cornell
Barnes
Murray
Walker
Cedar Hills
Johnson Creek
Skyline
Thompson
Orchard
Vancouver
Hillsboro
Beaverton
Project Location
26
30
99
5
405
84
14

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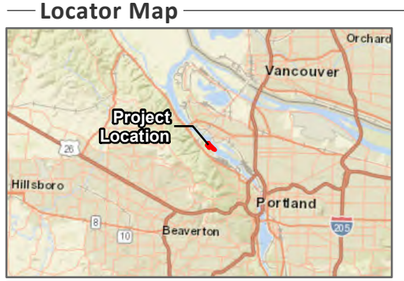
Locator Map



Legend

- Limited Access
- Highway
- Major Road
- Railroads (Local)
- Rivers (Local)
- Stream/River
- City
- Hospital
- Willbridge Cove Project Area
- Local Park
- State Park or Forest
- Golf Course
- Stadium or Arena

Map Notes
* Base data from ESRI Basemaps

0 5 10 20 Miles

N

FIGURE B-1-1
SITE LOCATION
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

Willamette River

RM 7.5
RM 7.6
RM 7.7
RM 7.8
RM 7.9
RM 8
RM 8.1

Preliminary SMA 1
Preliminary SMA 2
Preliminary SMA 3
Preliminary SMA 4
Preliminary SMA 5

Kinder Morgan Dock
Chevron Dock
Phillips 66 Dock
Tidewater Barge
McCall Dock

Atofina Chemicals - former Arkema Portland
Glacier Northwest, Inc.
GS Roofing
Kinder Morgan Liquids Terminals LLC
Chevron Willbridge Terminal
Phillips 66 Willbridge Terminal
McCall Oil and Chemical Corporation
Tube Forgings of America
NW Front Ave

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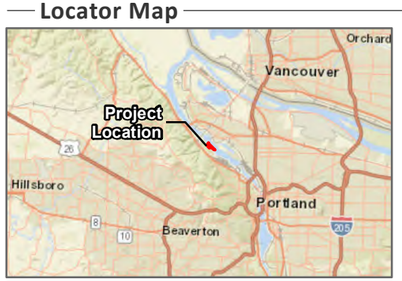
Locator Map



Legend

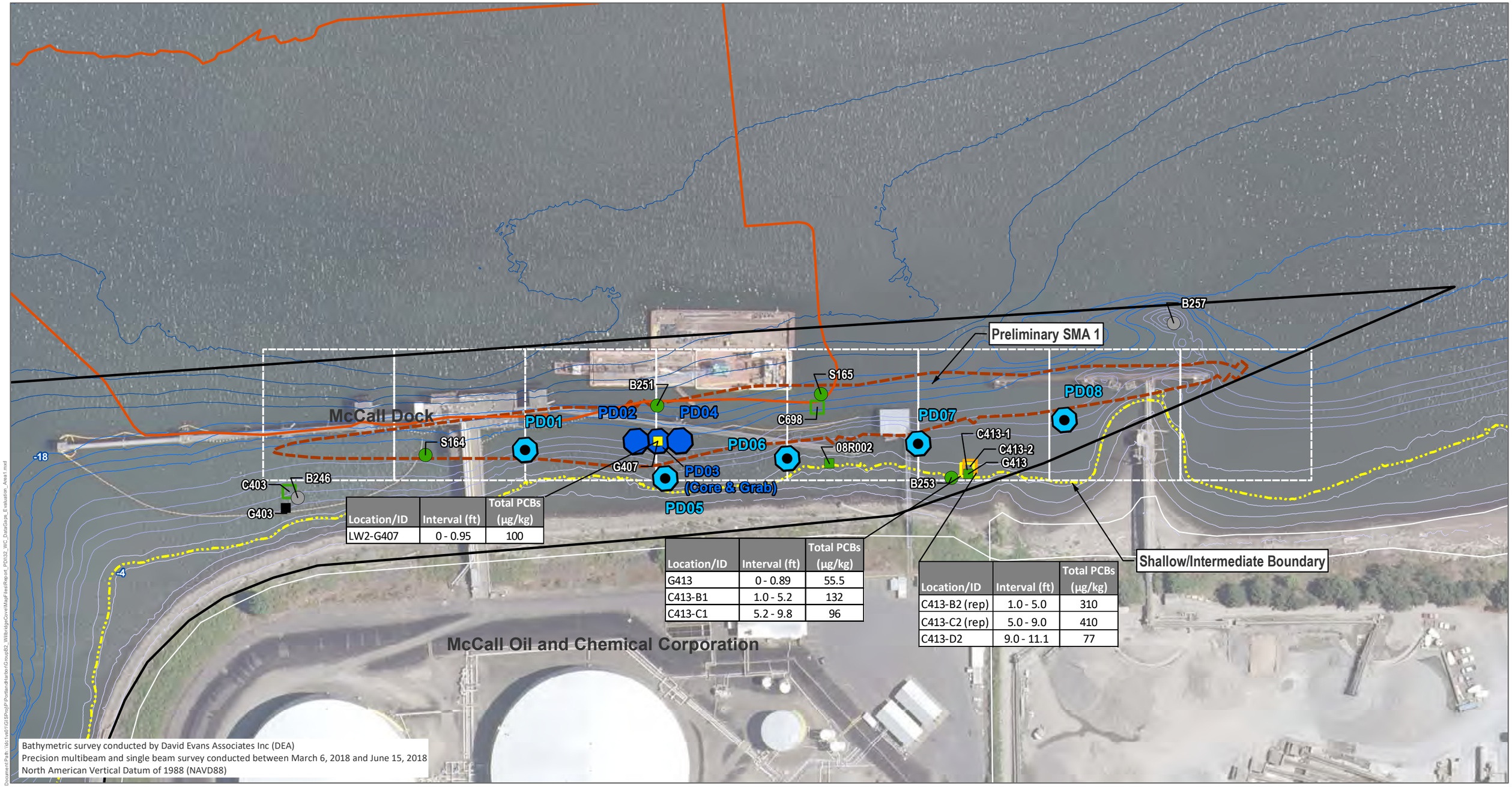
- Top_of_Bank
- River Miles (Tenths)
- Navigation Channel
- Tax Lots (Metro RLIS)
- Sediment Management Area (ROD ESD 2019)
- Willbridge Cove Project Area

Map Notes
* Metro RLIS Tax Lots (Tax Lots Identification TLID)
* Metro 2019 Aerial Imagery
* Base data from Portland Harbor Environmental Data Portal (http://ph-public-data.com/)

0 187.5 375 Feet

FIGURE B-1-2
PRELIMINARY SEDIMENT MANAGEMENT AREAS
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs



Location/ID	Interval (ft)	Total PCBs (µg/kg)
LW2-G407	0 - 0.95	100

Location/ID	Interval (ft)	Total PCBs (µg/kg)
G413	0 - 0.89	55.5
C413-B1	1.0 - 5.2	132
C413-C1	5.2 - 9.8	96

Location/ID	Interval (ft)	Total PCBs (µg/kg)
C413-B2 (rep)	1.0 - 5.0	310
C413-C2 (rep)	5.0 - 9.0	410
C413-D2	9.0 - 11.1	77

Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

Legend

Proposed Samples
- Primary Core/Grab (10ft)
- Primary Surface Grab
- Stepout Core/Grab (10ft)

PCB µg/kg - Surface Sediment
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

PCB µg/kg - Subsurface Sediment
Maximum detected concentration in core
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

- Bathymetry (2ft) Contours
- Shallow/Intermediate Boundary
- Willbridge Cove Project Area
- 2020-2021 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 100 200 Feet

FIGURE B-2-1

PROPOSED SAMPLE LOCATIONS
PRELIMINARY SMA 1 EVALUATION
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

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Location/ID	Sample Interval (ft) 2002	Sample Interval (ft) 2009	Total PCBs (µg/kg)	Adjusted Sample Interval due to Deposition (ft)
AN-CTPD-03	0 - 0.07	0 - 0.07	2002: 130 2009: ND	3 - 3.07
AN-CTPD-03	0-0.5		2002: 140	3 - 3.5

Location/ID	Sample Interval (ft) 2002	Sample Interval (ft) 2009	Total PCBs (µg/kg)
AN-CTPD-06	0 - 0.07	0 - 0.07	2002: 130 2009: ND
AN-CTPD-06	0-0.5		2002: 110

Location/ID	Interval (ft)	Total PCBs (µg/kg)
AN-CTPD-02	0 - 0.07	110
AN-CTPD-02	0-0.5	91

Location/ID	Interval (ft)	Total PCBs (µg/kg)
DMMU2SDO4	0 - 5.4	99

Location/ID	Interval (ft)	Total PCBs (µg/kg)
AN-CTPD-07	0 - 0.07	120
AN-CTPD-07	0-0.5	92

PD09
PD10
PD11
PD12
PD13
PD14 (Core & Grab)
PD15
PD16
PD17
PD18 (Core & Grab)
PD19
PD20
PD21
PD22
PD23
PD24
PD25
PD26
PD27
PD28

AN-CTPD-03 (2018 bathymetry data indicate mudline elevation is shallower than when the 2002 sample was collected)

S161 (2018 PCB congener results for surface sample at same interval and elevation as TPD-06 0-0.5 ft sample)

AN-CTPD-06

S158
SC-S157
S157
S159
S162
DMMU2

Preliminary SMA 2

AN-CTPD-02 (Removed during 2011 dredging)

DMMU-1-C (Surface interval removed during 2011 dredging. Subsurface result shown represents leave surface)

AN-CTPD-07 (Removed during 2011 dredging)

DMMU-2-C (Surface interval removed during 2011 dredging. Subsurface result shown represents leave surface

DMMU-1B

Kinder Morgan Dock
Chevron Dock
Phillips 66 Dock
Tidewater Barge

-32 -38 -36 -28 -34 -26 -24 -18 -16 -14 -12 -8 -6 -4 -2 -22 2

Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

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Legend

Proposed Samples
- Primary Core/Grab (10ft)
- Stepout Core/Grab (10ft)
- Primary Surface Grab
- Stepout Grab
- Under Dock Grab

PCB µg/kg - Surface Sediment
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

PCB µg/kg - Subsurface Sediment
Maximum detected concentration in core
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

- Bathymetry (2ft) Contours
- Shallow/Intermediate Boundary
- 2011 Dredge Prism
- Willbridge Cove Project Area
- Sediment Management Area (ROD ESD 2018)

- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 75 150 Feet

FIGURE B-2-2
PROPOSED SAMPLE LOCATIONS
PRELIMINARY SMA 2 EVALUATION
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

Location/ID	Interval (ft)	Total PCBs (µg/kg)
URB-2Z	3.9 - 4.9	200

Location/ID	Interval (ft)	Total PCBs (µg/kg)
URB-DP	0 - 6.4	83

Location/ID	Interval (ft)	Total PCBs (µg/kg)
URB-1Z	9.7 - 10.7	130

Location/ID	Interval (ft)	Total PCBs (µg/kg)
C532-A	0 - 0.98	150
C532-B	1.0 - 3.2	180
C532-C	3.2 - 6.5	120
C532-D	6.49 - 9.61	380
C532-E	9.6 - 11.1	49
C532-F	11.1 - 12.2	ND

Location/ID	Interval (ft)	Total PCBs (µg/kg)
SC-S163	0 - 1.0	15.3
	0 - 2.0	27
	2.0 - 4.0	177
	4.0 - 6.0	59.8
	6.0 - 8.0	88.2
	8.0 - 10.0	50.8
	10.0 - 12.7	285
	12.7 - 13.0	2.7

Location/ID	Interval (ft)	Total PCBs (µg/kg)
G694	0 - 0.7	89

Location/ID	Interval (ft)	Total PCBs (µg/kg)
SD117	0 - 0.3	96
SD117	0 - 2.9	160

Location/ID	Interval (ft)	Total PCBs (µg/kg)
APS-3T	0 - 1.9	86
APS-3B	2.0 - 4.0	110

S162, PD29, PD30, URB-2, PD31, NA-3B, PD32, URB-3, URB-D, G694, PD33, SC-S163, S163, SD117, PD34, C532, URB-1, PD36, Preliminary SMA 3, APS-3, Tidewater Barge, PD37, PD35, APS-2, BT021, B241, PD38, PD39, C401, Phillips 66 Dock, Shallow/Intermediate Boundary

Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

Legend

Proposed Samples
- Primary Core/Grab (10ft)
- Primary Core/Grab (15ft)
- Under Dock Grab
- Stepout Core/Grab (10ft)
- Stepout Core/Grab (15ft)
- Stepout Grab

PCB µg/kg - Surface Sediment
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

PCB µg/kg - Subsurface Sediment
Maximum detected concentration in core
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

- Bathymetry (2ft) Contours
- Shallow/Intermediate Boundary
- Sediment Management Area (ROD ESD 2018)
- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 75 150 Feet

FIGURE B-2-3
PROPOSED SAMPLE LOCATIONS
PRELIMINARY SMA 3 EVALUATION
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Document Path: \\dcs1vs01\GIS\Proj\PortlandHarbor\GroupB2_Willbridge\CoveMapFiles\Report_PDI\34_WC_DataGaps_Evaluation_Area3.mxd

Kinder Morgan Dock

Chevron Dock

Phillips 66 Dock

Preliminary SMA 5

Preliminary SMA 4

DMMU-1-B
(Surface interval removed during 2011 dredging. Subsurface result shown represents leave surface)

DMMU-1-A
(Surface interval removed during 2011 dredging. Subsurface result shown represents leave surface)

DMMU-2-B
(Surface interval removed during 2011 dredging. Subsurface result shown represents leave surface)

DMMU-2-A
(Surface interval removed during 2011 dredging. Subsurface result shown represents leave surface)

AN-CTPD-01
(Removed during 2011 dredging)

AN-CTPD-08
(Removed during 2011 dredging)

Location/ID	Interval (ft)	Total PCBs (µg/kg)
AN-CTPD-01A	0 - 0.07	120
AN-CTPD-01B	0 - 0.5	110

Location/ID	Interval (ft)	Total PCBs (µg/kg)
AN-CTPD-08A	0 - 0.07	130
AN-CTPD-08B	0 - 0.5	120

Location/ID	Interval (ft)	Total PCBs (µg/kg)
DMMU1/SK091-A	0 - 5.8	390
DMMU1/SK091-Z	6.8 - 10.7	270

Location/ID	Interval (ft)	Total PCBs (µg/kg)
DRB1/DRB-1Z	10.1 - 11.1	79

Location/ID	Interval (ft)	Total PCBs (µg/kg)
DMMU3B	0 - 0.07	560
DMMU3B13	0-3.0	261

Location/ID	Interval (ft)	Total PCBs (µg/kg)
LW2-G394	0 - 0.8	790

PD40 PD41 PD42 PD43 PD44 PD45 PD46 PD47 PD48 PD49 PD50 PD51 PD52 PD53

B239 S160 G389 G394 GSP07W

DMMU-3-A DMMU-3-B DMMU-3-D DMMU-3-E DMMU1 DRB-1 APS-1

Shallow/Intermediate Boundary

Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

Legend

Proposed Samples
- Primary Core/Grab (10ft)
- Primary Core/Grab (15ft)
- Under Dock Grab
- Stepout Core/Grab (10ft)
- Stepout Core/Grab (15ft)
- Stepout Grab

PCB µg/kg - Surface Sediment
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

PCB µg/kg - Subsurface Sediment
Maximum detected concentration in core
- <9 (less than CUL)
- 9 to 75 (CUL to Sitewide RAL)
- 75 to 200 (Sitewide RAL to PTW)
- 200 to 1000 (PTW to NAV RAL)
- Non-Detect

- Bathymetry (2ft) Contours
- Shallow/Intermediate Boundary
- 2011 Dredge Prism
- Sediment Management Area (ROD ESD 2018)

- FS Database (Portland Harbor)
- PDI-BL Sample Database (Pre-RD; Portland Harbor)

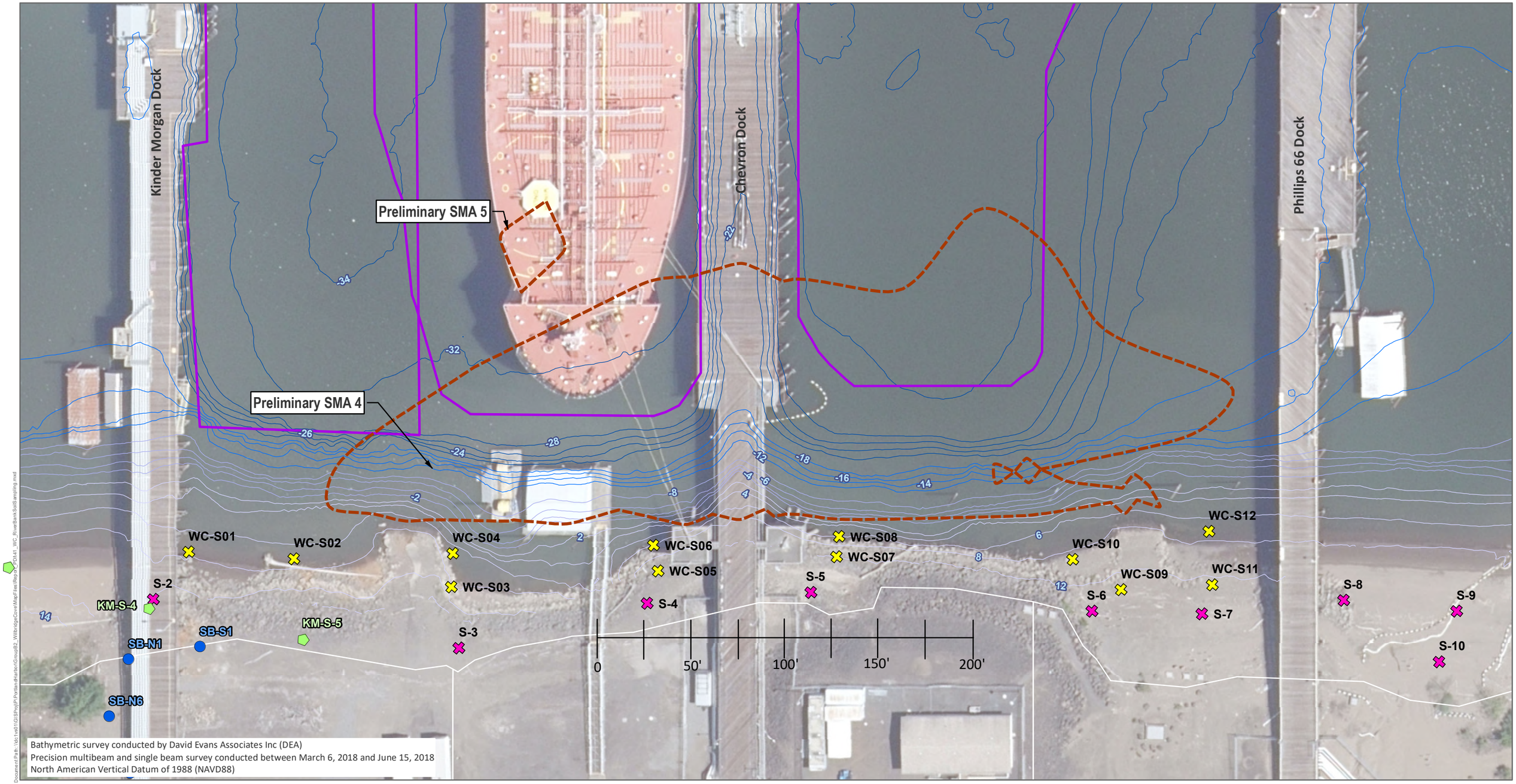
CUL – Clean up Level
RAL – Remedial Action Level
NAV RAL – Navigation Channel RAL
PTW – Principal Threat Waste

0 62.5 125 Feet

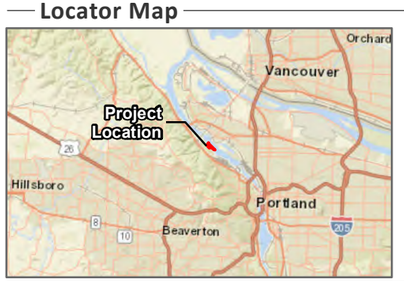
FIGURE B-2-4
PROPOSED SAMPLE LOCATIONS
PRELIMINARY SMA 4 AND SMA 5 EVALUATION
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

Document Path: \\dc-tvs01\GISPro\P\PortlandHarbor\Groups\B2_WillbridgeCove\MapFiles\Report_PDI\31_WC_DataGaps_Evaluation_Area4.mxd



Locator Map



Legend

- Proposed River Bank Soil Sample
- Surface Soils Sample (2008)
- KMLT Surface Sample Locations (2012)
- KMLT Soil Boring/LIF Locations (2018)
- Preliminary Sediment Management Area
- Dredge Area
- Bathymetry (2ft) Contours

Map Notes
* Metro RLIS Tax Lots
* Metro 2019 Aerial Imagery

0 62.5 125 Feet

FIGURE B-2-5

PROPOSED RIVER BANK SOIL SAMPLE LOCATIONS

Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Jacobs

Attachment 1
Standard Operating Procedures

STANDARD OPERATING PROCEDURE-1

1. Field Administration and Records

The purpose of this standard operating procedure (SOP) is to delineate protocols for field administration and recordkeeping of field data. The forms and field logs for recordkeeping for specific procedures are discussed in the appropriate sections. Other forms and records are the responsibility of the site coordinator.

1.1 Associated Procedures

- SOP-2: Field Logbooks
- SOP-3: Photographic Documentation

1.2 Equipment and Supplies

- Various preprinted forms (see Appendix B to the FSP)
- Pens with indelible ink
- Copy machine

1.3 Procedures

Forms and records will be completed in indelible ink and stored in file cabinets at the Jacobs office in Portland, Oregon. The field lead will be responsible for maintenance and control of forms.

1.4 Recordkeeping

Equipment rental and provided services records, including receipts for materials, will be the responsibility of the field team lead. Records will be copied at the end of each field event and stored on in the project field notes folder on the server.

End of SOP-1

STANDARD OPERATING PROCEDURE-2

2. Field Logbooks

The purpose of this standard operating procedure (SOP) is to delineate protocols for recording field survey and sampling information in the field logbook.

2.1 Associated Procedures

Almost all procedures are associated with a logbook. The bound field logbook is the primary field documentation instrument. Site logbooks will be used for documentation of field visits, site monitoring, surface water sampling, groundwater sampling, and site inspections.

2.2 Equipment and Supplies

- Waterproof, bound logbooks with consecutively numbered pages
- Pens with indelible ink

2.3 Procedure

The field logbook serves as the primary record of field activities. Field logs such as calibration logs, grout tube inspection sheets, groundwater survey forms, low flow well monitoring data sheets, purge water drum log, and wellhead inspection checklists also should be used.

The field lead will be responsible for custody of the logbooks when they are not being used.

Pertinent work and sampling information will be recorded in a logbook during each day of the field effort. The field team leader on a task is responsible for completing the logbook and ensuring its custody when the logbook is outside the office.

When a new logbook is opened, it will be given a unique document control number, which will be written on the inside cover and on the top of page 1. Each page in the book will be numbered sequentially. The first two pages will be reserved for an index. If the logbook is a site logbook, index columns will be set up (left to right) for site identification, function, pages where the information will be found, and initials of the person that completed the work.

Each morning, the investigation team leader will obtain the appropriate logbook(s) and complete the required information. The record for the day will be kept sequentially. Entries will be made in indelible ink. Each page's entries will be initialed at the bottom of the page. Entries will be made chronologically and in sufficient detail to allow the writer or reviewer to reconstruct each day's events. Any changes to the Sampling and Analysis Plan will be documented in the field logbook. If changes need to be made to the sampling procedures or analysis, the field staff will contact the task manager and project manager to determine the appropriate action. At the end of the day, a line will be drawn through any blank space on the page, and the author will sign and date the bottom of the final page. All corrections will consist of lined-out deletions that are initialed.

The heading for each day's work will contain the following information:

Date: _____

Tasks for the day: _____

Start Time: _____ AM Weather: _____

Field staff onsite:

Pre-task Safety Planning (PTSP) Meeting:

Strict chain-of-custody (CoC) procedures will be maintained with the field logbooks. While being used in the field, the logbook will remain with the assigned log keeper at all times. At the end of the day, logbooks will be scanned, and the images stored on the server.

2.4 Recordkeeping

No rules can specify the complete content of information that should be recorded. Each logbook will contain sufficient information to allow reconstruction of the field activity without relying on the memory of the field crew. If the information changes during the day, the changes should be noted. In addition to the heading information, the types of information that will be in the logbook include:

- Health and safety protection (level)
- Site entry conditions (observations)
- Sampler's name
- Date and time of sampling
- Sample location and description; a sketch with measured distances from reference points will be recorded if there is no established identification for the sample location
- Sample designation as composite or grab
- Sample matrix
- Sample methodology
- Sample identification
- Sample preservation
- Associated quality control blanks
- Type and identification of sampling equipment used
- Field measurement data (such as, pH, temperature, and conductivity)
- Field observations that may be relevant to the analysis or sample integrity (odor, color, and weather conditions)
- Lot numbers of sample containers, CoC number, and custody seal number
- Quality control sample(s) collected
- Variances or changes to the field sampling plan
- Site conditions at the time of completion of sampling
- Time left site
- Shipping arrangement
- Destination laboratory
- Signature and date by personnel collection

End of SOP-2

STANDARD OPERATING PROCEDURE-3

3. Photographic Documentation

The purpose of this standard operating procedure (SOP) is to ensure that representative digital photographic evidence of site conditions, field technician observations, site work procedures, and completed project work are documented. The photographs or videos produced may also include project-related items of significant and noteworthy interest, such as coverage of major project events, innovative field techniques, or procedures that should be documented.

It is the responsibility of the field team leader to determine when and if photographs will be made during a project.

3.1 Definitions

None.

3.2 Associated Procedures

- SOP-1: Field Administration and Records
- SOP-2: Field Logbooks

3.3 Equipment and Supplies

- Camera, with internal date stamp
- Batteries for the camera
- Pens with indelible ink

3.4 Procedures

Follow the camera directions for taking photographs. At the end of the field day, load all photos into the field photo directory on the server. Include several relevant photos for the daily report.

3.5 Recordkeeping

Photographs will be listed in a photo log form. The information, at a minimum, will include:

- Photographic log sheet number
- Photograph number
- Location
- Time
- Description of picture
- Names of any persons in the picture

End of SOP-3

STANDARD OPERATING PROCEDURE-4

4. Documentation in ESRI ArcGIS Collector App

The purpose of this standard operating procedure (SOP) is to assure that sampling information and other relevant field data are documented and recorded through the ESRI ArcGIS Collector app. The app will be used to document and organize photographs, GPS coordinates of sampling locations, field observations, etc.

4.1 Definitions

None.

4.2 Associated Procedures

- SOP-1: Field Administration and Records
- SOP-2: Field Logbooks
- SOP-3: Photographic Documentation
- SOP-4b Mobile Data Collection App Set up

4.3 Equipment and Supplies

- iPad
- Trimble R1 GPS receiver

4.4 Procedures

Follow the attached instructions for iPad setup. At the end of the field day, load all data into the field data directory on the server.

4.5 Attachments

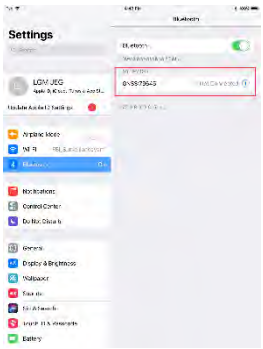
- SOP-4b - iPad, Collector and GPS Receiver Setup

End of SOP--4

iPad, Collector and GPS Receiver Setup

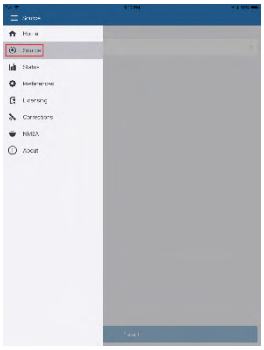
1. Download apps on to device from the App Store (“Trimble GNSS Status”, “ESRI Collector).

2. Power on the Trimble R1 GPS receiver. Refer to Appendix I for details.
3. On your iPad go to “Settings” and turn on Bluetooth and connect to the R1 under “MY DEVICES”. It will display as ‘GNSS: XXXXX’ where “X” is the name of the R1 GPS receiver unit.

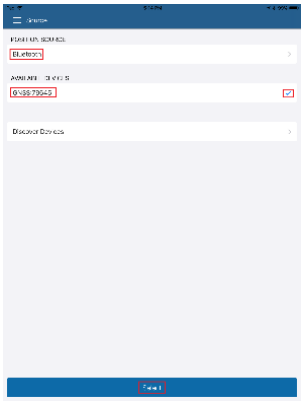


4. Once connected via Bluetooth, launch the “GNSS Status” app.

   a. Press the drop-down menu in the upper left and select “Source”.

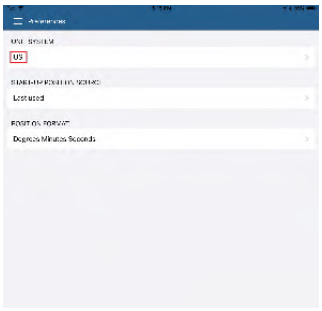


b. Under “POSITION SOURCE” select “Bluetooth”. Then Under “AVAILABLE DEVICES” select your GNSS receiver and press the “Select” bar at the bottom.

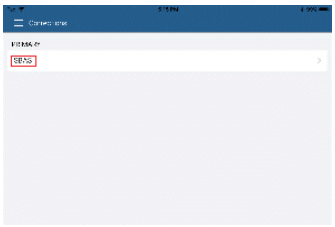


c. The app will then take you to the home screen where it will show your receiver status and estimated accuracy. Place the receiver in an unobstructed area with a clear view of the sky and let it rest for 2-5 minutes to establish a strong connection to satellites.

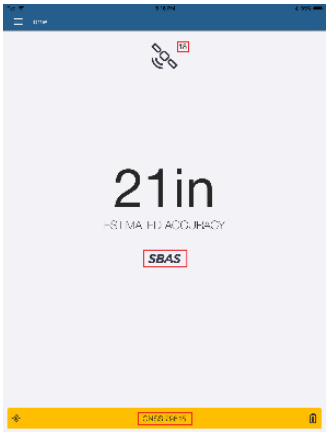
d. From the menu bar select “Preferences”. Under ‘Unit System’ select “US”.



e. From the menu bar select “Corrections” and under the ‘Primary’ option select “SBAS”.



f. Go back to the home screen and wait for “SBAS” to display under the estimated accuracy reading.



5. Go back to the tablet's home screen and launch the "Collector" app.

Sign into Collector

You will now need to sign on to your organization's account as follows: On the mobile device locate the Collector for ArcGIS and Tap the Icon

1. Select ArcGIS Online

2. Select Enterprise Login

3. Input **ch2mhill** and select continue

4. Select CH2M HILL
5. Follow the prompts using your **CH2M HILL Email and the PASSWORD you use to login to your computer every morning**. This will prompt Multi Factor Authentication. Verify yourself.

You should now be logged in.

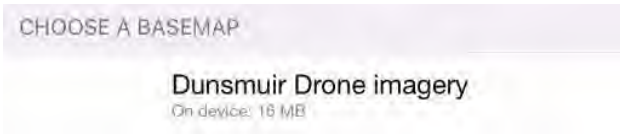
Access and Download Map

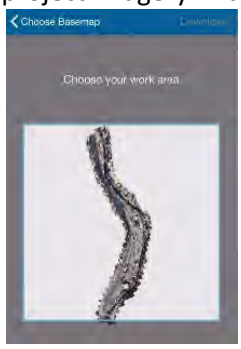
6. You will now see all the Collector maps you have access to. If you don't see the one you are looking for contact your GIS administrator. Search for maps or select a map from the group (project) you are working on.

7. Once you find the map you would like to use, you can click on the thumbnail to launch, or for offline use select the cloud icon to download the map to your device.

8. You will then need to zoom to your work area and orient it in the red box. Then press the "Map Detail" option at the bottom and zoom in until your reach your desired imagery detail. Select Download.

If you have project specific flown imagery loaded onto the ipad you would select project imagery. The "Map Detail" will automatically zoom into project imagery. Adjust extent if needed and select Download.





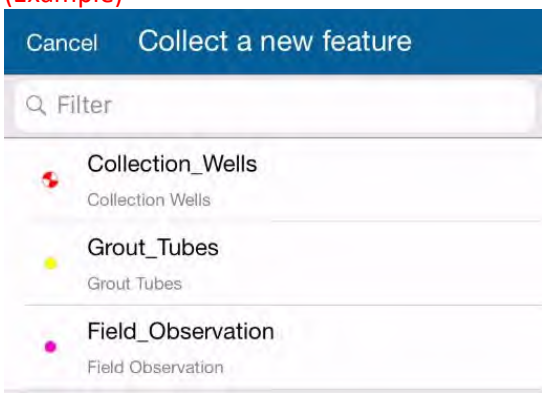
9. After downloading, open the map and allow Collector to access your location.

10. You can now begin using Collector.

Collect Data

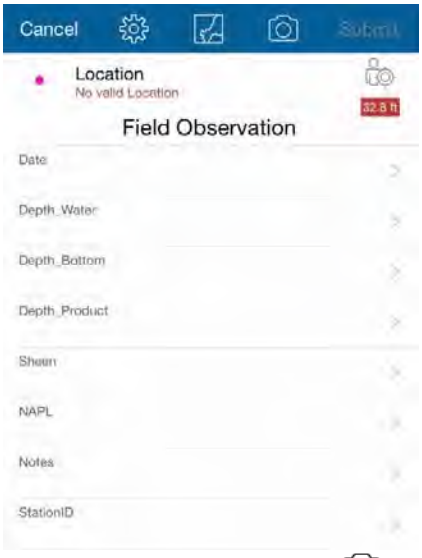
1. Tap **Collect New** + (should be on top bar on IPHONE or on the RIGHT SIDE OF IPAD SCREEN)
2. Select the feature you would like to collect

(Example)



3. Fill out the form by tapping attributes to edit their values. There might be a list of values to select from, a date to choose, or you might have to type in a value. Once you've provided the information, tap **Done** to return to the list of attributes and review the provided information.

(Example)

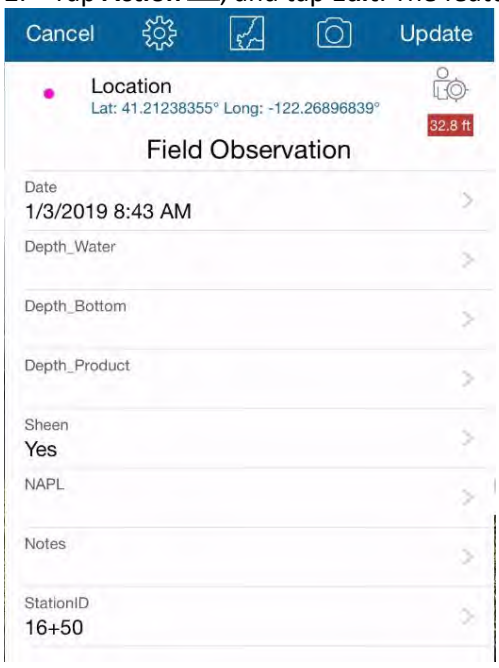


3. (optional) Tap **Attachments** and tap **Add** to add a photo. Tap a photo to **Rename**, **Download** or **Delete** it. Tap **Done** once all attachments are in place.
4. Tap **Map** to see the feature on the map.
5. Add the feature to the map:
    a. **[Points placed with GPS]** Tap **Use My Location** to update the point on the map to your current location and Collect your LAT/LONG.
    b. **[Lines and polygons created with GPS]** Tap **Use My Location** to use your current location to add a point to the feature. Continue until the feature is drawn.
    c. **[Lines and polygons created by streaming]** When ready to start streaming your location to create the feature, tap **Stream**.
    d. **[Lines and polygons created with a mix of manual and GPS]** Tap **Use My Location** to use your current location to add a point to the feature. Continue until the feature is drawn. You can also add a point by tapping on the map.
6. Tap **Collect Attributes** to return to the form, if needed.
7. Tap **Submit**.

To Edit/Update a Feature You Collected

1. Tap the feature on the map that you are updating.

2. Tap **Action**, and tap **Edit**. The feature's attributes are displayed.



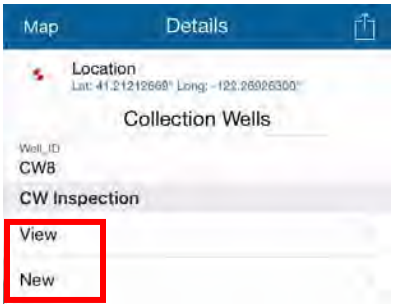
1. Update the information in the form by tapping attributes to edit their values. There might be a list of values to select from, a date to choose, or you might have to type in a value. Once you've provided the information, tap **Done** to return to the list of attributes and review the provided information.

2. (optional) Tap **Attachments** . Tap **Add** to add a photo. Tap a photo to **Rename**, **Download** or **Delete** it. Tap **Done** once all attachment changes are in place.
3. To change the feature's location or shape, tap **Map** and update the feature on the map:
    a. [Points updated with GPS] Tap **Use My Location** to update the point on the map to your current location.
    b. [Lines and polygons updated with GPS] Tap **Use My Location** to use your current location to add a point to the feature. Select and drag vertices to update the shape. Continue until the feature is complete.
    c. [Lines and polygons updated by streaming] To add your current location onto the feature, tap **Stream**.
    d. [Lines and polygons created with a mix of manual and GPS] Tap **Use My Location** to use your current location to add a point to the feature. Select and drag vertices to update the shape. You can also add a point by tapping on the map. Continue until the feature is complete.
4. Tap **Collect Attributes** to return to the form, if needed.
5. Tap **Update**.
   You'll see "Stored on device" and your updates are saved and ready to be synced.
6. You're returned to the map and can see your updated feature.

To Create/View Inspections

1. Tap the feature on the map that you are inspecting.

2. Tap **feature name** selected at bottom. The feature's attributes are displayed. Tap **View** to see past inspections or Tap **New** to create a new inspection.



3. Create a new inspection, update the information in the form by tapping attributes to edit their values. There might be a list of values to select from, a date to choose, or you might have to type in a value. Once you've provided the information, tap **Done** to return to the list of attributes and review the provided information.
4. (optional) Tap **Attachments** . Tap **Add** to add a photo. Tap a photo to **Rename**, **Download** or **Delete** it. Tap **Done** once all attachment changes are in place.

Delete a feature

On the map, tap the feature that you want to delete and tap **Action** . Tap **Delete** and tap **Yes** to delete it.

Sync Offline Maps

Sync updated data when you have been working with a map offline. You should sync only when you have good wifi connectivity.

1. Tap **All Maps** to go to the list of maps
2. If a count shows next to **Sync** you have local changes, so tap **Sync**
3. Wait for it to complete.

Sign out

Sign out of your account before you turn the device back in. Tap **Action** and tap **Sign Out**.

Problems?

Some field troubleshooting:

1. I can't sign in
   - Need to be connected (WIFI) for initial sign in.
   - Check for typos in your username and password. Your password is case sensitive.
   - Check for typos in your organization's URL.
2. Can't add a feature
   - Make sure it has a location (check the map).
   - Make sure you have permissions to edit the data (ask **Sarah Jenniges**).
3. Can't get GPS fix (no location available)
   - Go to an open area (away from trees, buildings, etc.) until get a location, then go back to the point of interest.
   - Make sure Collector can use your location (in your device's settings).
4. Get poor location accuracy message and can't collect a feature.

- Wait and see if accuracy improves.
- Lower your required accuracy (in Settings).

5. Sync failed
   - Make sure you have good connectivity.
   - Make sure you aren't in airplane mode.
   - Try enabling **Push Only** synchronization.
   - Specify a smaller preferred attachment size in Settings, delete images, and retake them.
6. My position consistently shows shifted from where I am
   - If shifted in the same direction, ensure the location profile is configured correctly.
   - If shifted randomly, check your location accuracy.

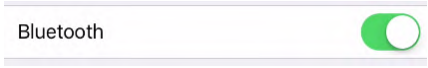
Quick reference: Map tools

1. Show list of maps available to you.
2. Go to your GPS location (tap again to remove your location point from the map).
3. See layer list, turn layers on and off.
4. Add a new feature (on the panel on iPad).
5. Measure (use Point & Shape Editing Tools).
6. Search for features (map dependent).
7. [iPhone] Show more tools.
8. Go to a pre-set bookmark.
9. Change the basemap.

Appendix I: Using the Trimble R1 GPS Receiver

R1 Receiver Set-up for iPad/iOS devices

1) Turn on iPad or other iOS devise and open the “settings” app
2) In the settings, click on ‘Bluetooth’
    a. Make sure Bluetooth is turned on/ enabled



3) Turn on the receiver by holding down the power button until both LED lights turn on and are green, then release power button. The lights may start flashing blue and yellow after you turn it on while it picks up satellites.

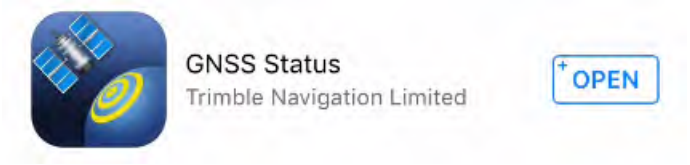
Image courtesy of Trimble Support

4) Next, place the R1 in pairing mode
    a. Hold the power button for roughly 5 seconds or so until the LED lights are blue.

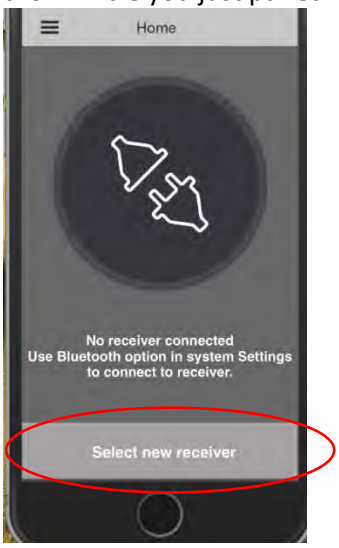
Image courtesy of Trimble Support

    b. In your Bluetooth settings, under “My devices”, the Trimble receiver name should pop up. (ex: “TrimbleGNSS 654995”)
    c. Click on the name to connect it. It may take a few seconds, but you should see the word “connected” next to the name once it is paired up correctly.

5) Close the settings app, and open the GNSS Status app
(this may need to be downloaded for free from the app store if it is not already included on your device.)

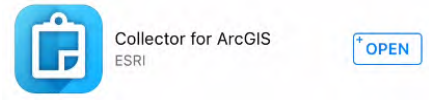


6) On the first screen that opens in the app, click on “Select new receiver” and then choose the name of the Trimble you just paired. (ex: GNSS Trimble 654995)



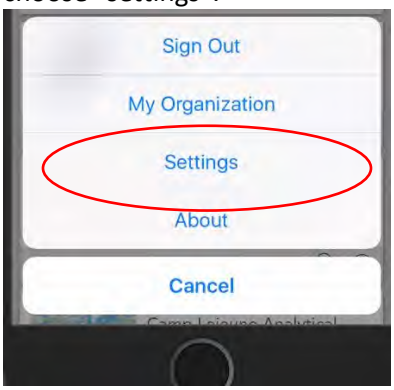
7) After a few seconds the receiver app should start picking up satellite signal and you should see an accuracy range appear on the screen, such as 120cm. This will fluctuate and eventually get smaller and more accurate, depending on where you are standing. *(Note: Like all Trimble/ GPS devices, if you are under heavy tree cover or near potential interference this may effect your accuracy.)*

8) Open the ArcGIS Collector App

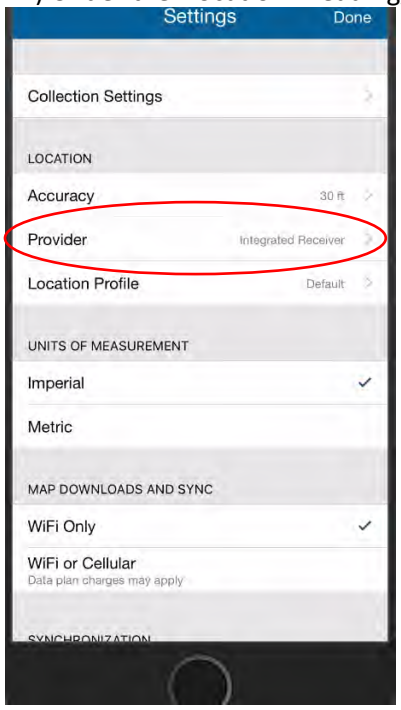


9) Sign into Portal or ArcGIS Online depending on your mapping needs.

10) In order to connect your R1 to collector *(so the collector app uses the R1’s accuracy instead of your IOS devices’ own integrated receiver)* click on the icon on the top blue bar of the app, and then choose “settings”.



11) Under the “location” heading, click on ‘Provider’ *(which should currently say ‘Integrated Receiver’)*



12) You should see two options on the next screen: “Integrated Receiver” *(which should have a check mark next to it)* and the ‘GNSS Trimble 654995’ name. Click on the Trimble R1 name.

13) Click the back to settings icon

to go back to the settings page. Under the same location heading, ‘Provider’ should now have the name ‘Trimble 654995’ next to it.

14) Click “Done” to return to the main collector page.

Troubleshooting note: You may need to hard-close the app and re-open it for the receiver to work. When you do this, the GNSS Status app will flash open while your collector screen is loading. This is a good sign that the two apps are talking to each other. ☺

15) Click on the map you want to use. *(If you are planning to run offline, click on the offline map stored on your device)* You should be able to click on the location icon at the top to get your location and the locator icon at the bottom should be green and showing your location accuracy under 5ft. *(Ideally 1 to 2ft).*

16) Begin data collection.

17) **To turn off R1**, hold the power button until both LEDs turn red. When the light is off and the battery light is red, release the power button and the R1 will switch off.

<u>Troubleshooting Notes:</u>

1) Lost R1 Bluetooth Connection

a. Keep the R1 receiver as close to your IOS devices as possible. Apple Bluetooth connections can be lost if you move your device too far away from your R1. If your connection is lost, you will see your R1 start flashing blue, saying it’s in pairing mode, and you will need to go to your IOS devise settings to re-pair it. You may also need to re select it in your GNSS Status app by clicking on “select new receiver”. (It’s good to check and make sure the GNSS Status app is connected before collecting any more data.)

b. If any apple product gets too hot, it can cause the bluetooth settings to be glitch or lose connection. Be sure to keep your equipment in the shade as much as possible.

c. Often if your iOS devise goes into “sleep” mode, the bluetooth setting will be lost. If this occurs, simply re-connect the R1 and check your GNSS status app.

2) If you have set up your receiver in the collector settings and you are unable to get your location to work

First try re-booting the collector app. If the GNSS screen does not flash when you open collector, then the two apps may not be communicating with each other. Check your Bluetooth settings and your GNSS status map to make sure the R1 is connected. If everything is connected, try opening collector back up and try getting your location again. *(Sometimes it will take 30 seconds or so for your location to show up.)* If it is still not working, then go back into your collector settings, and under the 'Provider' option (see step 11 above), click 'Integrated Receiver', so the check mark is next to it and not next to the Trimble name. Then swipe left on the 'Trimble 654995' name and choose 'delete'. Then hard close collector. Open it again, go back into settings and try re-adding the R1 (see steps 10-14 above). Hard close collector and re-open it. Open your map and your location should be working.

3) Additional R1/ GNSS settings to check if you are having location problems

Open the GNSS Status app. On the menu list *(this may show as an '☰' icon, or may be a list on the left, depending on your device)*, choose 'Receiver Options'. Make sure this is showing your R1 name ('GNSS Trimble 654995') as connected. Click on 'Real-time Config' and choose 'SBAS' if it is not already the default. Click on 'NMEA Settings' and move all of the list items from the right to the left using the double < icon at the bottom. Click 'save' (it may pop up a screen for you to choose a list of options such as ssp01, etc. Click on ssp01 and hit 'apply' and then hit 'save'.) Close out of the GNSS app then hard close the collector app and then re-open it. You may have to delete & re-add the R1 in the main settings again as well after this process.

3) R1 LED lights guide:

Bluetooth / GNSS LED ((•))

	ᛒ	GNSS
▮-▮-▮-▮	Pairing mode	N/A
▮---▮---▮---▮	✗	✗
▮---▮---▮---▮	✗	✓
▮---▮---▮---▮	✓	✗
▮------▮------▮------▮	✓	✓
▮ Solid	✓	✓ Corrected

Image courtesy of Trimble Support

Flashing blue = in pairing mode
Flashing blue and yellow = not pairing or getting GNSS satellites
Flashing blue and green = not pairing and is receiving GNSS satellites
Flashing yellow = paired, but no satellite connection
Flashing green = paired, receiving satellites but not correcting
Solid green = paired, receiving satellites and corrected

Power LED

Solid	<100%
Solid	100%
Off	>50%
--- --- ---	<50%
--- --- ---	<15%
- - - -	

Flashing yellow = less than 50% battery life
Flashing Red = less than 15% battery life
Red = Battery dead, needs to be charged
Image courtesy of Trimble Support
When charging, connected via USB, the battery icon light will be yellow.
When fully charged it will turn green.

STANDARD OPERATING PROCEDURE-5 (REVISED MARCH 2021)

5. Sediment Core Collection, Characterization, and Processing

5.1 Purpose

The purpose of this standard operating procedure (SOP) is to describe protocols for collecting, characterizing, and processing sediment cores, including sample collection.

5.2 Collection Procedure

Sediment core collection is performed by a qualified subcontractor that will deploy vibratory coring equipment (or vibracores) from an appropriately sized and outfitted work platform, such as a barge, pontoon boat, or other vessel. The vessel maneuvering, positioning, and sediment core collection will be performed by the qualified subcontractor in accordance with their standard procedures and specifications noted in a scope of work. Where possible, the sampling area will be probed with a thin metal rod prior to attempting to collect sediment cores. If the substrate or debris are believed to preclude core collect, the station will be offset. In shoreline areas that are exposed at low tide, cores may be collected using a backpack type vibracore. The specific approach will be determined in the field by the field team lead and the subcontractor and will consider all health and safety aspects.

The minimum following information must be recorded in the field log book by the project team representative on the vessel:

- Time and date of core collection
- Location coordinates (as-sampled)
- Surface water elevation (feet NAVD 88)
- Water depth
- Calculated sediment surface (mudline) elevation (feet NAVD 88)
- Core recovery (length of recovered core / length of core barrel penetration).
- Note any problems with core collection (e.g., number of attempts needed)
- Make note of any debris in area
- Note where signs of non-aqueous phase liquid (NAPL) begin and end (include olfactory, visual, and MultiRAE/photoionization detector [PID] observations)
- Any other pertinent information

If continuous vibracores are collected, they will be segmented into manageable lengths (e.g., 4 to 5 feet) capped, tapped, and labeled (location ID, a top/bottom designator, and indication of upper, middle, or lower core segment). This designation will be determined in the field by the field team leader. Cores will be retained upright on the sampling vessel until transfer to the shore-based processing crew.

Cores collected in stainless-steel tubes will be sealed, frozen on dry ice, and shipped to the laboratory.

5.3 Materials

The following materials are needed for core characterization, processing, and sampling:

- Plastic sheeting and duct tape
- Field table
- Standard coolers (for shipment of samples)
- Ice (for shipment of samples)
- Electric double cut shears
- Straight bladed knife and cut resistant gloves
- Measuring tape (engineering scale)
- Dry erase board and marker
- Digital camera
- Core logging form
- Munsell color chart
- Bottleware for samples
- Personal protective equipment (gloves, safety glasses, face shields)
- Disposable, dedicated single-use pans and scoops (either stainless-steel bowls and utensils, or aluminum pans and high-density polyethylene scoops)
- PID

5.3.1 Opening and Photographing the Core

Prepare the processing tables by covering them with plastic sheeting secured with heavy duty tape (e.g., duct tape). Place the bulk core segments on the table so that the top of the core is to the left and the bottom is to the right.

Once the core segments are arranged, cut slits in the taped caps approximately 0.75 inch wide on opposite sides of the liner and cut away a small triangular portion of the cap (enough to allow the shears to access the liner).

Make two parallel cuts for each segment on opposite sides of the core and then open the core so that it splits into two separate halves that lie flat on the processing table with the fresh sediment surfaces exposed.

Prior to field screening and visual characterization or the freshly exposed undisturbed sediment, take color photographs of the total core/sample length, using a whiteboard to record information about the core and the section being photographed (station ID, indication of depth interval, indication of top orientation, and date of core collection). Arrange tape measure next to core so that it will be visible in photos. Take photographs of the core in 1- to 3-foot increments. To ensure that sediment features and contaminant distribution (if evidenced) are visible in core/sample photographs, take photographs under bright natural light. Additional light sources may be needed when photographing cores indoors or under low lighting. If needed, additional close-up photographs should be taken of zones of interest, such as NAPL-bearing zones. Additional close up photographs may be warranted following detailed sediment core characterization. Review core/sample photographs prior to moving on to the next step in the procedure to

verify that photographs are in focus and bright, and have the appropriate contrast to capture the sediment characteristics of interested. Retake photographs if needed.

The contact between the accumulated soft sediment and the underlying native sediment should be identified (if present). Sample intervals should be delineated prior to the collection of PID readings and samples for chemical analysis.

5.3.2 Field screening

PID readings will be collected from each 1-foot interval. The readings will be collected from the mid-point of the sampling interval unless there is an area within the interval with a notable odor or staining. Note on the core log the interval from which the reading was collected. The reading will be taken from the headspace of a sealed plastic bag that is allowed to equilibrate for several minutes prior to obtaining the reading.

5.3.3 Sediment Core Characterization

The sediment cores will be visually characterized for sediment type, color, moisture content, texture, grain size and shape, consistency, visible evidence of staining, and any other observations. ***The observations recorded must be factual and accurate and must not contain any subjective conclusions.***

The Core Log Key (attached) is to be used for this characterization. The sediment will be described using the Unified Soil Classification System (modified slightly for sediment characterization) based visual-manual identification in accordance with ASTM-2488 standard practice.

The colors will be designated using a Munsell color chart. Sediment logs will be recorded on the Core Log Field Form (attached).

Odor: Olfactory observations (odors) or positive responses to an organic vapor detector will be recorded on the sediment core log.

Use the descriptors ***none, strong, moderate, or faint*** *to characterize odor.*

Examples of odors may include, but are not limited to, sulfur/sulfide, hydrocarbon, and fuel type odors.

Evidence of contamination: The following descriptors should be used to characterize any visible evidence of NAPL impact:

- **Stained** – Color (i.e., black or brown stained) is used to indicate that the soil matrix is stained a color other than the natural (unimpacted) color of the soil. Stained soil can be present in the absence of sheen or NAPL Stained or NAPL impacted soil (see below) may or may not be present with the staining. If NAPL or sheen is also present within the stained intervals these must be noted also as described below
- **Sheen** – Indicates a thin film at the surface of the pore water that has accumulated in core sleeve or in the pore spaces or at the surface of the sediment along any interval. The color of the sheen present should be noted using the following descriptors:
  - Silvery: metallic, silver gray in color
  - Rainbow: multicolored
  - Dark Rainbow: multicolored with some dark metallic or black/brown coloring

  - Dark metallic: reflects/mirrors the color of the sky or brown/black colored
- The intervals of observed sheen and the relative amounts should also be described as trace (<5%), slight (5-15%), moderate (15-40%), moderate to heavy (40-70%), and heavy (>70%). If the sheen is easily fractured upon touch or disturbance this should be noted also.
- **NAPL** – Any free phase NAPL observed in cores should be described in terms of color, distribution, and viscosity (if determinable) as described further below. Free phase product should be described as NAPL in the "Comments" column on the core logs. ***Do not draw any conclusions about the type of NAPL (e.g., oil, tar, fuel, coal tar, etc.).*** The following standard terminology should be used to describe the relative amount of NAPL impacts:
  - **Blebs** – These are observed discrete spheres or droplets of NAPL, but for the most part the sediment matrix is not visibly contaminated or saturated with NAPL.
  - **Coated** – Sediment grains are coated with NAPL. There is not sufficient free-phase material present to saturate the pore spaces.
  - **Saturated** – The entirety of the pore space for a given interval is saturated with free NAPL. Care should be taken to ensure that the pore spaces are saturated with NAPL rather than water if this term is used.

  If the NAPL is present in sufficient quantities, then the following ***viscosity modifiers*** should also be used in the description:
- High viscosity – Taffy-like (sticky)
- Viscous – molasses-like (thick liquid)
- Low viscosity – runny (thin liquid)
- **Non-liquid Tars** – If non-liquid contamination is observed it should be classified as soft or hard and its depth, color, size and general appearance should be recorded.

5.4 Sampling

Sediment cores will be sampled continuously, in 1-foot intervals, starting at the top of the core (that is, the mudline). After the cores have been described and the sample intervals have been determined, sediment will be collected and homogenized within each sample interval until uniform in color and texture and placed into appropriate sample containers for laboratory analysis. Samples from each interval will be collected and transferred to a single, decontaminated, clean stainless-steel compositing bowl. The samples will be field screened to remove gravel-sized material and unrepresentative material (e.g., woody debris) prior to field processing. Samples will then be thoroughly homogenized to a uniform appearance using a decontaminated, clean, stainless-steel spoon, and spooned into appropriate clean containers as identified in the Quality Assurance Project Plan (Appendix C of the Pre-Design Investigation Work Plan). Sediment samples for analysis of potentially volatile chemicals (chlorobenzene) will be placed immediately into sample containers and will not be composited or homogenized. Samples will be transported in coolers on ice to the field lab for packaging and shipment. Any remaining excess sample will be disposed of and the sampling equipment decontaminated before reuse.

5.5 Maintenance

The blades on the electric shears used for opening the cores should be changed when there is a noticeable difference in the amount of force required to advance the shears through the liner. These blades typically need to be changed after opening approximately 80 to 100 feet of core liner. Wear rates with different liner materials will vary. If too much time lapses between blade changes, the bearings within the cutting

head of the shears will begin to wear very quickly and will result in quite a bit of additional vibration. The shears should also be lubricated per the manufacturer directions when the blades are changed.

5.6 Precautions

There are several precautions that should be taken when handling, opening, and processing the sediment cores:

- When opening the cores, knives and/or other cutting instruments must be used. Cut resistant gloves should be worn under nitrile gloves when cores are opened.
- Staff opening cores should wear face shields; very wet sediment can spatter from the notched ends of the cores.
- The cut edges of the core liner can be very sharp and splinter like. Caution should be used and gloves should be worn when handling cut pieces of core liners.
- Sediment cores can be very heavy and awkward to move (due the shape of the core). The core collection team on the vessel will cut the cores to manageable lengths to the extent possible, but proper lifting technique and team lifting should be used when handling heavy cores.
- Do not transport dry ice or coolers containing dry ice in the cabin space of a vehicle due to danger of asphyxiation.
- Do not place liquid samples of groundwater and/or NAPL in the cooler with dry ice as the samples will freeze.

5.7 Attachments

- Generic Sediment Core Log Field Form (can be modified to include project-specific requirements)
- Core Log Key

End of SOP-5

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Site Name:
Project Number:
Project Location:

Core Log Part 1 - Collection Information

Station Information

Station/Core ID: _____ Target X: _____ Collection Date: _____

Vessel: _____ Target Y: _____ St. Arrival Time: _____

Captain: _____ X,Y Datum: *NAD83 Oregon St. Plane North* St.Depart Time: _____

Crew: _____ Tide Gage: _____ Target Core Depth: _____

_____ Tide Datum: _____ Coring Equip: _____

Notes by: _____ Depth Sounding Equip: _____ Barrel length (ft): _____

EPA Rep Onsite:

Core Collection Attempts

Attempt	Time	Actual X	Actual Y	Ft from Target	Water Depth (ft)	Time of water depth	Water Sur. Elev. (ft)	Mudline Elev. (ft) / Datum	Pene-tration (ft)	Rec. (ft)	% Rec.	Description of core collection	Core accepted?

Additional Collection Notes (debris, rip rap, probing information, material in catcher etc):

Description of Core Sectioning for Transport to Shore - Note the intervals that are sectioned (e.g., 0 to 5 feet, 5 to 10 feet) and note how much headspace is in each tube.

Core Log Part 2 - Characterization & Sampling

Sample Summary (check boxes for analysis) - See back page for Core Log form

Sample ID	Sample Type (N/FD/MSD)	Sample Date/Time	Depth Interval (ft)	No. Jars	PAHs	PCBs	Dioxin/Furans	Archive	Grainsize	TOC						
A																
B																
C																
D																
E																
F																
G																
H																
I																
J																
K																
L																
M																
N																
O																
P																
Q																
R																
S																
T																
U																

Station/Core ID: _____ Logged by: _____

Sampling Date: _____ Sampled by: _____

Sampling Time: Description below is as-recovered and does not include any correction for less than 100% recovery.

Recovered depth below mudline (ft)	Lithology (pattern)	Type (USCS Code)	Color (Munsell)	Consistency	Cementation	Structure	Moisture Content	Maximum particle size	Odor	% gravel	% sand	% fines	PID Reading (ppm)	Sample IDs (Single Letter - see page 1)		Comments
1																
2																
3																
4																
5																
6																
7																
8																
9																
10																
11																
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Reviewed by: Date:

Designation: D 2488 – 93

Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)[1]

This standard is issued under the fixed designation D 2488; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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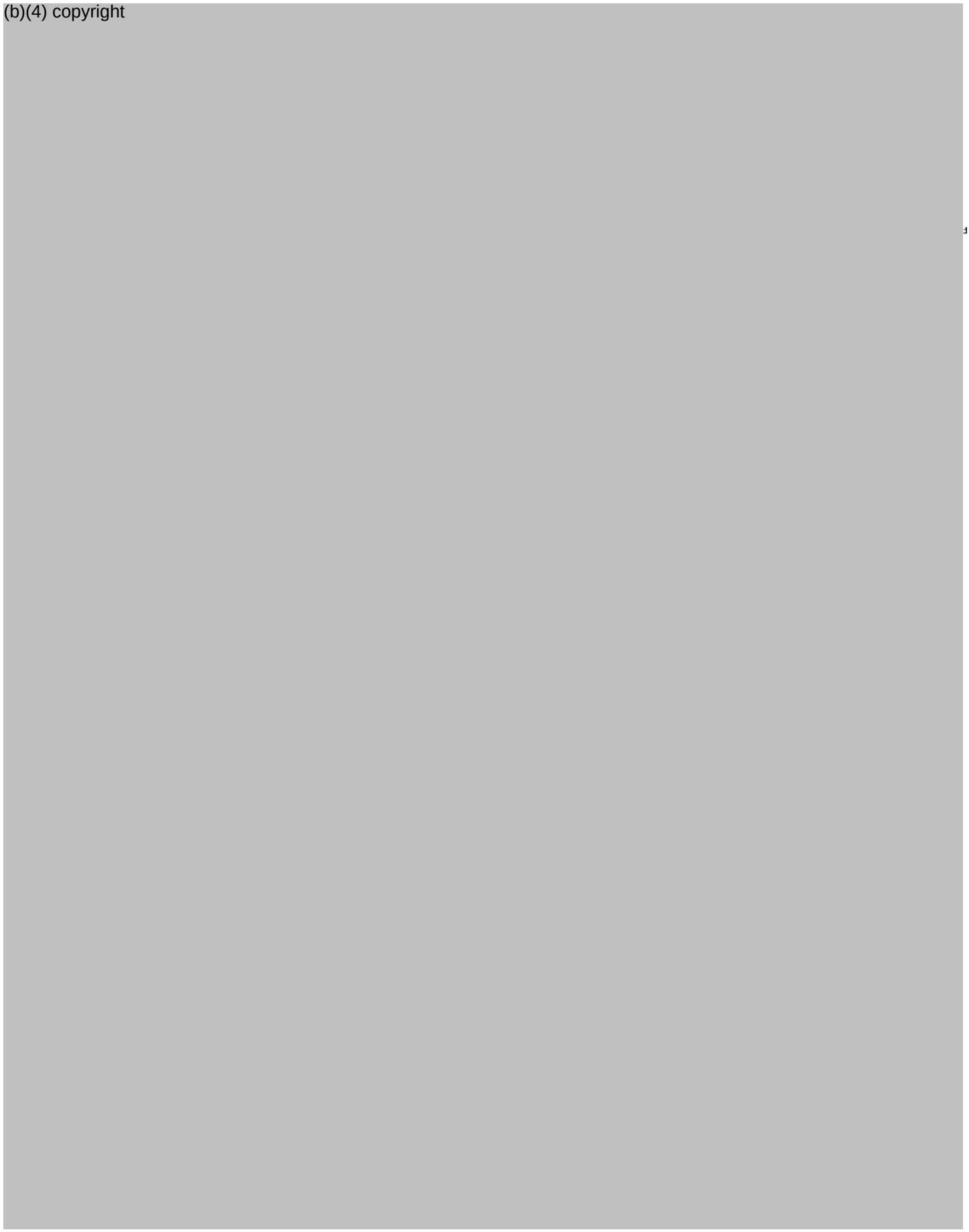
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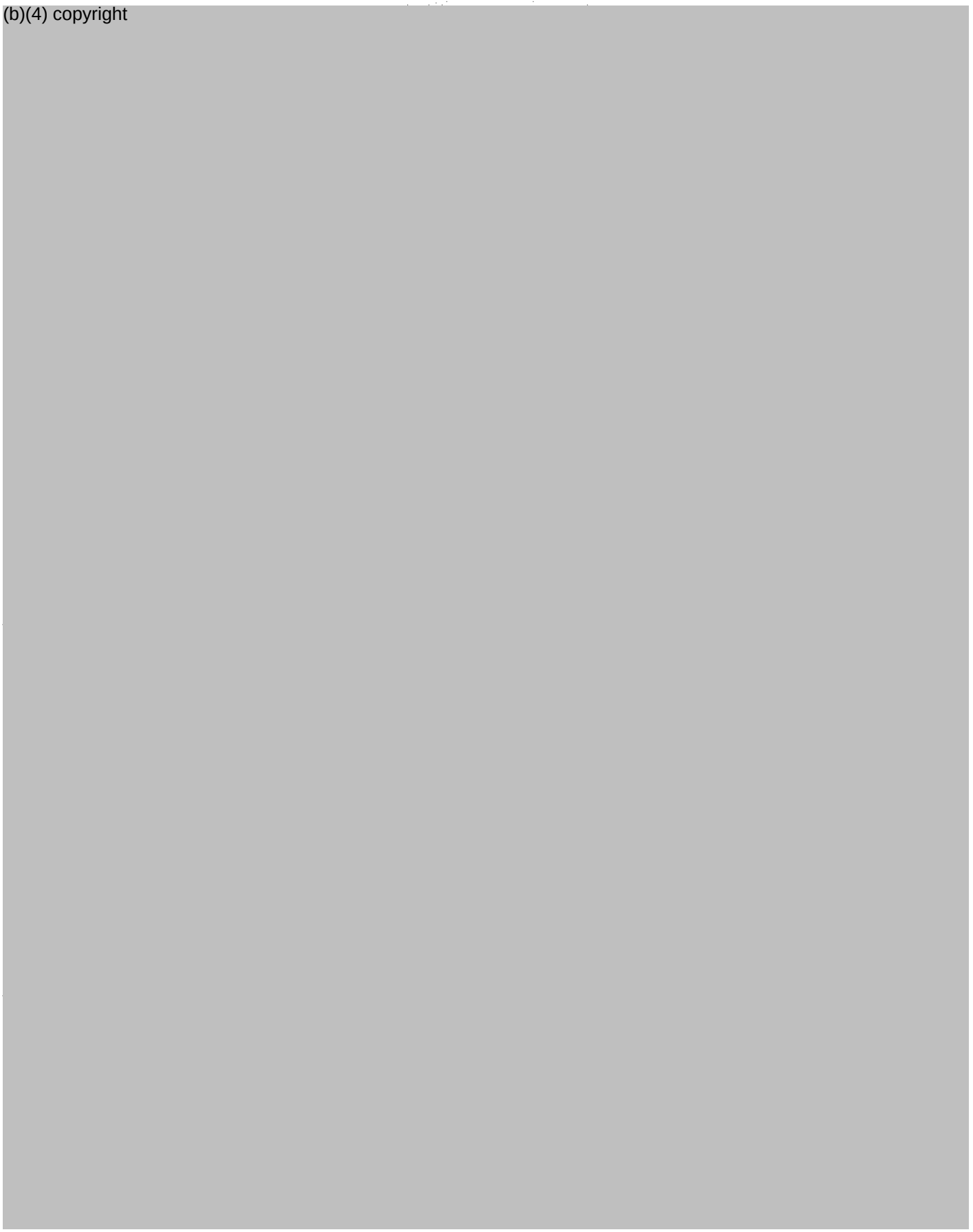
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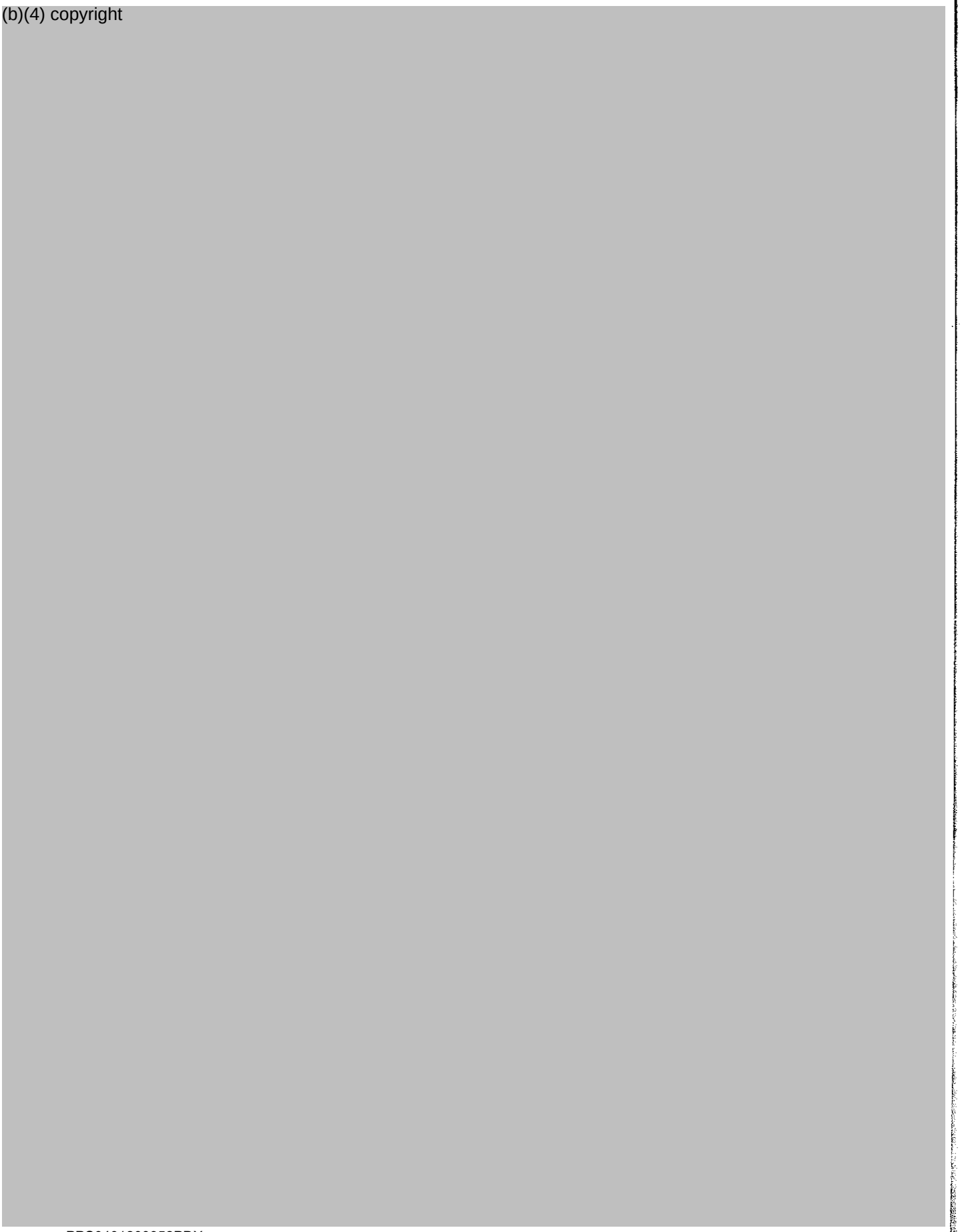

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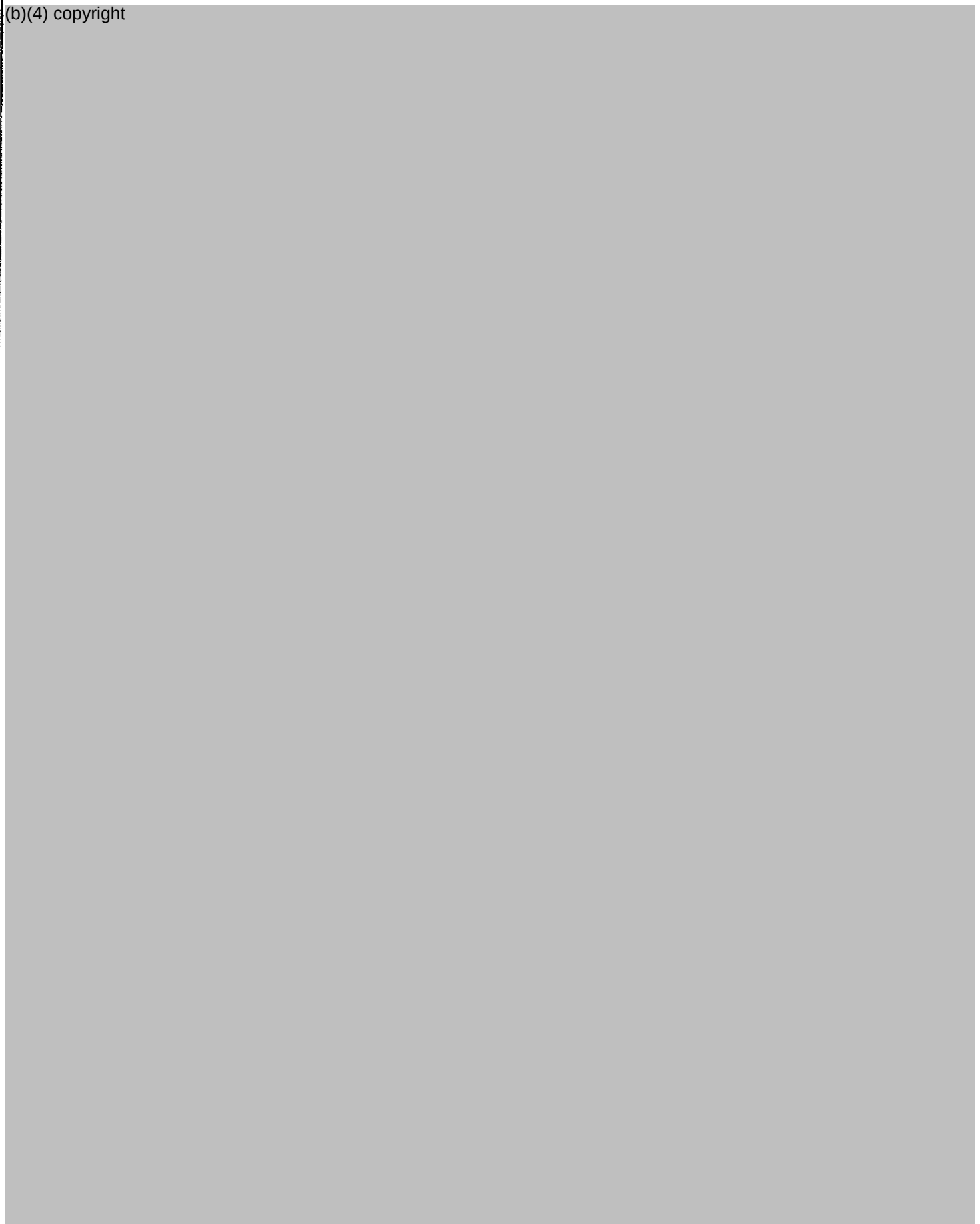
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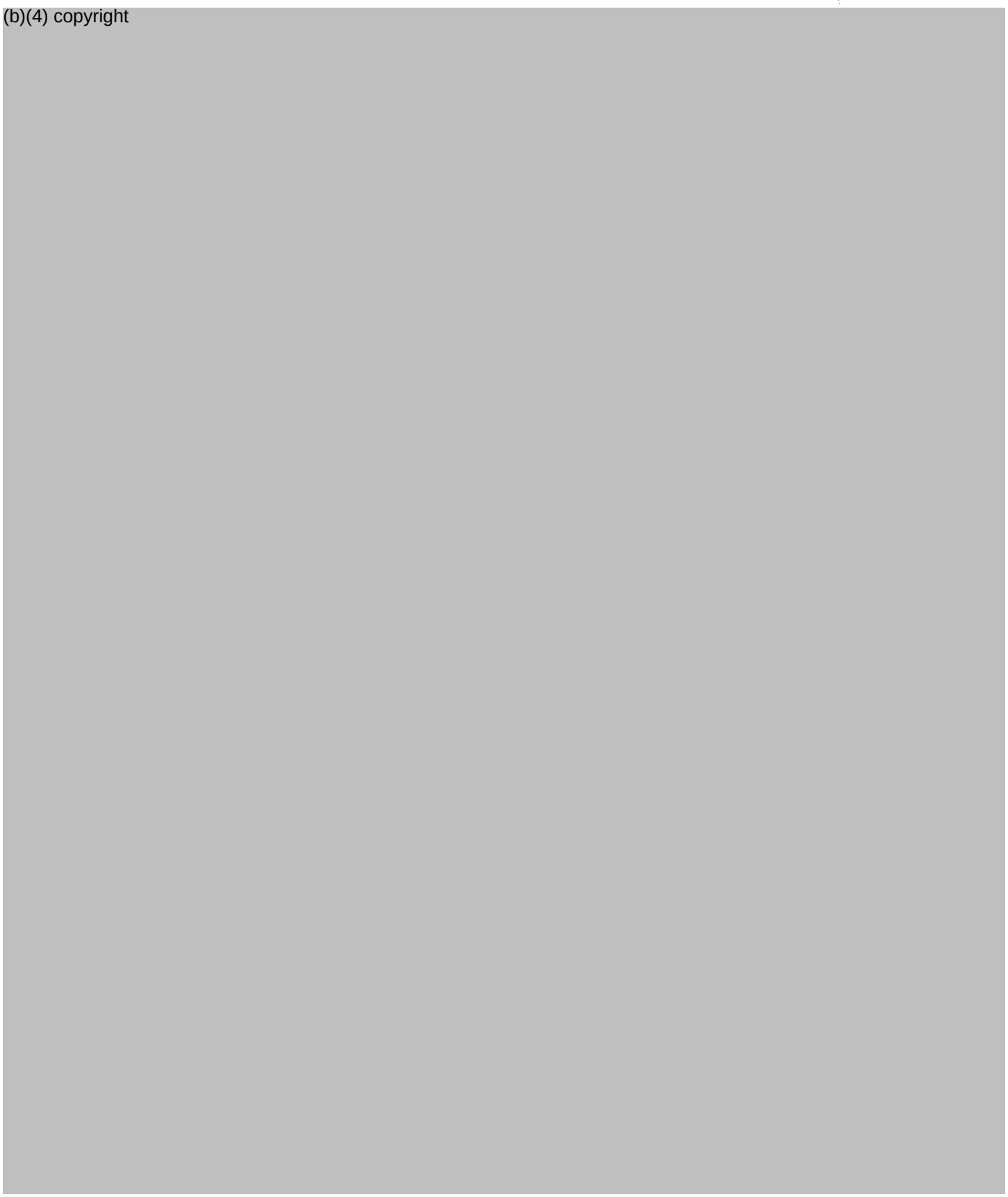
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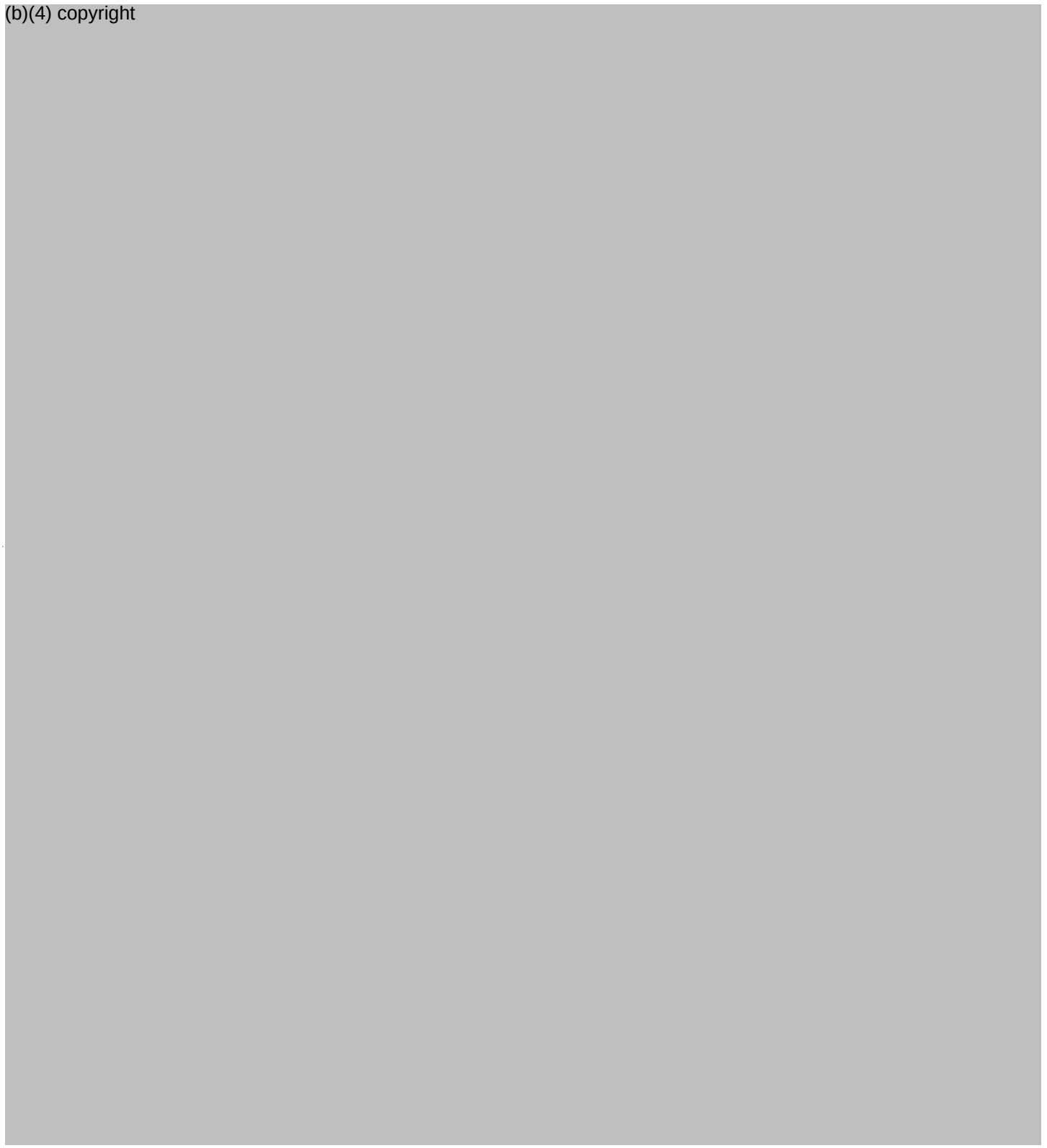

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STANDARD OPERATING PROCEDURE-6

6. Field Measurements of Air Quality Parameters

The purpose of this standard operating procedure (SOP) is to provide a general guideline for using a photoionization detector (PID) for detection of organic vapors. The operator's manual should be consulted for detailed operating procedures. PID readings will be collected from each 1-foot interval of collected sediment cores. Monitoring is for both safety and for obtaining information on possible contaminants.

6.1 Definitions

Terms are self-explanatory.

6.2 Associated Procedures

- None.

6.3 Equipment and Supplies

- PID (MultiRAE)
- Mix gas and Isobutylene calibration gases
- Calibration tubing and sampling-T

6.4 Calibration

Prior to each day's use, the PID will be calibrated using the provided standard calibration gasses.

6.4.1 Calibrating the Photoionization Detector

These instructions are specific to a MultiRAE from RAE Systems.

1) Turn on the PID by holding the power button and wait for the unit to cycle through the warm-up screens.
2) When the VOC sensor displays a reading, press and hold the "Power" and the "N/-" buttons simultaneously. When the calibration screen appears, press the arrow key and the "N/-" button to advance to the calibration menu.
3) Select "Fresh Air" to calibrate the sensors to fresh air. Note: Be sure you are in a well-ventilated area, free from any organic vapors.
4) Select "Multi Sensor Span" to calibrate the oxygen, lower exposure limit, carbon monoxide, and hydrogen sulfide sensors.
5) Connect the multi-gas canister to the PID using the calibration tubing and sampling-T. Note: Be sure to use the sampling "T" so that the gas is not forced into the PID pump. This can damage the unit. Never use master flex tubing as a substitute for the calibration tubing; master flex tubing can damage the lower exposure limit sensor.
6) Press "Start" on the PID and open the valve on the multi-gas canister. Leave the valve open until the PID has finished calibrating and has generated a reading for each sensor.

7) Record calibration reading for each sensor in the calibration log.
8) Select "Single Sensor Span," then "VOC" and repeat steps 5 through 7 using the isobutylene gas canister.

6.4.2 Measurement of Organic Vapors

Before approaching an area where air monitoring is required, ensure that the PID is on, that it has already cycled through the warm-up screens, and that all sensors are operational. Keep the device running while work is being performed and periodically take readings of the sediment core and in the breathing zone (area where you are actively working). If the sensors detect elevated (or deficient) gas concentrations, the PID will emit a tone, light up, and vibrate to alert the user of the conditions. One or more PIDs will be used at the following times:

- During core processing
- Periodically, when working over sediment cores

Record all readings in the field notebook.

6.5 Key Checks and Preventive Maintenance

- Calibrate meter
- Refer to operations manual for recommended maintenance
- Check batteries and have a replacement set on hand
- The field team should have a spare unit readily available in case of an equipment malfunction because of the importance of obtaining all of these parameters

6.6 Recordkeeping

The following calibration and operation information and each measurement made will be logged in the field logbook for quality assurance documentation:

- Instrument model and serial number, lamp if appropriate
- Calibration gas concentrations or values, including lot numbers and expiration dates
- Time of calibration
- Initial span setting (if appropriate)
- Actual reading
- Measurement conditions, breathing level (gases), in situ, or open container
- Comments on adjustments, cleaning requirements, and miscellaneous

End of SOP-6

STANDARD OPERATING PROCEDURE-7

7. Sample Management

The purpose of this standard operating procedure (SOP) is to delineate protocols for assuring representative environmental sample data by documenting the management of samples from time of collection through analysis and final disposition. This SOP includes information on selecting and using sample containers, how samples are handled in the field, chain-of-custody (CoC), and shipping procedures.

7.1 Definitions

- **Representative environmental data** is a term used to describe the degree to which data portray the true conditions at a specific location and time.
- **Sample management** is a set of procedures used to ensure the integrity of a sample is maintained. Stringent documentation of the implementation of these procedures is required to satisfy legal requirements if sample data are used in litigation.
- **Custody** is the physical control of an object, in this case, of an environmental sample.
- **CoC** refers to the process identifying those individuals who sequentially maintain physical custody of the sample.
- **CoC record** is the documentation of the CoC showing times, dates, and names of the individuals relinquishing and receiving the samples identified on the record.
- **Custody seals** are narrow strips of adhesive paper used to document that tampering with the sample cooler has not occurred from the time of collection to laboratory receipt.

7.2 Associated Procedures

- SOP-2: Field Administration and Records
- SOP-3: Photographs

7.3 Equipment and Supplies

- Sample containers
- Various preprinted forms (if used)
- Pens with indelible ink
- Preservatives, if not added prior to field work
- Sampling labels

7.4 Procedures

All samples will be collected as described by the Field Sampling Plan and the media-specific SOPs. The following procedures describe sample collection, handling, and custody procedures.

7.4.1 Sample Containers from the Laboratory

Sample containers should be received from the laboratory pre-labeled and with the preservative (where needed). The sample identification and date and time of sampling will be entered on the label immediately after collection. The containers will be left closed until needed for the sample.

7.4.2 Sample Collection and Handling in the Field

Following appropriate sample handling procedures will ensure samples arrive at the laboratory intact, at the proper temperature, and free of external contamination. Samples may be shipped to designated laboratories via FedEx or a courier service may be utilized. CoC procedures will be followed during any form of transport.

Any samples defined as hazardous materials will be packaged and shipped in accordance with requirements defined in the *Code of Federal Regulations* (CFR) (40 CFR 49, Chapter 1, Part 171). These requirements detail the proper classification and identification procedures for transporting hazardous materials.

When samples are required to be stored at 4 degrees Celsius or less, generous amounts of bagged ice will be packed with the samples. The ice will contact each sample and will be present at the top and bottom of the container. Samples will be cooled with ice or in a refrigerator (if available) before being packed for shipment.

The following procedures will be used to prevent bottle breakage and cross contamination:

- Sample bottles will be sealed in individual plastic bags.
- All samples will be transported inside appropriate containers.
- All 40 milliliter (mL) volatile organic analysis bottles will be placed in blocks of foam or in plastic mesh sleeves.
- All other glass bottles will be placed in plastic mesh sleeves to prevent glass to glass contact.
- Bagged ice and/or foam blocks will be used to separate glass bottles.
- The original CoC form will be packed inside the shipping container.
- The containers will be taped shut and sealed with signed custody seals.
- Samples that are known or suspected to be highly contaminated (based on field screening data or observation) will be packaged and shipped separately from other samples.
- Laboratories will be notified of any known or suspected highly contaminated samples. These samples will be stored separately from less contaminated samples to minimize the potential for contamination.

7.4.3 Sample Custody

Sample custody will be documented through the use of CoC forms. These forms will be used to track sample custody from the point of sample collection through sample disposal. Security of samples will be ensured by the use of the following procedures.

The field lead will maintain a supply of custody documents and will be responsible for inventory of serialized documents.

7.4.3.1 Field Custody

The field sampler is personally responsible for the care and custody of the samples collected until they are delivered to the laboratory. The field sampler will enter the sample information on the CoC forms in the field as each sample is collected.

Whenever samples are split with another party or government agency, the field sampler will note this split in the Remarks section of the CoC form. The note will indicate with whom the samples were split and will be signed by both the sampler and the recipient. If a representative is unavailable or refuses to sign, the sampler will note this in the Remarks section of the CoC form.

Sample custody during all sampling efforts must be traceable from the time of collection until the results are verified and reported by the laboratory and the samples are disposed. According to *NEIC Policies and Procedures* (NEIC 1987), a sample is under custody if:

- It is in your possession
- It is in your view, after being in your possession
- It was in your possession and you locked it up
- It is in a designated secure area

7.4.3.2 Chain-of-Custody Form

The CoC form is a vital document for all samples collected and must be properly completed. The form serves as a record of sample collection information, analysis requests, and sample tracking. It is a crucial record from the time of sample collection to final reporting and decision-making.

Field personnel will also log individual samples onto CoC forms when a sample is collected. These forms may also serve as the request for analyses. The samplers will sign the CoC form signifying they were the personnel who collected the samples. The CoC form will accompany the samples from the field to the laboratory. If the samples are given to another person for shipment, the sampler must transfer custody of the samples to that person. This transfer will be indicated on the CoC form. When the sample shipping container (cooler) is ready for shipment to the analytical laboratory, the name of the shipping company, along with the tracking number, will be documented on the CoC form, indicating a transfer of custody from the field to the shipping company. One copy of the CoC form will be retained by the field personnel while the original CoC form is placed in a plastic zip sleeve and taped to the inside of the sample shipping container (cooler) cover.

Each sample shipping container (cooler) must contain a CoC form that documents custody for all samples shipped in that container.

The electronic version of the CoC form will be sent by e-mail or fax to both the laboratory and the project manager to alert them that the samples are in transit to the laboratory.

The CoC form, sample labels, and field documentation are cross checked to verify sample identification, type of analysis, number of containers, sample volume, preservatives, collection time, and type of sample container.

The following information will be recorded on the CoC form:

- Project name and location
- Work number
- Project number

- Project manager's name
- Date and time of collection
- Sample identification unique for each sample
- Number of containers for each sample
- Sampler initials
- Analytical method(s) requested
- Sample volume (e.g., three 40-mL vials)
- Sample matrix (e.g., soil or groundwater)
- Preservative (e.g., hydrochloric acid)
- Request for matrix spike analysis or other quality control analysis
- Signature blocks for release and acceptance of samples
- Time blocks for release and acceptance of samples
- Any comments to identify special conditions or requests
- Laboratory name

Each CoC is assigned a unique alpha-numerical identifier by the laboratory upon receipt. Project and/or subcontractor staff will fill out CoC forms either electronically or by hand. The CoC forms will be sent or emailed to the site project manager.

7.4.3.3 Transfer of Custody

The CoC form must leave with the samples. When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the CoC. The time of relinquishment and receipt will be identical to document unbroken CoC. Notation of the bill of lading number and the signature of the shipping company agent on that bill of lading serve to keep the CoC intact during shipping.

7.4.3.4 Sample Custody During Shipment

Completion of sample CoC forms and sample packaging for shipment is performed in the supplier's staging area. Designated field and/or sample control staff will complete and verify CoC forms and pack samples for shipment. If samples are not shipped on the collection day, they will be refrigerated or stored on ice in the sample staging area.

7.4.4 Sample Packaging and Shipping

Collection of samples to be shipped offsite will be coordinated to ensure shipping can occur in a timely manner and that the holding times of the samples will not be exceeded. This condition may mean that sampling cannot occur after a certain hour of the day or on specific days of the week.

The protected sample containers will be placed into an ice chest with adequate ice for shipment to the laboratory. The seals will be placed over the left and right sides of the cooler cover. Each custody seal must be signed and dated. Clear tape should be placed over each custody seal to prevent the custody seals from easily tearing or falling off. The CoC, with the shipper's bill of lading number, will be placed in a plastic bag and taped to the inside of lid of the shipping container. Note: Several CoC forms and associated samples may be shipped in a single ice chest.

7.5 Recordkeeping

The CoC form will be scanned and sent by email to the field task lead, the project chemist, and the laboratory project manager upon shipment of the sample shipping containers (coolers). The original CoC form will be submitted by the laboratory, along with the final data packages. Any changes to the analytical requests on the CoC form or the laboratory purchase order will be made in writing to the laboratory. A copy of this written change will be sent to the project manager, laboratory, and placed in the project files. The reason for the change will be included in the sample log and project files so that recurring problems can be easily identified.

Following the completion of project activities, the sample logbooks, sample logs, and CoC forms will be scanned and saved on the project server.

Records of new, unused sampling containers will be retained in the field files. Information of whether preservatives are added at the laboratory, by the sample manager, or in the field will be recorded in the same locations as the sample identification.

The sample number will be recorded in all appropriate places, including the field logbook, the CoC, sample logbook, and each sample jar.

End of SOP-7

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Site Name:
Project Number:
Project Location:

Station ID: _____ Datum: _____

Sample Date: _____ Water Elevation: _____

Sampling Crew: _____ Station Arrival: _____

_____ Station Depart: _____

_____ Logged by: _____

_____ _____

_____ Collection method: _____

Vessel:

Attempt #	Dishtance from Target Center (ft)	Time	As-Sampled Coordinates		Water Depth (ft)	Recovery (cm)	Sample Interval (cm)	In composite?
			Easting	Northing				
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								

Sediment description (grain size, odor, color) for each grab:

Sample ID:

Analytical Suite:

Notes: Sample depth reported in field summary table will be average depth of the three grabs included in composite.

STANDARD OPERATING PROCEDURE-8 (REVISED MARCH 2021)

8. Sediment Grab Sampling

The purpose of this standard operating procedure (SOP) is to describe the collecting and handling of sediment grab samples.

8.1 Associated Procedures

- SOP-2: Field Logbooks
- SOP-7: Sample Management
- SOP-9: Decontamination – Wash and Rinse Method

8.2 Equipment and Supplies

- Pre-assembled sampling containers
- Field logbook and pens with indelible ink
- Stainless-steel bowl
- Stainless-steel spatula/spoon
- Spade or shovel
- Sediment sampler (e.g., Ponar or Ekman dredge or power grab sampler)
- Sediment coring device (tubes, drive head, drop hammer, "eggshell" check valve devices, acetate cores).
- Nylon rope or steel cable
- Bucket auger
- Large plastic tub or disposable food-grade aluminum pans
- Peristaltic pump

8.3 Procedures

8.3.1 Sample Collection

Suites of sample bottles will be pre-assembled. The volatile organic compound (VOC) bottles will be the first samples collected to minimize the loss of volatile compounds from the sample.

Sediment is to be collected either using a hand-held device such as a shovel or trowel or using an Ekman or Ponar grab. Following collection, the sediment sample is placed into a decontaminated container such as a stainless-steel bowl, homogenized, and placed into appropriate sample containers. **If VOC analyses are needed, sediment samples should be homogenized only after the portion for VOC analysis has been collected to avoid the loss of VOC contaminants** during the homogenization process.

Before sub-samples of the surficial sediments are taken, the overlying water must be removed. The preferred method of removing this water is by slowly siphoning it off near one side of the sampler. This can be done using a peristaltic pump or similar siphoning device. Methods such as decanting the water or slightly cracking the grab to let the water run out are not recommended, as they may result in unacceptable disturbance or loss of fine-grained surficial sediment and organic matter.

The field team will determine which of the following procedures will be used to collect the sediment samples given the various site conditions including sediment depth to refusal.

8.3.1.1 Hand Trowel or Shovel

The following procedures will be used for collecting sediment with a hand trowel or shovel:

- Remove the desired thickness and volume of sediment from the sampling area.
- Transfer the sample into a stainless-steel bowl.
- Decant overlying water.
- Collect VOC and gasoline-range organics (GRO) samples. For VOCs and GRO, a dedicated coring device (such as the laboratory provided plunger) should be used to transfer a predetermined amount of sediment into a pre-weighed sample container containing preservative, if possible.
- After the VOC fraction of sediment is collected, mix the remaining sediment thoroughly to obtain a homogenous sample representative of the entire sampling interval. A dedicated sampling scoop or trowel will be used to collect the sample material and place it into the appropriate sample container. Where possible, dedicated scoops or trowels are used to prevent the possibility of cross-contamination between sample stations as well as eliminating the need for any decontamination procedures between samples. The container cap will then be secured, and the sample will be preserved by immediately placing containers in a cooler with ice.

8.3.1.2 Sediment Sampler

This technique consists of lowering a sampling device to the sediment by use of a rope, cable, or extended handle. The mechanism is triggered, and the device entraps sediment in spring-loaded jaws, or within lever-operated jaws. The following procedures are used for collecting sediment samples with a sediment sampler such as an Ekman Bottom Grab Sampler, vanVeen . Ponar Dredge Sampler, or power grab sampler:

- Attach a sturdy nylon or steel cable to the hook provided on top of the dredge or grab sampler.
- Arrange sampler in the open position.
- Lower the sampler to a point just above the sediment surface.
- Drop the sampler sharply onto the sediment.
- Trigger the release mechanism to close the sampler.
- Raise the sampler and slowly decant any free liquid through the top of the sampler. Be careful to retain fine sediments.
- Inspect the grab for acceptability. If the surface shows signs of disturbance (channeling, loss of surface integrity), discard the sample. Sediment samples are considered acceptable if they meet the following criteria:
  - Sampler is not overfilled with sediment; the jaws are fully closed and the top of the sediment is below the level of the opening doors.
  - Overlying water is present and not excessively turbid.
  - In certain locations slight over-penetration (greater than 6 inches) may be accepted at the discretion of the Field Team Leader. Mild over-penetration may be accepted according to the following standards:
    - Sediment surface is intact on at least one side of the grab
    - Little or no evidence of surface sediment pushing through the grid surface of the grab (i.e., no visible imprint from the screening outside of that grid).

  - Illustrations of acceptable grabs are shown on Figure 1.
- Gently siphon off overlying water, taking care to not disturb the sediment sample.
- Upon removal of the overlying water, take a photograph of the grab, so that the sediment type is observable. A dry erase board should be utilized to include the Station ID, grab number, and date in the photo. The description of the sediment should be recorded in the field logbook.
- Open the dredge and transfer the sediment into a stainless-steel or high-density polyethylene bucket or a single use aluminum pan. Continue to collect additional sediment until sufficient material has been secured. Only collect sediment that has not been in contact with the sides of the sampling device for chemical analyses.
- Collect samples for VOC and GRO samples by inserting the coring device directly into the dredge. The remaining sample will then be placed into a stainless-steel bowl (or disposable pan) and homogenized using a dedicated stainless-steel scoop. Any extraneous material not considered to be relevant for analysis is removed from the sample during homogenization and any water present will be decanted. The sample is then placed into the appropriate size, pre-labeled sample containers for each analysis to be performed. The container lid(s) will then be secured, and the samples will be preserved by immediately placing the containers in a cooler on ice.
- If multi-location composite samples are needed, refer to the project-specific associated field sampling plan for instruction.

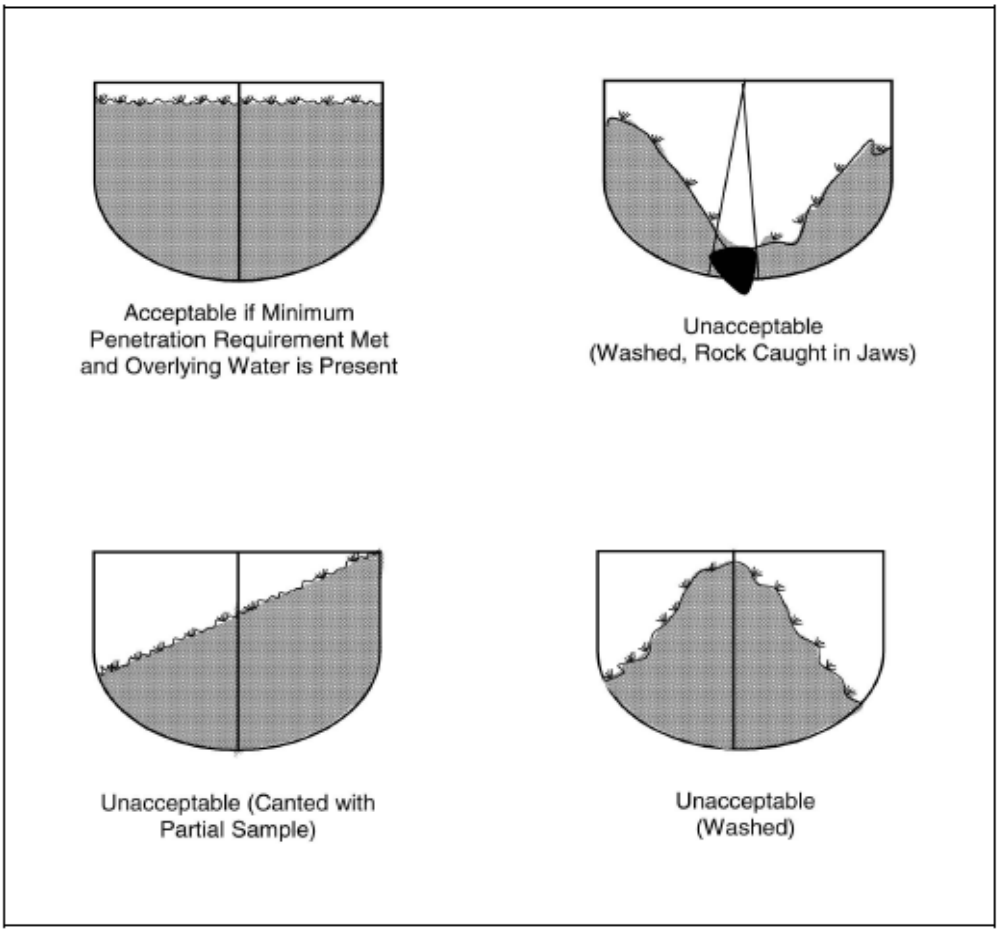


Figure 1. Depictions of Acceptable and Unacceptable Sediment Grab Samples (from EPA 2001)

8.3.1.3 Sample Hand Coring Device

A hand coring device consists of a coring device, handle, and acetate core. The following procedures are used for collecting sediment samples with a sample coring device:

- Assemble the coring device by inserting the acetate core into the sampling tube.
- Insert the "eggshell" check valve mechanisms into the tip of the sampling tube with the convex surface positioned inside the acetate core.
- Screw the handle onto the upper end of the sampling tube and add extension rods as needed.
- Place the sampler in a perpendicular position on the material to be sampled.
- This sampler may be used with either a drive hammer for firm consolidated sediments, or a "T" handle for soft sediments. If the "T" handle is used, place downward pressure on the device until the desired depth is reached. Rotate the sampler to shear off the core of the bottom and retrieve the device.
- Slide the acetate core out of the sampler tube. The acetate core may be lipped at both ends. The sample may be used in this fashion, or samples may be collected for various analytical parameters as described using hand tools.
- Collect samples for VOC and GRO samples by inserting the coring device directly into the down-hole end of the acetate core. The remaining sample will then be placed into a stainless-steel bowl (or disposable pan) and homogenized using a dedicated stainless-steel scoop. Any extraneous material not considered to be relevant for analysis is removed from the sample during homogenization and any water present will be decanted. The sample is then placed into the appropriate size, pre-labeled sample containers for each analysis to be performed. The container lid(s) will then be secured, and the samples will be preserved by immediately placing the containers in a cooler on ice.

8.3.1.4 Drive Hammer

If the drive hammer is selected, use the following procedures for collecting a sediment sample:

- Insert the tapered handle (drive head) of the drive hammer through the drive head.
- With left hand holding the tube, drive the sampler into the material to the desired depth. Do not drive the tube further than the tip of the hammer's guide.
- Record the length of the tube that penetrated the sample material, and the number of blows required to obtain this depth.
- Remove the drive hammer and fit the keyhole-like opening on the flat side of the hammer onto the drive head. In this position, the hammer serves as a handle for the sampler.
- Rotate the sampler at least two revolutions to shear off the sample at the bottom.
- Lower the sampler handle (hammer) until it just clears the two ear-like protrusions on the drive head and rotate about 90 degrees.
- Withdraw the sampler by pulling the handle (hammer) upwards and dislodging the hammer from the sampler.
- Slide the acetate core out of the sampler tube. The acetate core may be lipped at both ends. The sample may be collected in this fashion or samples may be collected for various analytical parameters as described using hand tools.
- Collect samples for VOC and GRO samples by inserting the coring device directly into the down-hole end of the acetate core. The remaining sample will then be placed into a stainless-steel bowl (or disposable pan) and homogenized using a dedicated stainless-steel scoop. Any extraneous material

not considered to be relevant for analysis is removed from the sample during homogenization and any water present will be decanted. The sample is then placed into the appropriate size, pre-labeled sample containers for each analysis to be performed. The container lid(s) will then be secured, and the samples will be preserved by immediately placing the containers in a cooler on ice.

8.4 Sediment Characterization

The sediment will be visually characterized for sediment type, color, moisture content, texture, grain size and shape, consistency, visible evidence of staining, and any other observations. The observations recorded must not contain any subjective conclusions about product type (i.e., non-aqueous phase liquid [NAPL] observations will consist of a description of the physical properties of the material including a description of odor as standardized below).

The sediment will be described using the Unified Soil Classification System (modified slightly for sediment characterization) based visual-manual identification in accordance with ASTM International Standard D2488.

The colors will be designated using a Munsell color chart. The information will be recorded in a field logbook or on a field form.

Prior to field screening and characterization, take color photographs of the top surface of the grab, using a whiteboard to record information about the sample and the section being photographed (station ID, indication of depth recovered, and date of collection). To ensure that sediment features and contaminant distribution (if evidenced) are visible in sample photographs, take photographs under bright natural light. Additional light sources may be needed when photographing cores indoors or under low lighting. If needed, additional close-up photographs should be taken of zones of interest, such as NAPL-bearing zones. Additional close up photographs may be warranted following detailed sediment characterization. Review sample photographs prior to moving on to the next step in the procedure to verify that photographs are in focus and bright, and have the appropriate contrast to capture the sediment characteristics of interested. Retake photographs if needed.

Odor: Olfactory observations (odors) or positive responses to an organic vapor detector will be recorded on the sediment core log.

Use the descriptors ***none, strong, moderate, or faint*** *to characterize odor.*

Examples of odors may include, but are not limited to sulfur/sulfide, hydrocarbon, and fuel type odors.

Evidence of contamination: The following descriptors should be used to characterize any visible evidence of NAPL impact:

- **Stained** – Color (i.e., black or brown stained) is used to indicate that the soil matrix is stained a color other than the natural (unimpacted) color of the soil. Stained soil can be present in the absence of sheen or NAPL Stained or NAPL impacted soil (see below) may or may not be present with the staining. If NAPL or sheen is also present within the stained intervals these must be noted also as described below
- **Sheen** – Indicates a thin film at the surface of the pore water that has accumulated in core sleeve or in the pore spaces or at the surface of the sediment along any interval. The color of the sheen present should be noted using the following descriptors:
  - Silvery: metallic, silver gray in color
  - Rainbow: multicolored

  - Dark Rainbow: multicolored with some dark metallic or black/brown coloring
  - Dark metallic: reflects/mirrors the color of the sky or brown/black colored
- The intervals of observed sheen and the relative amounts should also be described as trace (<5%), slight (5-15%), moderate (15-40%), moderate to heavy (40-70%), and heavy (>70%). If the sheen is easily fractured upon touch or disturbance this should be noted also..
- **NAPL** – Any free phase NAPL observed in cores should be described in terms of color, distribution, and viscosity (if determinable) as described further below. Free phase product should be described as NAPL in the "Comments" column on the core logs. ***Do not draw any conclusions about the type of NAPL (e.g., oil, tar, fuel, coal tar, etc.).*** The following standard terminology should be used to describe the relative amount of NAPL impacts:
  - **Blebs** – These are observed discrete spheres or droplets of NAPL, but for the most part the sediment matrix is not visibly contaminated or saturated with NAPL.
  - **Coated** – Sediment grains are coated with NAPL. There is not sufficient free-phase material present to saturate the pore spaces.
  - **Saturated** – The entirety of the pore space for a given interval is saturated with free NAPL. Care should be taken to ensure that the pore spaces are saturated with NAPL rather than water if this term is used.

  If the NAPL is present in sufficient quantities, then the following ***viscosity modifiers*** should also be used in the description:
    - High viscosity – Taffy-like (sticky)
    - Viscous – molasses-like (thick liquid)
    - Low viscosity – runny (thin liquid)
- **Non-liquid Tars** – If non-liquid contamination is observed it should be classified as soft or hard and its depth, color, size and general appearance should be recorded.

8.5 Recordkeeping

Records will be kept in the field logbooks and follow SOP-1 and SOP-2. Information to be recorded specific to this procedure includes the following:

- Sample location and number
- Date and time of collection
- Station coordinates (as-sampled)
- Water depth
- Make note of any debris in area
- Other information (e.g., odor or sheen produced when grab brought to surface or sectioned)
- Grab penetration
- Collection method used
- Sediment color, texture, odor, organic content, and grain size distribution
- Photoionization detector readings, if required by project
- Number of attempts needed or other issues with sediment recovery

- Sample Identification and analytical samples collected
- Designation of quality control samples, including any blanks, duplicates, or laboratory matrix spikes/matrix spike duplicates
- Other pertinent observations

8.5 Attachments

Generic Sediment Grab Field Form (can be modified to include project-specific requirements)

8.6 References

U.S. Environmental Protection Agency (EPA). 2001. Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual. EPA-823-B-01-002. October.

End of SOP-8

STANDARD OPERATING PROCEDURE-9

9. Decontamination – Wash and Rinse Method

The purpose of this standard operating procedure (SOP) is to delineate protocols for decontaminating relatively small, reusable sampling equipment. Decontamination of equipment and personal protective equipment is designed to ensure that the introduction and transfer of contamination is minimized. Equipment used for sediment sampling will be decontaminated using the wash and rinse method, prior to and after each use. This equipment includes shovels, trowels, grab samplers, and vibracore samplers. Decontamination wastes are containerized and transported offsite to a licensed treatment, storage, and disposal facility.

9.1 Definitions

- **Deionized water** is prepared by reverse osmosis.
- **Distilled water** is prepared by distillation. Note that deionized and distilled are often used interchangeably. The term deionized is used here, but in most instances distilled water also could be substituted.
- **Potable water** is water that is considered fit to drink or drinkable, usually obtained from a drinking water well or a public water supply system.

9.2 Associated Procedures

- SOP-5: Sediment Core Collection, Characterization and Processing
- SOP-8: Sediment Sampling

9.3 Equipment and Supplies

- Equipment or materials to be cleaned
- Tubs or other containers for wash and rinse solution
- Low-phosphate soap
- Bottled water (potable and deionized)
- Scrub brushes
- Spray bottles
- Methanol
- Alcohol
- Hexane
- Clean plastic wrap
- Containers for storing or moving residual waste products

9.4 Procedures

Equipment used for sediment sampling that is cleaned and reused in the field without returning to the decontamination pad will be decontaminated using the following procedure:

- Excess sediment will be rinsed from the equipment, to the extent possible, with site water and returned to the point of collection.

- The equipment will be scrubbed clean with the low-phosphate soap solution and a stiff, long-bristle scrub brush.
- The equipment will be rinsed with site water.
- If sheens are observed, the equipment may be spray rinsed with either methanol (solution containing less than 10 percent methanol) and hexane or deionized water, based on the target analytes, then allowed to drain into a container and air dry.
- The equipment will be placed on a clean surface.

To verify that the sampling devices have been decontaminated, equipment rinsate blanks will be collected. Equipment blanks consist of organic free reagent water to be poured into, over, or pumped through the sampling device. They will be collected in a sample container and submitted to the laboratory for analysis by the same analytical methods that the primary samples collected by the sampling device prior to decontamination were analyzed.

Any solvent containing waste, if generated during field decontamination, will be containerized and moved to the waste staging area. Smaller amounts of decontamination waste may be containerized separately in 55-gallon drums or in larger containers with compatible waste types.

9.5 Recordkeeping

Decontamination will be documented in the same logbook (site or function) used to document the use of the equipment. The documentation will include the following:

- Date and time
- Sampling location where the items became contaminated
- Method of decontamination
- Person completing and witnessing the decontamination procedure
- Items decontaminated

End of SOP-9

STANDARD OPERATING PROCEDURE-10

10. Waste Management

This standard operating procedure (SOP) describes procedures for analyzing and characterizing waste streams generated during investigation phase field work. This provides procedures for collection and analysis of waste characterization samples and is applicable to project activities that generate waste. This SOP supplements the requirements provided in the Project Waste Management Plan.

10.1 Definitions

- **Chain -of -custody form:** A form that documents the transfer of custody and responsibility from the sampling personnel to the laboratory. This form also documents the identification of the samples and the analyses requested for each sample.
- **Characterization:** The process where waste constituent concentrations are identified and compared to legal requirements or threshold levels that affect waste disposal methods. The generator of the waste is required by law to perform this process. The process may include evaluation of historical or safety data sheet (SDS) information and/or analytical sampling and analysis.
- **Detection limit:** The expression for the minimum value reported by the laboratory for an analytical test method for a given constituent. Other terms that may be used in consultation with the laboratory include practical quantitation limit, sample quantitation limit, method detection limit, and project -specific reporting limit.
- **Generator:** A generator is any person whose act or process produces waste or whose act first causes waste to become subject to regulation.
- **Field logbook:** A bound field notebook, with consecutively pre-numbered pages, that is clearly identified with the name of the project activity, the person responsible for maintenance of the notebook, and the beginning and ending dates of the entries.
- **Regulatory level:** A regulatory level is the legal standard or threshold level that determines a waste classification.
- **Sample label:** A label with adhesive backing that is affixed to each individual container containing the identification of the sample, preservatives applied to the sample, analytical method to be used, sampling date and time, and name of the sampler.
- **Transporter:** A transporter is engaged in the offsite transportation of waste by air, rail, highway, or water.

10.2 Associated Procedures

- SOP-1: Field Administration and Records
- SOP-2: Field Logbooks
- SOP-7: Sample Management
- SOP-9: Decontamination – Wash and Rinse Method

10.3 Equipment and Supplies

- Sampling supplies (ice chest, Ziploc bags, tape, ice)
- Nonreactive mixing container (to composite samples)

- Bailer
- Peristaltic or low-flow pump

10.4 Procedures

Following generation of liquid and solid waste, waste must be characterized in accordance with applicable regulations including:

1) Oregon Administrative Rule 340 – Divisions 100 through 106 – Oregon has adopted and incorporates by reference the Federal Resource Conservation and Recovery Act hazardous waste management program.
2) Oregon State-Only Waste Codes
3) 40 *Code of Federal Regulations* (CFR) Part 761.61(b) – Toxic Substances Control Act – Management of polychlorinated biphenyl waste.
4) Applicable, Relevant and Appropriate Regulations as provided in the Record of Decision.
5) U.S. Department of Transportation shipping requirements as provided in 49 CFR 172.

These regulations require the waste generator to properly characterize and classify waste streams to allow for appropriate management, treatment, and disposal. The following provide guidelines for collection and analysis of waste characterization samples.

Waste stream characterization should be documented in the project file. The following should be considered when assisting a generator with waste characterization:

- Waste characterization is made using generator knowledge and analytical testing. The generator is responsible for identifying the source of contamination and determining whether the source is a listed hazardous waste.
- Is there adequate information (generator knowledge or analytical results from previously collected samples) available to characterize waste before it is generated (e.g., results of in situ soil samples that can be used for characterization before the soil is excavated)?
- What analytical methods are needed to properly characterize the waste?
- Are waste streams exempt or excluded from regulation as a hazardous waste?
- Is the waste regulated as a universal waste or recyclable hazardous waste?
- Does the waste require treatment prior to land disposal?
- Can alternatives be identified to reduce the amount of waste generated or recycle the waste?
- What are the estimate waste volumes?
- Identify transportation, storage, and disposal facility (TSDF) sampling and analytical requirements.
- Identify appropriate licensed TSDFs for the waste streams to be generated.

Waste characterization requirements vary depending on the waste streams generated and the chemicals of concern associated with each site. Consult the Environmental Manager (EM) for assistance.

Sampling and analysis may be required when a waste stream cannot be properly characterized based on historical or process knowledge, to determine whether treatment is required prior to land disposal, or whether treatment is required pursuant to a legal requirement. The following outlines the key elements for

identifying analytical requirements to characterize waste and provides general guideline for collection of waste characterization samples. If analysis is required, the following elements should be considered:

- Selecting proper analytical methods and determining the necessary analytical reporting format
- Identifying proper sampling protocols

Waste characterization may require multiple analytical test methods, depending on the potential composition of the waste and regulatory requirements. Analytical test methods will depend on the following considerations:

- **Waste nature and quantity.** Evaluate the historical and process information related to waste generation. This information is usually obtained from the generator. Prior to collecting waste characterization samples, a review of historical records associated with past site practices, review of analytical results from previous investigations, and review of applicable SDSs should be performed. A determination should also be made by the generator regarding whether the waste is considered a listed hazardous waste. If sampling is required to determine waste characterization, an understanding of the known and expected constituents will help determine what analytical parameters are required. The quantity of waste to be generated will also be used to determine the number of waste characterization samples needed.
- **Regulatory Requirements.** Proper characterization of waste requires collection of representative waste characterization samples. The number of samples needed, sampling frequency, and sampling methodology should be considered in the planning phases of the work. Additionally, regulatory limits are established on the federal and state level that are used to classify the waste for treatment or disposal purposes. The generator is responsible for waste classification in accordance with applicable requirements. Consult the transportation and disposal (T&D) coordinator and EM regarding applicable waste characterization requirements.
- **Detection limits.** Consult with the project chemist to identify appropriate laboratories and confirm detection limits of the proposed test methods. Confirm that the identified laboratories can provide the necessary detection limits and that detection limits are less than federal and state waste limits. Confirm that analytical methods satisfy all the analytical requirements necessary to characterize and safely transport, treat, and/or dispose of the waste.
- **Disposal facility requirements.** Many disposal facilities have waste acceptance criteria that are in addition to the regulatory waste characterization requirements (e.g., Paint Filter Liquids Test [U.S. Environmental Protection Agency (EPA) Method 9095]). The waste acceptance criteria may affect the test method or number of samples required per volume of waste. Contact EM or T&D coordinator to evaluate proposed disposal facilities (including landfill and wastewater treatment plants) and to identify the waste acceptance criteria for each project waste stream. Waste originating from sites under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) requires disposal at a facility approved by EPA for offsite disposal of CERCLA waste.

Separate waste characterization samples of solid waste (primarily soil) and liquid waste will be collected and analyzed in accordance with the sample collection procedures provide below. Waste from different sites with similar chemicals of concern may be sampled as one waste stream. Consult with an EM or T&D coordinator prior to combining waste streams. A Level II data package will be required for the waste characterization and incidental waste stream samples.

Needed analyses will be determined by identifying chemicals of concern associated with the site or by requirements associated with the receiving facility. The following procedures will be used for liquids characterization.

Waste characterization samples will be collected from the soil or sediment waste containers prior to disposal; one composite sample and one grab for volatile organic compounds (VOCs) and total petroleum hydrocarbons (TPH) will be collected per 10 drums, and one sample will be collected per portable tank or container per location and/or area. Samples from drums will be collected from the container with the highest photoionization detection/flame ionization detection reading.

Composite samples will be collected by selecting five random aliquots from each container using a using a stainless-steel or disposal sampling utensil. The aliquots will be paced into a stainless steel-bowl and homogenized by using quartering techniques. Sample containers should be filled completely. Close, label, and package the sample container for shipment to the laboratory.

Water samples will be collected as follows (one from every 10 drums, and one sample will be collected per portable tank or container per location and/or area) and will be analyzed for the parameters listed in Table 10-1.

1) Using a bailer or dip jar, collect a water sample from its containment.
2) Fill the sample containers for VOC and TPH gasoline-range organics (GRO) analysis first (grab sample). Fill the 40-milliliter (mL) vials so that there is no headspace in each vial.
3) Fill the sample containers for the remaining analyses.
4) Label and package the samples for shipment to the laboratory.

A sampling plan should be developed to ensure analysis is based on representative samples. Consult the EM and project chemist to determine sampling requirements.

Standards for documenting field sampling activities, labeling the samples, documenting sample custody, and completing the chain-of-custody form are provided in SOP-7. The standards presented in this SOP will be followed to ensure that samples collected are maintained for their intended purpose and the conditions encountered during field activities are documented. This procedure will apply to all sample collection conducted during field activities.

Table 10-1. Sample Containers, Preservation, and Holding Times – Aqueous Waste Characterization

Analyte	Method	Container and Minimum Quantity	Preservation	Holding Time
Metals	SW6010B/ SW7470A	1-L/P, G	Nitric acid to pH < 2; chill to < 6°C (field filter prior to acid preservation if dissolved metals required)	28 days for mercury; 180 days for all other metals
TPH-DRO/RRO	SW8015B	2 × 250-mL/ G-TLC	Cool < 6°C	7 days to extraction; 40 days to analysis
SVOCs	SW8270C	2 × 1,000-mL/G, AG	Cool < 6°C	7 days to extraction; 40 days to analysis
VOCs	SW8260B	3 × 40-mL/G-TLC	Hydrochloric acid to pH < 2; chill to < 6°C; no headspace	14 days (preserved); 7 days (unpreserved)
TPH-GRO	SW8015	3 × 40-mL/G-TLC	Hydrochloric acid to pH < 2; chill to < 6°C; no headspace	14 days (preserved); 7 days (unpreserved)

Table 10-1. Sample Containers, Preservation, and Holding Times – Aqueous Waste Characterization

Analyte	Method	Container and Minimum Quantity	Preservation	Holding Time
Pesticides	SW8081A	2 × 1,000-mL/G, AG	Cool < 6°C	7 days to extraction; 40 days to analysis
Herbicides	SW8151A	2 × 1,000-mL/G, AG	Cool < 6°C	7 days to extraction; 40 days to analysis
Corrosivity	SW9045C	1 × 500-mL/P	Cool < 6°C	Immediately
Ignitability	SW1010	1 × 500-mL/P	None	Not applicable

Notes:
Waste samples are not validated, methods listed above may not be included in quality assurance/quality control tables

°C = degree(s) Celsius
AG = amber glass
G = glass
G-TLC = glass with Teflon-lined cap
L = liter(s)
P = polyethylene
SVOC = semivolatile organic compound

Table 10-2. Sample Containers, Preservation, and Holding Times – Solid Waste Characterization

Analyte	Method	Container and Minimum Quantity	Preservation	Holding Time
TCLP Metals	SW1311/SW6010B/ SW7470A	4-oz, G	None	180 days to leaching (28 days for mercury); then 28 days for mercury; 180 days for all other metals
TPH-DRO	SW8015	4-oz, G	Cool < 6°C	14 days to extraction; 40 days to analysis
TCLP SVOC	SW1311/SW8270C	4-oz, G	Cool < 6°C	14 days to leaching, then 14 days to extraction; 40 days to analysis
TCLP VOCs	SW1311/SW8260B	4-oz, G	Cool < 6°C; no headspace	48 hours to leaching, then 7 days to analysis (unpreserved) or 14 days to analysis (preserved)
TPH-GRO	SW8015	4-oz, G	Cool < 6°C; no headspace	14 days (preserved in methanol); 48 hours (unpreserved)
TCLP Pesticides	SW1311/SW8081A	4-oz, G	Cool < 6°C	14 days to leaching, then 14 days to extraction; 40 days to analysis

Table 10-2. Sample Containers, Preservation, and Holding Times – Solid Waste Characterization

Analyte	Method	Container and Minimum Quantity	Preservation	Holding Time
TCLP Herbicides	SW1311/SW8151A	4-oz, G	Cool < 6°C	14 days to leaching, then 14 days to extraction; 40 days to analysis
PCBs	SW8082	4-oz, G	Cool < 6°C	14 days to extraction; 40 days to analysis
Corrosivity	SW9045C	4-oz, G	Cool < 6°C	28 days
Ignitability	SW1010	4-oz, G	None	Not applicable

Notes:
Waste samples are not validated, methods listed above may not be included in quality assurance/quality control tables

°C = degree(s) Celsius
G = glass
oz = ounce(s)
SVOC = semivolatile organic compound
TCLP = toxicity characteristic leaching procedure

End of SOP-10

STANDARD OPERATING PROCEDURE-11

11. Shallow Soil Sampling

11.1 Purpose

To provide general guidelines for the collection and handling of surface soil samples during field operations.

11.2 Scope

The method described for surface soil sampling is applicable for loosely packed earth and is used to collect disturbed-soil samples.

11.3 Equipment and Materials

- Sample jars
- A hand auger or other device that can be used to remove the soil from the ground. Stainless-steel is preferred; however, split spoons, which are most commonly available in carbon steel, are acceptable for use only if they are not rusty.
- A stainless-steel spatula or disposable plastic scoop should be used to remove material from the sampling device.
- Unpainted wooden stakes or pin flags
- Fiberglass measuring tape (at least 200 feet in length)
- Global positioning system (GPS) Unit (if available)

11.4 Procedures and Guidelines

1) Wear protective gear, as specified in the Health and Safety Plan.
2) To locate samples, identify the correct location using the pin flags or stakes. Proceed to collect a sample from the undisturbed soil adjacent to the marker following steps C and D. If markers are not present, the following procedures will be used.
    a) For samples on a grid:
        i) Use measuring tape to locate each sampling point on the first grid line as prescribed in the sampling plan. As each point is located, drive a numbered stake in the ground and record its location on the site map and in the logbook.
        ii) Proceed to sample the points on the grid line.
        iii) Measure to location where next grid line is to start and stake first sample. For subsequent samples on the line take two orthogonal measurements: one to the previous grid line, and one to the previous sample on the same grid line.
        iv) Proceed to sample the points on the grid line as described in Section C below.

v) Repeat 1c and 1d above until all samples are collected from the area.

vi) Or, a GPS unit can be used to identify each location based on map coordinated, if available.

b) For non-grid samples:

i) Use steel measuring tape to position the sampling point at the location described in the sampling plan by taking two measurements from fixed landmarks (e.g., corner of house and fence post).

ii) Note measurements, landmarks, and sampling point on a sketch in the field notebook, and on a site location map.

iii) Proceed to sample as described in Section C below.

iv) Repeat 2a through 2c above until all samples are collected from the area.

v) Or, a GPS unit can be used to identify each location based on map coordinated, if available.

3) To the extent possible, differentiate between fill and natural soil. If both are encountered at a boring location, sample both as prescribed in the field sampling plan. Do not locate samples in debris, tree roots, or standing water. In residential areas, do not sample in areas where residents' activities may impact the sample (e.g., barbecue areas, beneath eaves of roofs, driveways, garbage areas). If an obstacle prevents sampling at a measured grid point, move as close as possible, but up to a distance of one half the grid spacing in any direction to locate an appropriate sample. If an appropriate location cannot be found, consult with the Field Team Leader and Project Manager for further direction.

4) To collect samples:

a) Use a decontaminated stainless-steel scoop/trowel or disposable plastic scoop to scrape away surficial organic material (grass, leaves, etc.) adjacent to the stake. New disposable scoops or trowels may also be used to reduce the need for equipment blanks.

b) If sampling:

i) Surface soil: Obtain soil sample by scooping soil using the augering scoop/trowel, starting from the surface and digging down to a depth of about 6 inches, or the depth specified in the workplan.

ii) Subsurface soil: Obtain the subsurface soil sample using an auger down to the depths prescribed in the field sampling plan.

c) Take a photoionization detector reading of the sampled soil if organics are anticipated to be present and record the response in the field notebook. Also record lithologic description and any pertinent observations (such as discoloration) in the logbook.

d) Empty the contents of the scoop/trowel into a decontaminated stainless-steel pan or dedicated sealable bag.

e) Repeat this procedure until sufficient soil is collected to meet volume requirements.

f) For Target Compound List (TCL) VOC aliquots, fill sample jars directly with the trowel or scoop or specialized sampling equipment (i.e., Encore® or Terra Core® sampler) and cap immediately upon filling. DO NOT HOMOGENIZE.

g) For TCL pesticides/polychlorinated biphenyls and SVOCs, Target Analyte List (TAL) metals, and field X-ray fluorescence aliquots, homogenize cuttings in the pan using a decontaminated stainless-steel utensil.

h) Transfer sample for analysis into appropriate containers with a decontaminated utensil.

i) Immediately upon collection, all samples for chemical analysis are to be placed in a closed container on ice unless it is not possible to do so. Although unusual and uncommon, there may be instances where it is not possible to have containers with ice at the sample location. In these instances, the samples should be placed on ice as soon as practical and during the time between collection and placing the samples on ice, the samples should be kept as cool as possible

j) Backfill the hole with soil removed from the borehole. To the extent possible, replace topsoil and grass and attempt to return appearance of sampling area to its pre-sampled condition. For samples in non-residential, unmowed areas, mark the sample number on the stake and leave stake in place. In mowed areas, remove stake.

11.5 Attachments

None.

11.6 Key Checks and Items

- Use phthalate-free latex or surgical gloves and other personal protective equipment.
- Transfer volatiles first, avoid mixing.
- Decontaminate utensils before reuse, or use dedicated, disposable utensils.

End of SOP-11

STANDARD OPERATING PROCEDURE-12 (REVISED MARCH 2021)

12. Logging of Soil Borings

12.1 Purpose and Scope

This standard operating procedure (SOP) provides guidance to obtain accurate and consistent descriptions of soil characteristics during soil-sampling operations. The characterization is based on visual examination and manual tests, not on laboratory determinations.

12.2 Equipment and Materials

- Indelible pens
- Tape measure or ruler
- Field logbook
- Spatula
- Hydrochloric acid (HCL), 10 percent solution
- Squirt bottle with water
- Rock- or soil-color chart (e.g., Munsell)
- Grain-size chart
- Hand lens
- Unified Soil Classification System (USCS) index charts and tables for soil classification (attached)

12.3 Procedures and Guidelines

This section covers several aspects of soil characterization: instructions for completing the soil boring log form (attached), field classification of soil, and standard penetration test procedures.

A. Instructions for Completing Soil Boring Logs

Soil boring logs will be completed in the field logbooks or on separate soil boring log sheets. Information will be collected using a standard soil boring log form (attached) or an equivalent form that supplies the same information. The information collected in the field to perform the soil characterization is described below.

Field personnel should review completed logs for accuracy, clarity, and thoroughness of detail. Samples also should be checked to see that information is correctly recorded on both sample jar labels and on the log sheets.

B. Heading Information

Boring/Well Number. Enter the boring/well number. A numbering system should be chosen that does not conflict with information recorded for previous exploratory work done at the site. Number the sheets consecutively for each boring.

Location. If station, coordinates, mileposts, or similar project layout information is available, indicate the position of the boring to that system using modifiers such as “approximate” or “estimated” as appropriate.

Elevation. Elevation will be determined at the conclusion of field activities through a survey.

Drilling Contractor. Enter the name of the drilling company and the city and state where the company is based.

Drilling Method and Equipment. Identify the bit size and type, drilling fluid (if used), and method of drilling (e.g., rotary, hollow-stem auger, sonic). Information about the drilling equipment (e.g., CME 55, Mobile B61) should also be noted.

Water Level and Date. Enter the depth below ground surface to the apparent water level in the borehole. The information should be recorded as a comment. If free water is not encountered during drilling or cannot be detected because of the drilling method, this information should be noted. Record date and time of day (for tides, river stage) of each water level measurement.

Date of Start and Finish. Enter the dates the boring was begun and completed. Time of day should be added if several borings are performed on the same day.

Logger. Enter the first and last name.

C. Technical Data

Depth Below Surface. Use a depth scale that is appropriate for the sample spacing and for the complexity of subsurface conditions.

Sample Interval. Note the depth at the top and bottom of the sample interval.

Sample Type and Number. Enter the sample type and number, (e.g., SS-1 = split spoon, first sample). Number samples consecutively regardless of type. Enter a sample number even if no material was recovered in the sampler.

Sample Recovery. Enter the length of the soil sample recovered from the sampler to the nearest 0.1 foot. Often, there will be some wash or caved material above the sample; do not include the wash material in the measurement. Record soil recovery in feet.

Standard Penetration Test Results (if applicable). In this column, enter the number of blows required for each 6 inches of sampler penetration and the "N" value, which is the sum of the blows in the middle two 6-inch penetration intervals. A typical standard penetration test involving successive blow counts of 2, 3, 4, and 5 is recorded as 2-3-4-5 and (7). The standard penetration test is terminated if the sampler encounters refusal. Refusal is a penetration of less than 6 inches with a blow count of 50. A partial penetration of 50 blows for 4 inches is recorded as 50/4 inches. Penetration by the weight of the slide hammer only is recorded as “WOH.”

Samples should be collected using a 140-pound hammer and 2-inch diameter split spoons. Samples may be collected using direct push sampling equipment. However, blow counts will not be available. A pocket penetrometer may be used instead to determine relative soil consistency of fine grained materials (silts and clays).

Sample also may be collected using a 300-pound hammer or 3-inch-diameter split-spoon samples at the site. However, use of either of these sample collection devices invalidates standard penetration test results and should be noted in the comments section of the log. The 300-pound hammer should only be used for collection of 3-inch-diameter split-spoon samples. Blow counts should be recorded for collection of samples using either a 3-inch split-spoon, or a 300-pound hammer. An “N” value need not be calculated.

Soil Description. The soil classification should follow the format described in the "Field Classification of Soil" subsection below.

Comments. Include all pertinent observations (changes in drilling fluid color, rod drops, drilling chatter, rod bounce as in driving on a cobble, damaged Shelby tubes, and equipment malfunctions). In addition, note if casing was used, the sizes and depths installed, and if drilling fluid was added or changed. You should instruct the driller to alert you to any significant changes in drilling (changes in material, occurrence of boulders, and loss of drilling fluid). Such information should be attributed to the driller and recorded in this column.

Specific information might include the following:

- The date and the time drilling began and ended each day
- The depth and size of casing and the method of installation
- The date, time, and depth of water level measurements
- Depth of rod chatter
- Depth and percentage of drilling fluid loss
- Depth of hole caving or heaving
- Depth of change in material
- Health and safety monitoring data
- Drilling interval through a boulder

D. Field Classification of Soil

This section presents the format for the field classification of soil. In general, the approach and format for classifying soils should conform to ASTM International (ASTM) Standard D2488 *Standard Practice for Description and Identification of Soils (Visual-Manual Procedures)*.

The USCS is based on numerical values of certain soil properties that are measured by laboratory tests; however, it is possible to estimate these values in the field with reasonable accuracy using visual-manual procedures (ASTM D2488). In addition, some elements of a complete soil description, such as the presence of cobbles or boulders, changes in strata, and the relative proportions of soil types in a bedded deposit, can be obtained only in the field.

Soil descriptions should be precise and comprehensive without being verbose. The correct overall impression of the soil should not be distorted by excessive emphasis on insignificant details. In general, similarities rather than differences between consecutive samples should be stressed.

Soil descriptions must be recorded for every soil sample collected. The format and order for soil descriptions should be as follows:

1) Soil name (synonymous with ASTM D2488 Group Name) with appropriate modifiers. Soil name should be in all capitals in the log, for example "POORLY-GRADED SAND."
2) Group symbol, in parentheses, for example, "(SP)."
3) Color, using Munsell color designation
4) Moisture content
5) Relative density (coarse grained) or consistency (fine grained)
6) Soil structure, mineralogy, or other descriptors

This order follows, in general, the format described in ASTM D2488.

E. Soil Name

The basic name of a soil should be the ASTM D2488 Group Name on the basis of visual estimates of gradation and plasticity. The soil name should be capitalized.

Examples of acceptable soil names are illustrated by the following descriptions:

- A soil sample is visually estimated to contain 15 percent gravel, 55 percent sand, and 30 percent fines (passing No. 200 sieve). The fines are estimated as either low or highly plastic silt. This visual classification is SILTY SAND WITH GRAVEL, with a Group Symbol of (SM).
- Another soil sample has the following visual estimate: 10 percent gravel, 30 percent sand, and 60 percent fines (passing the No. 200 sieve). The fines are estimated as low plastic silt. This visual classification is SANDY SILT. The gravel portion is not included in the soil name because the gravel portion was estimated as less than 15 percent. The Group Symbol is (ML).

The gradation of coarse-grained soil (more than 50 percent retained on No. 200 sieve) is included in the specific soil name in accordance with ASTM D2488. There is no need to further document the gradation. However, the maximum size and angularity or roundness of gravel and sand-sized particles should be recorded. For fine-grained soil (50 percent or more passing the No. 200 sieve), the name is modified by the appropriate plasticity/elasticity term in accordance with ASTM D2488.

Interlayered soil should each be described starting with the predominant type. An introductory name, such as "Interlayered Sand and Silt," should be used. In addition, the relative proportion of each soil type should be indicated (see **Table 1** for example).

Where helpful, the evaluation of plasticity/elasticity can be justified by describing results from any of the visual-manual procedures for identifying fine-grained soils, such as reaction to shaking, toughness of a soil thread, or dry strength as described in ASTM D2488.

F. Group Symbol

The appropriate group symbol from ASTM D2488 must be given after each soil name. The group symbol should be placed in parentheses to indicate that the classification has been estimated.

In accordance with ASTM D2488, dual symbols (e.g., GP-GM or SW-SC) can be used to indicate that a soil is estimated to have about 10 percent fines. Borderline symbols (e.g., GM/SM or SW/SP) can be used to indicate that a soil sample has been identified as having properties that do not distinctly place the soil into a specific group. Generally, the group name assigned to a soil with a borderline symbol should be the group name for the first symbol. The use of a borderline symbol should not be used indiscriminately. Every effort should be made to first place the soil into a single group.

G. Color

The color of a soil must be given. The color description should be based on the Munsell system. The color name and the hue, value, and chroma should be given.

H. Moisture Content

The degree of moisture present in a soil sample should be defined as dry, moist, or wet. Moisture content can be estimated from the criteria listed on **Table 2**.

I. Relative Density or Consistency

Relative density of a coarse-grained (cohesionless) soil is based on N-values (ASTM D1586). If the presence of large gravel, disturbance of the sample, or non-standard sample collection makes determination of the in situ relative density or consistency difficult, then this item should be left out of the description and explained in the Comments column of the soil boring log.

Consistency of fine-grained (cohesive) soil is properly based on results of pocket penetrometer or torvane results. In the absence of this information, consistency can be estimated from N-values. Relationships for determining relative density or consistency of soil samples are given in **Tables 3 and 4**.

J. Evidence of Contamination

- **Odor:** Olfactory observations (odors, or lack of odor) or positive responses to an organic vapor detector will be recorded on the soil boring log.

The following descriptors should be used to characterize any visible evidence of NAPL impact:

- **Stained** – Color (i.e., black or brown stained) is used to indicate that the soil matrix is stained a color other than the natural (unimpacted) color of the soil. Stained soil can be present in the absence of sheen or NAPL Stained or NAPL impacted soil (see below) may or may not be present with the staining. If NAPL or sheen is also present within the stained intervals these must be noted also as described below
- **Sheen** – Indicates a thin film at the surface of the pore water that has accumulated in the pore spaces or at the surface of the soil along any interval. The color of the sheen present should be noted using the following descriptors:
  - Silvery: metallic, silver gray in color
  - Rainbow: multicolored
  - Dark Rainbow: multicolored with some dark metallic or black/brown coloring
  - Dark metallic: reflects/mirrors the color of the sky or brown/black colored
- The intervals of observed sheen and the relative amounts should also be described as trace (<5%), slight (5-15%), moderate (15-40%), moderate to heavy (40-70%), and heavy (>70%). If the sheen is easily fractured upon touch or disturbance this should be noted also.
- **NAPL** – Any free phase NAPL observed in cores should be described in terms of color, distribution, and viscosity (if determinable) as described further below. Free phase product should be described as NAPL in the "Comments" column on the core logs. ***Do not draw any conclusions about the type of NAPL (e.g., oil, tar, fuel, coal tar, etc.).*** The following standard terminology should be used to describe the relative amount of NAPL impacts:
  - **Blebs** – These are observed discrete spheres or droplets of NAPL, but for the most part the soil matrix is not visibly contaminated or saturated with NAPL.
  - **Coated** – Soil grains are coated with NAPL. There is not sufficient free-phase material present to saturate the pore spaces.
  - **Saturated** – The entirety of the pore space for a given interval is saturated with free NAPL. Care should be taken to ensure that the pore spaces are saturated with NAPL rather than water if this term is used.

  If the NAPL is present in sufficient quantities, then the following ***viscosity modifiers*** should also be used in the description:
    - High viscosity – Taffy-like (sticky)
    - Viscous – molasses-like (thick liquid)
    - Low viscosity – runny (thin liquid)
- **Non-liquid Tars** – If non-liquid contamination is observed it should be classified as soft or hard and its depth, color, size and general appearance should be recorded.

K. Soil Structure, Mineralogy, and Other Descriptors

Discontinuities and inclusions are important and should be described. Such features include joints or fissures, slickensides, bedding or laminations, veins, root holes, and wood debris.

Significant mineralogical information such as cementation, abundant mica, or unusual mineralogy should be described.

Other descriptors may include particle size range or percentages, particle angularity or shape, maximum particle size, hardness of large particles, plasticity of fines, dry strength, dilatancy, toughness, reaction to HCl, and staining, as well as other information such as organic debris, odor, or presence of free product.

12.4 Attachments

Tables 1 through 4 (Tables 1-4.pdf)

Soil Boring Log and Key (Sample Soil Boring Log.xls)

12.5 Key Checks and Preventive Maintenance

- Check entries to the soil-boring log and field logbook in the field; because the samples will be disposed of at the end of fieldwork, confirmation and corrections cannot be made later.
- Check that sample numbers and intervals are properly specified.
- Check that drilling and sampling equipment is decontaminated per project SOP.

End of SOP-12

Table 1
EXAMPLE SOIL DESCRIPTIONS

POORLY GRADED SAND (SP), light brown, moist, loose, fine sand size

FAT CLAY (CH), dark gray, moist, stiff

SILT (ML), light greenish gray, wet, very loose, some mica, lacustrine

WELL-GRADED SAND WITH GRAVEL (SM), reddish brown, moist, dense, subangular gravel to 0.6 inches max

POORLY GRADED SAND WITH SILT (SP-SM), white, wet, medium dense

ORGANIC SOIL WITH SAND (OH), dark brown to black, wet, firm to stiff but spongy undisturbed, becomes soft and sticky when remolded, many fine roots, trace of mica

SILTY GRAVEL WITH SAND (GM), brownish red, moist, very dense, subrounded gravel to 1.2 inches max

INTERLAYERED SILT (60 percent) AND CLAY (40 percent): SILT WITH SAND (ML), medium greenish gray, nonplastic, sudden reaction to shaking, layers mostly 1.5 to 8.3 inches thick; LEAN CLAY (CL), dark gray, firm and brittle undisturbed, becomes very soft and sticky when remolded, layers 0.2 to 1.2 inches thick

SILTY SAND WITH GRAVEL (SM), light yellowish brown, moist, medium dense, weak gravel to 1.0 inches max, very few small particles of coal, fill

SANDY ELASTIC SILT (MH), very light gray to white, wet, stiff, weak calcareous cementation

LEAN CLAY WITH SAND (CL/MH), dark brownish gray, moist, stiff

WELL-GRADED GRAVEL WITH SILT (GW-GM), brown, moist, very dense, rounded gravel to 1.0 inches max

Table 2
CRITERIA FOR DESCRIBING MOISTURE CONDITION

Description	Criteria
Dry	Absence of moisture, dusty, dry to the touch
Moist	Damp, but no visible water
Wet	Visible free water, usually soil is below water table

Table 3
RELATIVE DENSITY OF COARSE-GRAINED SOIL
(Developed from Sowers, 1979)

Blows/Ft	Relative Density	Field Test
0-4	Very loose	Easily penetrated with ½-in. steel rod pushed by hand
5-10	Loose	Easily penetrated with ½-in. steel rod pushed by hand
11-30	Medium	Easily penetrated with ½-in. steel rod driven with 5-lb hammer
31-50	Dense	Penetrated a foot with ½-in. steel rod driven with 5-lb hammer
>50	Very dense	Penetrated only a few inches with ½-in. steel rod driven with 5-lb hammer

Table 4
CONSISTENCY OF FINE-GRAINED SOIL
(Developed from Sowers, 1979)

Blows/Ft	Consistency	Pocket Penetrometer (TSF)	Torvane (TSF)	Field Test
<2	Very soft	<0.25	<0.12	Easily penetrated several inches by fist
2-4	Soft	0.25-0.50	0.12-0.25	Easily penetrated several inches by thumb
5-8	Firm	0.50-1.0	0.25-0.5	Can be penetrated several inches by thumb with moderate effort
9-15	Stiff	1.0-2.0	0.5-1.0	Readily indented by thumb, but penetrated only with great effort
16-30	Very stiff	2.0-4.0	1.0-2.0	Readily indented by thumbnail
>30	Hard	>4.0	>2.0	Indented with difficulty by thumbnail

Jacobs

PROJECT NUMBER	BORING NUMBER

SOIL BORING LOG

PROJECT : LOCATION :

ELEVATION : DRILLING CONTRACTOR :

DRILLING METHOD AND EQUIPMENT USED :

ATD WATER LEVEL : START : END : LOGGER :

DEPTH BELOW SURFACE (FT)	INTERVAL (FT)	RECOVERY (FT)	SAMPLE #/TYPE	STANDARD PENETRATION TEST RESULTS 6"-6"-6"-6" (N')	SOIL DESCRIPTION	USCS	COMMENTS
					SOIL NAME, USCS GROUP SYMBOL, COLOR, MOISTURE CONTENT, RELATIVE DENSITY, OR CONSISTENCY, SOIL STRUCTURE, MINERALOGY.		DEPTH OF CASING, DRILLING RATE, DRILLING FLUID LOSS, TESTS, AND INSTRUMENTATION. DRILLING ACTIONS/DRILLER COMMENTS PID Readings: Breathing Zone: Above Hole:

Sediment Core Log Key

MAJOR DIVISIONS			GRAPHIC SYMBOL	GROUP SYMBOL	DESCRIPTION
COARSE-GRAINED MATERIAL	GRAVELS	CLEAN GRAVELS		GW	Well-graded gravel Well-graded gravel with sand
				GP	Poorly graded gravel Poorly graded gravel with sand
		GRAVELS WITH FINES		GW-GM	Well-graded gravel with silt Well-graded gravel with silt and sand
				GW-GC	Well-graded gravel with clay Well graded gravel with clay and sand
				GP-GM	Poorly graded gravel with silt Poorly graded gravel with silt and sand
				GP-GC	Poorly graded gravel with clay Poorly graded gravel with clay and sand
				GM	Silty gravel Silty gravel with sand
				GC	Clayey gravel Clayey gravel with sand
	SANDS	CLEAN SANDS		SW	Well-graded sands Well-graded sand and gravel
				SP	Poorly-graded sands Poorly graded sand with gravel
		SANDS WITH FINES		SW-SM	Well-graded sand with silt Well-graded sand with silt and gravel
				SW-SC	Well-graded sand with clay Well-graded sand with clay and gravel
				SP-SM	Poorly-graded sand with silt Poorly-graded sand with silt and gravel
				SP-SC	Poorly-graded sand with clay Poorly-graded sand with clay and gravel
				SM	Silty sand Silty sand and with gravel
				SC	Clayey sand Clayey sand and with gravel
FINE-GRAINED MATERIALS	SILTS AND CLAYS			CL	Lean clay * Lean clay with sand or gravel * Sandy lean clay * Sandy lean clay with gravel * Gravelly lean clay * Gravelly lean clay with sand
				ML	Silt * Silty with sand or gravel * Sandy silt * Sandy silt with gravel * Gravelly silt * Gravelly silt with sand
				CH	Fat clay * Fat clay with sand or gravel * Sandy fat clay * Gravelly fat clay * Gravelly fat clay with sand
				MH	Elastic silt * Elastic silt with sand or gravel * Sandy elastic silt * Sandy elastic silt with gravel * Gravelly elastic silt * Gravelly elastic silt with sand
				OL/OH	Organic silt * Organic silt with sand or gravel * Sandy organic silt * Sandy organic soil with gravel * Gravelly organic soil * Gravelly organic soil with sand

Well Graded (Engineering) = Poorly Sorted (Geological) = grains of all different sizes mixed together

Poorly Graded (Engineering) = Well Sorted (Geological) = grains are all same size

Shell hash

λλλλ Peat/organic matter

CONSISTENCY
Penetration of thumb:
<0.25 cm = hard (H)
0.25 - 2.0 cm = firm (F)
2.0 - 4.0 cm = soft (S)
>4.0 cm = very soft (VS)

CEMENTATION
N = not cemented
W = weakly cemented
M = Moderately cemented
S = Strongly cemented

STRUCTURE
H = Homogeneous
S = Stratified
L = Laminated
M = Mottled

COLOR
from Munsell chart

MAXIMUM PARTICLE SIZE
SC = Small Cobble
CP = Coarse Pebble
MP = Medium Pebble
SP = Small Pebble
CS = Coarse Sand
MS = Medium Sand
FS = Fine Sand
VFS = Very Fine Sand
Z = Silt

SA = Sub-angular
VA = Very angular

ODOR
N = None
S = Sulfur-like/sulfide
HC = Hydrocarbon-like
F = Fuel-like

Quantifying Descriptors for Odor
Strong
Moderate
Faint
None

Moisture Content
Wet
Moist
Dry

VISIBLE CONTAMINATION DESCRIPTORS

The following descriptors should be used to characterize any visible evidence of NAPL impact:

• **Stained** – Color (i.e., black or brown stained) is used to indicate that the soil matrix is stained a color other than the natural (unimpacted) color of the soil. Stained soil can be present in the absence of sheen or NAPL Stained or NAPL impacted soil (see below) may or may not be present with the staining. If NAPL or sheen is also present within the stained intervals these must be noted also as described below

• **Sheen** – Indicates an a thin film at the surface of the pore water that has accumulated in core sleeve or in the pore spaces or at the surface of the sediment along any interval. The color of the sheen present should be noted using the following descriptors:

– *Silvery: metallic, silver gray in color*

– *Rainbow: multicolored*

– *Dark Rainbow: multicolored with some dark metallic or black/brown coloring*

The intervals of observed sheen and the relative amounts should also be described as trace (<5%), slight (5-15%), moderate (15-40%), moderate to heavy (40-70%), and heavy (>70%). If the sheen is easily fractured upon touch or disturbance this should be noted also.

• **NAPL** – Any free phase NAPL observed in cores should be described in terms of color, distribution, and viscosity (if determinable) as described further below. Free phase product should be described as NAPL in the “Comments” column on the core logs. Do not draw any conclusions about the type of NAPL (e.g., oil, tar, fuel, coal tar, etc.). The following standard terminology should be used to describe the relative amount of NAPL impacts:

– *Blebs* – These are observed discrete spheres or droplets of NAPL, but for the most part the sediment matrix is not visibly contaminated or saturated with NAPL.

– *Coated* – Sediment grains are coated with NAPL. There is not sufficient free-phase material present to saturate the pore spaces.

– *Saturated* – The entirety of the pore space for a given interval is saturated with free NAPL. Care should be taken to ensure that the pore spaces are saturated with NAPL rather than water if this term is used.

If the NAPL is present in sufficient quantities, then the following viscosity modifiers should also be used in the description:

- *High viscosity – Taffy-like (sticky)*
- *Viscous – molasses-like (thick liquid)*
- *Low viscosity – runny (thin liquid)*

Non-liquid Tars – If non-liquid contamination is observed it should be classified as soft or hard and its depth, color, size and general appearance should be recorded.

STANDARD OPERATING PROCEDURE-13

13. BANCS Model Parameters Field Assessment

The purpose of this standard operating procedure (SOP) is to establish a procedure for assessing input parameters to be used in the Bank Assessment for Non-Point Source Consequences of Sediment (BANCS) model. The data collected by the field team will be used to assess the erodibility of stream banks within the project area.

The work will be conducted on the oversite of a registered geotechnical engineer in the State of Oregon.

13.1 Associated Procedures

The BANCS model predicts river bank erosion using the erodibility potential of the bank determined by two factors: bank erosion hazard index (BEHI) and erosional forces represented by the nearbank stress (NBS) of the river acting on the river bank. The BANCS model was developed based on empirical relationships between BEHI, NBS, and erosion rates documented at active streams and rivers in case studies. The case studies used to develop the BANCS model did not include large rivers in industrial settings, such as Portland Harbor, which has other river bank erosion mechanics not covered by the BANCS model (e.g., wind- and boat-induced waves, tidal action, and overland runoff erosion of the bank). However, the BANCS model is useful for evaluating river bank erosion related to BEHI and NBS (EPA, 2019).

The BEHI and NBS procedures outlined below are based on modified procedures described in Newton and Drenton (2015), Bigham (2016), EPA (2019). Each of these references is based on the BANCS model outlined in Rosgen (2009).

13.2 Equipment and Supplies

Relevant field data is documented and recorded through the ERSI ArcGIS Collector app. The app will be used to document and organize photographs, GPS coordinates of sampling locations, field observations, etc. as described in SOP-4a.

The collector will be preloaded with maps that show publicly available aerial photography, parcel boundaries and topographic contours.

- Waterproof, bound logbooks with consecutively numbered pages
- Pens with indelible ink
- Evaluation Worksheets
- Clinometer
- Measuring tape
- Camera

13.3 Bank Erosion Hazard Index Procedure

Representative bank segments will be selected prior to field work based on aerial photography and bathymetric and topographic contours. Field work will then be performed to collect the parameters listed below. Data will be recorded on the collector which will display BEHI Evaluation Worksheet (Attachment A).

1) **Stream Identification**

   a) Record the date, the weather, location (property), station (river mile), and field observers.

   b) Take GPS coordinates at the most upstream (where the water is flowing from) and most downstream (where the water is flowing to) points of the bank.

   c) Take pictures at both the upstream and downstream points on the bank. Capture the main features of the bank in the pictures. Take additional pictures to capture features on the bank or in the channel (*i.e.*, stratification, large woody debris jams, etc.). Record the picture numbers for reference.

   d) Measure the bank length at the toe, in feet, between the most upstream and most downstream points on the bank.

   e) Note the distance to infrastructure and the type of infrastructure (*i.e.*, bridges, culverts, roads, utilities, houses etc.).

   f) Note any of the following qualitative indicators that are present: Unvegetated mid-channel bar/braided channel, exposed tree roots on both sides, leaning trees on both sides, exposed infrastructure, downstream of a dam, slumping streambanks, headcuts, perched tributaries, walls, gabion baskets or culverts.

2) **Height of the bank**

   a) Record the river bank height (in feet) in Box A in the BEHI Evaluation Worksheet. River bank height is measured from the top of the bank, defined as the point where the slope of the land surface changes from toward the river to toward the uplands, to the toe of the slope, defined as the first significant break in slope that is below ordinary high water (OHW) but above mean high water. Bank height will be determined from topographic and bathymetric maps. Banks were remedial design is implemented will need detailed survey data in the future for design.

3) **Bankfull level**

   a) Record the bankfull level (in feet) in Box B. Bankfull level is the point on the river bank that contains normal non-flood-level flows of the river throughout the year and is typically identifiable by visible changes in topography, vegetation type, or sediment grain size (EPA, 2019). For the Willamette River, the bankfull level is approximated by the OHW elevation, which is equivalent to 20.08 feet NAVD-88. OHW elevation will be used as the default value.

4) **Bank angle**

   a) Measure the maximum bank angle between the toe of the slope and the top of the bank. The bank angle will be determined from topographic or bathymetric maps or measured in the field using a clinometer or other suitable direct field measurement technique. Take an average of the bank angle along the entire section under evaluation. Record the bank angle on the collector.

5) **Surface protection**

   a) Determine the percentage of the river bank that is covered and protected by woody debris, rooted vegetation, embedded boulders, revetment, bedrock, or other embedded materials that protect the bank from erosion (EPA, 2019). Assess the type and size of protection; location relative to the toe of the slope, top of the bank, and OHW elevation; percent of the river bank surface protected; and general condition of protection (i.e., determine whether the material is stable, unstable, or sloughing into the river). If the protection is afforded by armoring, the general condition of the armoring should be noted to determine whether the material is stable, unstable, or sloughing into the river. If the river bank is vegetated, the assessment should include the vegetation type, rooting

depth, root density, and the percent of the river bank surface that is covered by vegetation. Record an estimate of surface protection (percent) along the entire bank in the collector.

6) **Rooting depth**

   a) For vegetated banks, rooting depth estimates will be based on pictures of the established vegetation and subsequent review by a trained biologist familiar with City of Portland greenway approved species.

   b) Rooting depth is the maximum depth of plant roots in the river bank. Do not consider roots that are void of bank material (*i.e.*, hanging roots).

7) **Root density**

   a) For vegetated banks, root density will be estimated. Root density is the percent of subsurface soil within the vegetation root penetration depth that is composed of roots. Do not consider roots that are void of bank material. Small, fibrous roots can be very dense and provide greater soil retention compared to the larger root systems of trees. Estimates of the coverage of each species will be based on drone photography obtained in 2019 and measured from top of the bank to the toe of the bank and verified in the field by a site visit by a trained biologist.

8) **Bank composition/soil type**

   a) Estimate the soil composition of the river bank based on available site data and verified in the field through visual inspection and by field classification methods. The Unified Soil Classification System (USCS) outlined by ASTM International (ASTM) Standard D2487 will be used to describe the soil types. Field classifications will be determined using the procedures described in ASTM Standard D2488, Standard Practice for Description and Identification of Soils (Visual-Manual Procedure). Field sieve analysis may be used to supplement visual classification and verify grain-size distribution for soil samples collected from the bank for chemical characterization.

9) **Bank material stratification**

   a) Estimate the number of stratified layers based on a visual inspection by a registered geotechnical engineer. Stratification refers to a clearly defined horizontal break in geology which has the potential to cause and enhance zones of preferential erosion. Record how many layers of stratification are present.

13.4 Near Bank Stress Procedure

NBS is determined in the BANCS model by one or more of seven methods presented by Rosgen (2009). NBS ratings range from Very Low to Extreme. There are seven methods that can be used to determine the NBS rating. They are listed below along with the data collection requirements:

13.4.1 Presence of Transverse/Central Bars or Channel Pattern Changes

13.4.1.1 This method requires the field practitioner to look for any kind of excess deposition, such as central or transverse bars, that may be directing flow and creating a high shear stress in the near-bank region of the streambank in interest. The river is relatively straight in this section and none of these features are expected.

13.4.2 Radius of Curvature-to-Bankfull Width ratio

13.4.2.1 In this method, the radius of curvature of the meander bend containing the streambank of interest is measured. The river is relatively straight in this section and none of these features are expected.

13.4.3 Pool Slope-to-Average Water Surface Slope ratio

13.4.3.1 This method can be used to determine NBS rating if the eroding streambank of interest is located along a pool. None are present in the WCPA.

13.4.4 Pool Slope-to-Riffle Slope ratio

13.4.4.1 Similar to pool slope, this method can be utilized to determine NBS rating if the eroding streambank of interest is located along a pool.

13.4.5 Near-bank Maximum Depth-to-Bankfull Mean Depth ratio

13.4.5.1 This method requires the field practitioner to survey a cross section at the eroding streambank of interest to obtain maximum depth and mean depth at bankfull stage. These data are collected in section 13.3

13.4.6 Near-bank Shear Stress-to-Bankfull Shear Stress ratio

13.4.6.1 This method calculates shar stress using river geometry and flow data. Site specific data on bankfull depth is needed that is collected in Section 13.3.

13.4.7 Velocity Isovels

13.4.8 This method requires the greatest amount of field data collection. Vertical velocity profiles across the channel perpendicular to the bank of interest must be collected using current meters during at or near bankfull flow events. This method will not be used as it is not appropriate for large rivers.

These methods involve considering stream geometry, presence of depositional and erosional geomorphic features, and actual stream velocity to determine the NBS parameter. The most appropriate method for NBS determination should be based on site-specific characteristics and professional judgement. NBS provides a rating using one of seven methods to generate a numerical score that determines a ranking from very low to extreme (EPA, 2019). Attachment A provides a summary of the rating methodologies for determining NBS.

13.5 Attachments

Attachment A – BEHI and NBS procedure summary including photos and worksheets. Obtained from Bigham (2011) .

13.6 References

American Society for Testing Materials (ASTM) International. 2018. *Annual book of ASTM Standards*. Philadelphia, PA.

Bigham, K.A. 2016. *Evaluation and application of the bank assessment for non-point source consequences of sediment (BANCS) model developed to predict annual streambank erosion rates*. Masters Thesis. Kansas State University.

Newton, S.E. and Drenten, D.M. 2015. *Modifying the Bank Erosion Hazard Index (BEHI) Protocol for Rapid Assessment of Streambank Erosion in Northeastern Ohio*. Journal of Visualized Experiments. Issue 96.

Rosgen, D.L. 2009. *Watershed Assessment of River Stability and Sediment Supply (WARSSS)*. Second edition. Fort Collins, CO: Wildland Hydrology.

U.S. Environmental Protection Agency (EPA). 2019. *Explanation of Significant Differences Portland Harbor Superfund Site,* Portland Oregon. Region 10. Seattle, Washington. December.

End of SOP-13

2.3.1 Bank Erosion Hazard Index (BEHI) Rating Methodology

The BEHI provides an adjective rating of bank erodibility based on visual and measured assessment of a streambank of interest. Seven variables comprise the BEHI method to develop an overall BEHI rating. These variables are:

1) Study Bank Height-to-Bankfull Height Ratio
2) Root Depth-to-Study Bank Height Ratio
3) Weighted Root Density
4) Bank Angle
5) Surface Protection
6) Bank Material
7) Stratification of Bank Material

These seven variables are then inputted into the BEHI worksheet, shown in Figure 2.5, and converted into a BEHI score/rating using Figure 2.6. The empirical relationships between streambank variables and ratings presented in Figure 2.6 are based on the conversion of field observation to erosion potential (Rosgen, 2001). Individual ratings of the seven variables vary from Very Low (0-2 points) to Extreme (9-10 points). These scores are then summed to determine the overall BEHI rating for a specific streambank. Overall BEHI ratings range from Very Low (0-9.5 total points) to Extreme (46-50 total points). More detail is provided in the next sections for assessing each of the seven BEHI variables (Rosgen, 2001; Rosgen, 1996b; Rosgen, 2009).

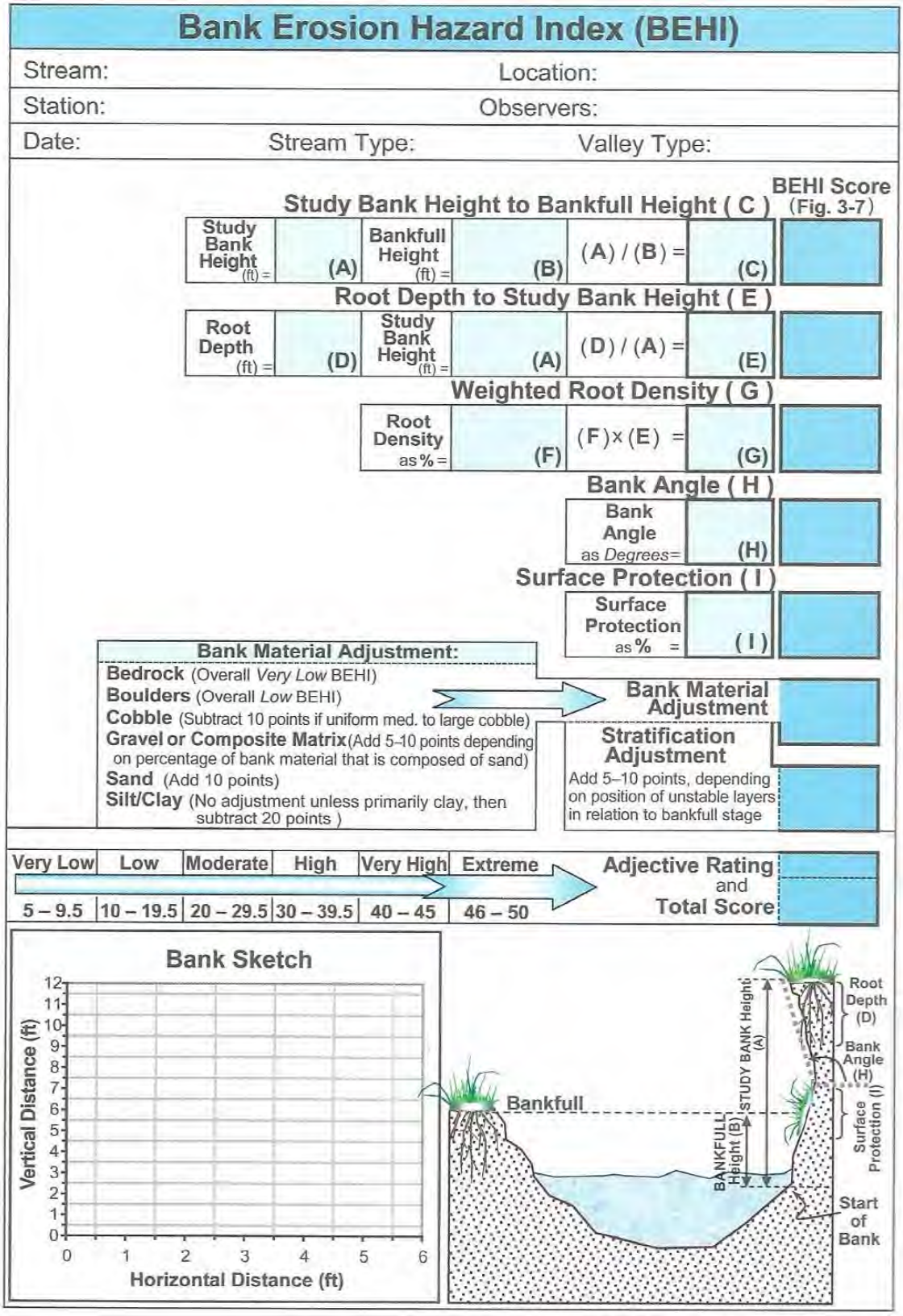


Figure 2.5 Bank Erosion Hazard Index (BEHI) Worksheet (Rosgen, 2014)

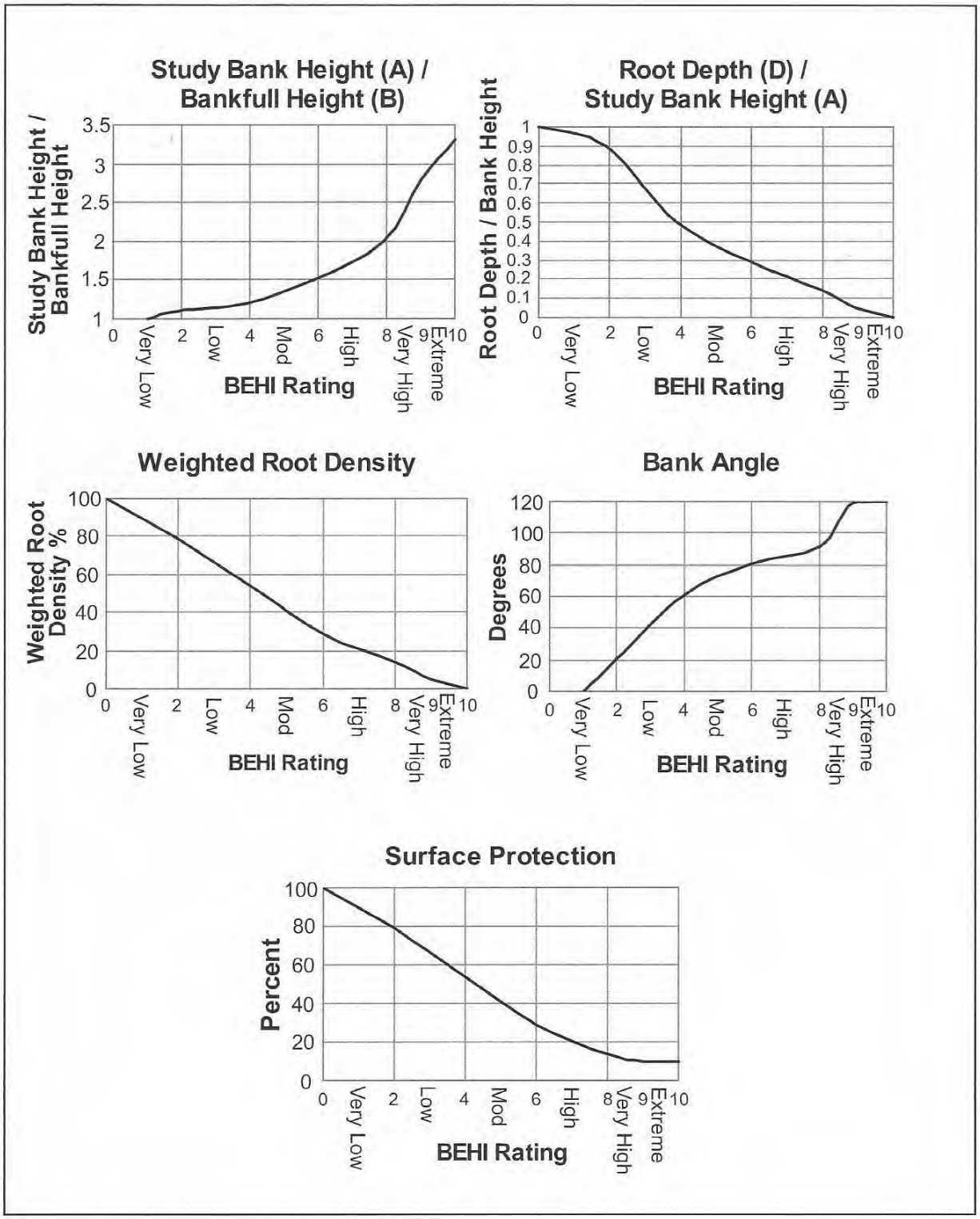


Figure 2.6 Relationships between Bank Erosion Hazard Index (BEHI) Variables and Ratings (Rosgen, 2009)

2.3.1.1 Study Bank Height-to-Bankfull Height Ratio

Study bank height and bankfull height are measured from the toe of the bank to the top of the bank of interest or to a bankfull indicator, respectively (see Figure 2.7). Study bank height is then divided by the bankfull height to adjust for scale, such that this measure can be applied to varying sizes of streams. As study bank height becomes greater than bankfull height, the risk of erosion as well as the amount of sediment potentially available to erode increases (Rosgen, 2009; Simon, 1989).

Figure 2.7 Study Bank Height-to-Bankfull Height Ratio Example – Middle Little Arkansas River Study Bank #3

2.3.1.2 Root Depth-to-Study Bank Height Ratio

The prevailing rooting depth, as shown in Figure 2.8, is measured and then divided by the study bank height, determined in the previous measurement. The deeper the roots, the greater the resistive force of the bank to erosion, as portrayed in the ratio-to-BEHI rating conversion (see Figure 2.6). If the roots do not extend to the toe of the bank, then the bank may become undercut which may lead to cantilever failure of the bank (Rosgen, 2009; Simon & Collison, 2002).

Figure 2.8 Root Depth-to-Study Bank Height Ratio and Root Density Example – Lower Little Arkansas River Study Bank #3

2.3.1.3 Weighted Root Density

Weighted root density is an ocular estimate of the amount of roots per unit volume of soil (see Figure 2.8). This estimate is taken only in the area of the bank where there are roots. This value is then multiplied by the root depth-to-study bank height ratio to get a weighted root density value for the entire height of the bank. The greater the weighted root density, the greater the soil strength (Simon & Collison, 2002), and the more resistive the bank is to erosion, as portrayed in the weighted root density-to-BEHI rating conversion (Rosgen, 2009).

2.3.1.4 Bank Angle

Bank angle is measured to determine the bank's susceptibility to erosion. Ninety degrees is a vertical bank, less than ninety degrees is a bank that is laid back, and greater than ninety degrees is a bank that is most likely undercut and highly susceptible to bank failure (Simon et al., 2000). Figure 2.9 below shows an example of bank angle (Rosgen, 2009).

Figure 2.9 Bank Angle Example – Lower West Emma Creek Study Bank # 1

2.3.1.5 Surface Protection

Surface protection is an ocular estimate of the percentage of bank surface area protected by some form of vegetation such as sod mats and/or woody debris (see Figure 2.10). The greater the streambank area protected by vegetation, the lower the potential for erosion (Rosgen, 2009; Simon & Hupp, 1987).

2.3.1.6 Bank Material

Bank material is determined either through an ocular or texture-by-feel estimate. Rosgen (2001; 2009) denotes six different types of bank materials: bedrock, boulders, cobble, gravel or composite matrix, sand, and silt/clay. Depending on what material the bank is composed of determines whether or not to give the bank a Very Low/Low overall BEHI rating (bedrock and boulders only) or if the field practitioner should add or subtract points from the overall BEHI score. Points may be added to the score if the material in the bank is considered to be highly erodible, such as gravel or composite matrix and sand. Points may be subtracted from the score if the bank material is highly resistive to erosion such as medium to large cobble and banks with high amounts of clay (Parker et al., 2008; Rosgen, 2009; Rosgen, 2014; Simon et al., 2000). As suggested by the RIVERMorph software, created by Stantec Consulting Services (2013) to easily analyze Rosgen stream geomorphology methodology, if the clay content is greater than 50% clay

content, then subtract 10 points. If bank material is predominately clay, then subtract 20 points. See Figure 2.5 for point assignments and Figure 2.11 for an example.

Figure 2.10 Surface Protection Example – Lower Little Arkansas River Study Bank #2

2.3.1.7 Stratification of Bank Material

Banks with different layers of bank material are considered stratified. This overall BEHI score adjustment allows the field practitioner to determine whether the layers in the bank are susceptible to erosion, either via surface erosion or piping. Piping may occur in layers of pervious materials such as sand or small gravel (Fox et al., 2007; Hagerty, 1991; Rosgen, 2009; Thorne & Tovey, 1981). If these kinds of layers are evident, five to ten points may be added to the overall BEHI score (Rosgen, 2009). Figure 2.11 shows an example of bank stratification.

Figure 2.11 Bank Material and Stratification Example – Middle Little Arkansas River Study Bank #1

2.3.2 Near-Bank Stress (NBS) Rating Methodology

The NBS rating provides an adjective rating of the amount of fluvial force (or applied shear stress) induced by the outer one-third of the channel flow adjacent to the eroding bank of interest (Rosgen, 2009). Fluvial forces are the result of the local energy slope and the channel pattern and dimensions (Rosgen, 1996b; Simon et al., 2000). NBS ratings range from Very Low to Extreme. There are seven methods that can be used to determine the NBS rating. They are as follows:

1) Presence of Transverse/Central Bars or Channel Pattern Changes
2) Radius of Curvature-to-Bankfull Width ratio
3) Pool Slope-to-Average Water Surface Slope ratio
4) Pool Slope-to-Riffle Slope ratio
5) Near-bank Maximum Depth-to-Bankfull Mean Depth ratio
6) Near-bank Shear Stress-to-Bankfull Shear Stress ratio
7) Velocity Isovels

Estimating Near-Bank Stress (NBS)

Stream: Location:

Station: Stream Type: Valley Type:

Observers: Date:

Methods for Estimating Near-Bank Stress (NBS)

(1)	Channel pattern, transverse bar, or split channel/central bar creating NBS.....	Level I	Reconaissance
(2)	Radius of curvature to bankfull width (R_c / W_{bkf}).....	Level II	General Prediction
(3)	Pool slope to average water surface slope (S_p / S).....	Level II	General Prediction
(4)	Pool slope to riffle slope (S_p / S_{rif}).....	Level II	General Prediction
(5)	Near-bank maximum depth to bankfull mean depth (d_{nb} / d_{bkf}).....	Level III	Detailed Prediction
(6)	Near-bank shear stress to bankfull shear stress (τ_{nb} / τ_{bkf}).....	Level III	Detailed Prediction
(7)	Velocity profiles / Isovels / Velocity gradient.....	Level IV	Validation

Level I

(1) Transverse or central bars - short or discontinuous.....NBS = *High / Very High*
Extensive deposition (continuous, cross-channel).....NBS = *Extreme*
Chute cutoffs, down-valley meander migration, converging flow.....NBS = *Extreme*

Level II

(2) Radius of Curvature R_c (ft)	Bankfull Width W_{bkf} (ft)	*Ratio* R_c / W_{bkf}	Near-Bank Stress (NBS)

(3) Pool Slope S_p	Average Slope S	*Ratio* S_p / S	Near-Bank Stress (NBS)

(4) Pool Slope S_p	Riffle Slope S_{rif}	*Ratio* S_p / S_{rif}	Near-Bank Stress (NBS)

Dominant Near-Bank Stress

Level III

(5) Near-Bank Max Depth d_{nb} (ft)	Mean Depth d_{bkf} (ft)	*Ratio* d_{nb} / d_{bkf}	Near-Bank Stress (NBS)

(6) Near-Bank Max Depth d_{nb} (ft)	Near-Bank Slope S_{nb}	Near-Bank Shear Stress τ_{nb} (lb/ft^2)	Mean Depth d_{bkf} (ft)	Average Slope S	Bankfull Shear Stress τ_{bkf} (lb/ft^2)	Ratio τ_{nb} / τ_{bkf}	Near-Bank Stress (NBS)

Level IV

(7) Velocity Gradient (ft / sec/ ft)	Near-Bank Stress (NBS)

Converting Values to a Near-Bank Stress (NBS) Rating

Near-Bank Stress (NBS) Ratings	Method Number (1)	(2)	(3)	(4)	(5)	(6)	(7)
Very Low	N / A	> 3.00	< 0.20	< 0.40	< 1.00	< 0.80	< 0.50
Low	N / A	2.21 – 3.00	0.20 – 0.40	0.41 – 0.60	1.00 – 1.50	0.80 – 1.05	0.50 – 1.00
Moderate	N / A	2.01 – 2.20	0.41 – 0.60	0.61 – 0.80	1.51 – 1.80	1.06 – 1.14	1.01 – 1.60
High	See	1.81 – 2.00	0.61 – 0.80	0.81 – 1.00	1.81 – 2.50	1.15 – 1.19	1.61 – 2.00
Very High	(1)	1.50 – 1.80	0.81 – 1.00	1.01 – 1.20	2.51 – 3.00	1.20 – 1.60	2.01 – 2.40
Extreme	Above	< 1.50	> 1.00	> 1.20	> 3.00	> 1.60	> 2.40

Overall Near-Bank Stress (NBS) Rating

Figure 2.12 Near-Bank Stress (NBS) Rating Worksheet (Rosgen, 2014)

These seven methods are ordered based on the amount of time and resources needed to conduct the method. Method 1 (Presence of Transverse/Central Bars or Channel Pattern Changes) is the one of the most cost-effective and rapid of the methods to conduct while method 7 (Velocity Isovels) is the most costly and time-consuming. The field practitioner selects one or several of these methods that best represent the site conditions and then uses the highest (not the average) of these ratings as the dominant near-bank stress (Rosgen, 2009). Rosgen (2009) cautions users by stating: "The [order of methods] are not necessarily synonymous with reliability of prediction (pg. 5-68)." In other words, just because method 7 requires greater level of detail, does not mean that this is the only method that could be used to determine the dominant NBS rating. Figure 2.12 shows the NBS rating worksheet. The next sections will provide more detail on each of the seven methods presented.

2.3.2.1 Method 1: Presence of Transverse/Central Bars or Channel Pattern Changes

This method requires the field practitioner to look for any kind of excess deposition, such as central or transverse bars, that may be directing flow and creating a high shear stress in the near-bank region of the streambank in interest. If this is the case, a NBS rating of High to Extreme may be given. Additionally, the user can give the streambank an Extreme NBS rating if it appears that the meander is migrating downstream, based on aerial imagery assessment (Rosgen, 2009).

2.3.2.2 Method 2: Radius of Curvature-to-Bankfull Width ratio

In this method, the radius of curvature of the meander bend containing the streambank of interest is measured. This is then divided by the bankfull width of the channel, as measured at a representative riffle within the reach. The radius of curvature can be obtained in the field or utilizing an aerial photograph. Figure 2.13 shows how to measure a meander's radius of curvature. The greater this ratio, the lower the expected near bank stress is on the streambank in interest (Rosgen, 2009). Rating cut-offs are presented at the bottom of the NBS rating worksheet in Figure 2.12. This method requires a cross-sectional survey at the midpoint of a representative riffle within the reach to determine bankfull width based on visual bankfull indicators and calibration using streamflow or regional curve data.

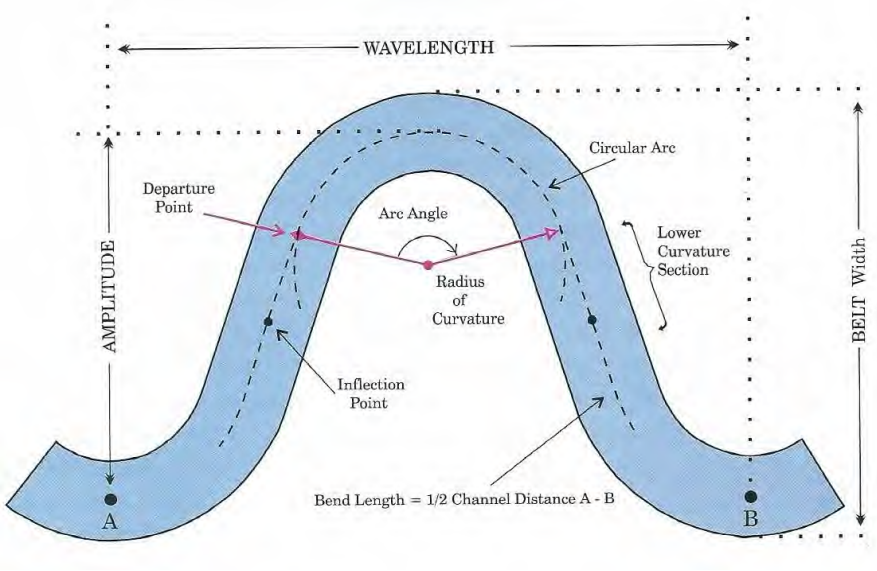


Figure 2.13 Radius of Curvature and Meander Geometry (Rosgen, 1996b)

2.3.2.3 Method 3: Pool Slope-to-Average Water Surface Slope ratio

This method can be used to determine NBS rating if the eroding streambank of interest is located along a pool. Pool slope is measured as the slope of the water surface from the end of a run to the start of glide along the pool of interest. The average water surface slope is measured from head-of-riffle to head-of-riffle of the given stream reach within which the streambank of interest is located. Alternatively, the average bankfull slope could also be used, as the average water surface slope and average bankfull slope should be equal. Average water surface slope, bankfull slope, and individual facet slopes for various features (i.e. pools, riffles) are depicted in Figure 2.14. The pool slope is then divided by the average slope of the stream to obtain a ratio value. The greater the ratio, the greater the near-bank stress on the eroding streambank (Rosgen, 2009). Rating cut-offs are presented at the bottom of the NBS rating worksheet in Figure 2.12. This method requires a longitudinal survey of a reach at least twenty bankfull widths long or two full meander wavelengths (Rosgen, 1996b).

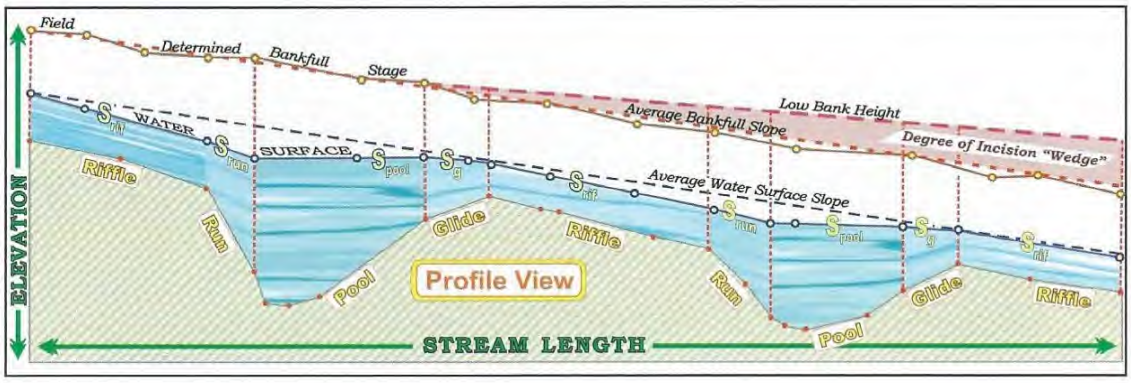


Figure 2.14 Slope Measurements (Rosgen, 2009)

2.3.2.4 Method 4: Pool Slope-to-Riffle Slope ratio

Once again, this method can be utilized to determine NBS rating if the eroding streambank of interest is located along a pool. If so, the pool slope is measured, as described for method 3, along the water surface from end of run to start of glide. The riffle slope must be measured along the riffle upstream of the pool. This slope is measured along the water surface from the head of riffle to start of run. These slope measurements are depicted in Figure 2.14. The pool slope is then divided by the riffle slope to obtain a ratio value. When pool slope is greater than the slope of the upstream riffle, also known as a slope reversal, streambank erosion increases. Therefore, as this ratio increases, so does the near bank stress along the streambank of interest (Rosgen, 2009). Rating cut-offs are presented at the bottom of the NBS rating worksheet in Figure 2.12. Similar to method 3, this method requires a longitudinal survey of a reach at least twenty bankfull widths long or two full meander wavelengths (Rosgen, 1996b).

2.3.2.5 Method 5: Near-bank Maximum Depth-to-Bankfull Mean Depth ratio

This method requires the field practitioner to survey a cross section at the eroding streambank of interest to obtain maximum depth and mean depth at bankfull stage (Rosgen, 2014). Bankfull mean depth is calculated by dividing the bankfull cross-sectional area by the bankfull width. Near-bank maximum bankfull depth is then divided by the bankfull mean depth to obtain a ratio value. As the ratio of the maximum bankfull depth to the average bankfull depth increases, the near bank stress rating also increases (Rosgen, 2009). Rating cut-offs are presented at the bottom of the NBS rating worksheet in Figure 2.12. It should be noted that in the WARSSS textbook (Rosgen, 2009), the description of this method states: "This method calculates the ratio of the near-bank maximum bankfull depth at a study site to mean depth from

a *riffle* cross-section (p 5-74)." This description is corrected in Rosgen (2014) and is in fact, the bankfull mean depth at the eroding streambank of interest.

2.3.2.6 Method 6: Near-bank Shear Stress-to-Bankfull Shear Stress ratio

Shear stress (τ) in steady, uniform flow conditions is calculated using the following equation:

$$\tau = \gamma RS \qquad \text{(Eq. 1)}$$

Where γ = specific weight of the fluid, R = hydraulic radius = cross-sectional area/wetted perimeter, and S = average water surface slope. This same equation is what is used to calculate applied shear stress, the driving force in the hydraulic force balance described in Section 2.1. For this application, Rosgen (2009) assumes that the channel is a wide channel and therefore R is roughly equal to the depth of the channel (d), simplifying equation 1 to:

$$\tau = \gamma dS \qquad \text{(Eq. 2)}$$

Therefore, the equations to calculate near-bank shear stress (τ_{nb}) and bankfull shear stress (τ_{bkf}) are as follows:

$$\tau_{nb} = \gamma d_{nb} S_{nb} \qquad \text{(Eq. 3)}$$

$$\tau_{bkf} = \gamma d_{bkf} S \qquad \text{(Eq. 4)}$$

Where d_{nb} = the near-bank maximum depth in line with the streambank of interest, d_{bkf} = mean bankfull depth also in line with the streambank of interest, and S_{nb} = near-bank water surface slope. S_{nb} is equal to the facet slope of the bed feature adjacent to the streambank of interest. In most cases, S_{nb} is just the pool slope. The near-bank shear stress is then divided by the bankfull shear stress to obtain a ratio value. The greater the near-bank shear stress is compared to the bankfull shear stress, the greater the erosion potential of the bank of interest and the higher the NBS rating (Rosgen, 2009). Rating cut-offs are presented at the bottom of the NBS rating worksheet in Figure 2.12. This method requires survey of a representative riffle as well as longitudinal profile survey of at least twenty bankfull widths long or two full meander wavelengths (Rosgen, 1996b).

2.3.2.7 Method 7: Velocity Isovels

This method requires the greatest amount of field data collection. Vertical velocity profiles across the channel perpendicular to the bank of interest must be collected using current meters during at or near bankfull flow events. Lines of equal velocities (isovels) are then plotted

on a cross section. Next, distance is measured from the streambank of interest to the core, or the fastest velocity, of the cross section. Velocity gradient is calculated by dividing the fastest velocity of the cross-section by this distance. An example of determining the velocity gradient is shown in Figure 2.15. The greater the velocity gradient is, the greater the near-bank stress and rating (Rosgen, 2009). Rating cut-offs are presented at the bottom of the NBS rating worksheet in Figure 2.12.

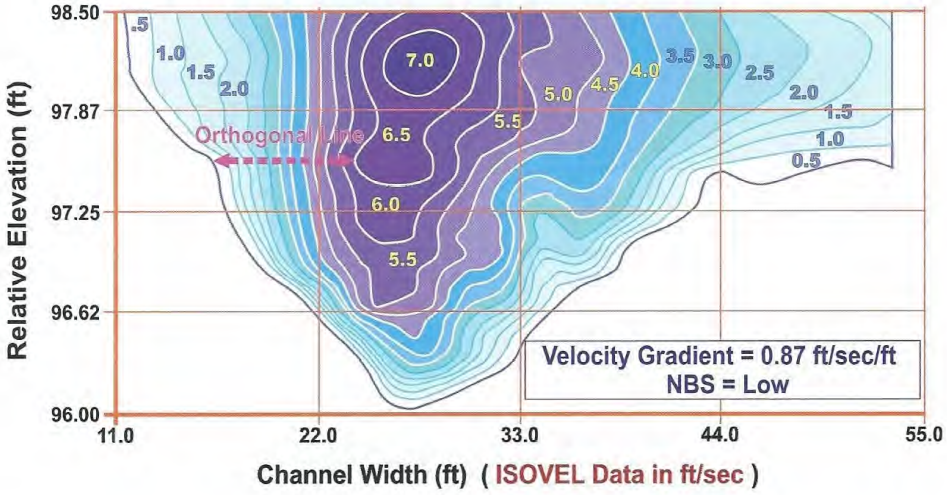


Figure 2.15 Example of Velocity Isovels and Gradient Calculation (Rosgen, 2009)

2.3.3 Measuring Annual Streambank Retreat Rates

The final item needed to create a BANCS model is manually measured bank retreat rates obtained from various BEHI/NBS-rated streambanks within a given hydrophysiographic region over a one-year time period. This can be accomplished in numerous ways, for example, utilizing erosion pins, terrestrial photogrammetry, and repeated cross profiling. Each technique has its advantages and limitations that should be taken into consideration before selection (Lawler, 1993a). For this application, Rosgen (2009) recommends the use of repeated cross profiling in combination with erosion pins.

Repeated cross-profiling or bank profiles are permanent, monumented cross sections at the streambanks of interest. These cross sections are marked with a toe pin of known elevation that is placed at or near the toe of the study streambank. A survey rod is then placed plumb on the toe pin so that the field practitioner can "profile" the study bank by taking level, horizontal measurements to the study bank at given vertical distances up the survey rod. The study bank can then be profiled at some time later (e.g. one year) so that when the two profiles of the same study

bank are superimposed on one another, it is possible to calculate the bank retreat or change in area over time. This change in area is divided by the study bank height to obtain an average bank retreat rate in unit length per time. To develop a BANCS model, the lateral retreat rate should be in units of length per year. Bank erosion pins may also be installed, but are not required. These pins are utilized to rapidly assess bank erosion rates throughout the rainy season (Rosgen, 2009). Figure 2.16 depicts the bank profile procedure.

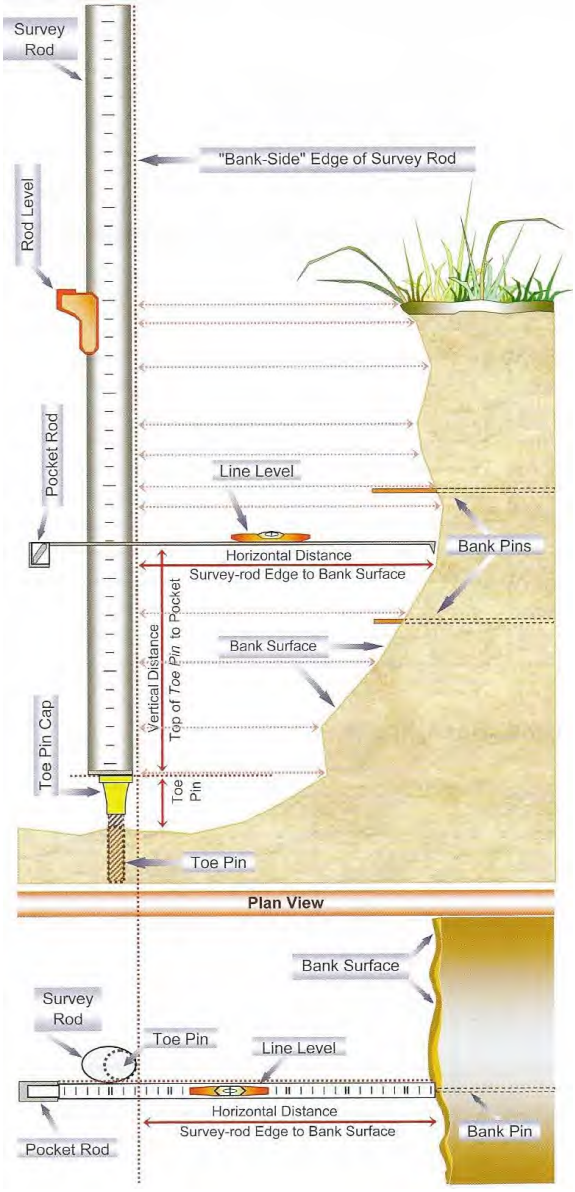


Figure 2.16 Example of Bank Profile Method and Erosion Pin Installation (Rosgen, 2009)

Appendix C
Quality Assurance Project Plan

Portland Harbor Superfund Site
Willbridge Cove Project Area Remedial Design

Pre-Design Investigation Quality Assurance Project Plan

Final

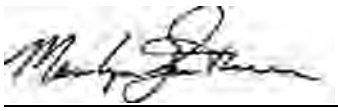
June 10, 2021

Prepared for the Willbridge Cove Group

Prepared by Jacobs (Willbridge Cove Project RD Supervising Contractor)
Reviewed by EHS Support (Willbridge Cove Project RD Project Coordinator)
For Submittal to EPA Region 10

Approvals

1. Jacobs Project Manager: Marilyn Gauthier

Signature:  Date: 05/03/2021

2. Jacobs Quality Assurance Manager: Kris Ivarson

Signature: Date: 05/03/2021

3. Jacobs Project Chemist: Bernice Kidd

Signature: _____ Date: 05/03/2021

4. EPA Region 10 Project Coordinator: Ben Leake

Signature: _____ Date: _____

5. EPA Region 10 Quality Assurance Officer: To Be Determined

Signature: _____ Date: _____

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Acronyms and Abbreviations

Acronym	Definition
≥	greater than or equal to
<	less than
?	less than or equal to
%R	percent recovery
°C	degree(s) Celsius
µg/kg	microgram(s) per kilogram
µg/L	microgram(s) per liter
ASAOC	Administrative Settlement Agreement and Order on Consent
CCB	continuing calibration blanks
CCV	continuing calibration verification
CUL	cleanup level
DDD	dichlorodiphenyldichloroethane
DDE	dichlorodiphenyldichloroethane
DDT	dichlorodiphenyltrichloroethane
DDx	sum of six isomers associated with DDT, DDD, and DDE
DFTPP	decafluorotriphenylphosphine
DMC	deuterated monitoring compound
DQO	data quality objective
DRO	diesel-range organics
EHS	Environmental, Health and Safety
EICP	extracted ion current profile
EPA	U.S. Environmental Protection Agency
FSP	Field Sampling Plan
FTL	Field Team Leader
FTM	Field Task Manager

GC	gas chromatography
GRO	gasoline-range organics
HSP	Health and Safety Plan
ICAL	initial calibration
ICP	inductively coupled plasma
ICS	interference check solution
ICV	initial calibration verification
IDW	investigation-derived waste
Jacobs	Jacobs Engineering Group Inc.
LCS/LCSD	laboratory control sample/laboratory control sample duplicate
MDL	method detection limit
mL	milliliter(s)
MS/MSD	matrix spike/matrix spike duplicate
N/A	not applicable
oz.	ounce
PAH	polycyclic aromatic hydrocarbon
PARCCS	precision, accuracy, representativeness, completeness, comparability, and sensitivity
PC	project coordinator
PCB	polychlorinated biphenyl
PDI	Pre-Design Investigation
PHSS	Portland Harbor Superfund Site
PM	project manager
QA	quality assurance
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
QC	quality control
RD	Remedial Design

RF	response factor
RL	reporting limit
RPD	relative percent difference
RRF	relative response factor
RRO	residual-range organics
RSD	relative standard deviation
SOP	standard operating procedure
SOW	Statement of Work
SVOC	semi-volatile organic compound
TBD	to be determined
TPH	total petroleum hydrocarbons
UFP	Uniform Federal Policy
VOA	volatile organic analysis
VOC	volatile organic compound
WCPA	Willbridge Cove Project Area
WP	Work Plan

Note: If using a screen reader, adjustment to your default settings may be required.

1. Introduction

This Quality Assurance Project Plan (QAPP) was prepared to present the project-specific quality assurance/quality control (QA/QC) requirements for the sediment and river bank soil sampling at the Willbridge Cove Project Area (WCPA) located within the Portland Harbor Superfund Site (PHSS) in Portland, Oregon. This QAPP is an integral part of the Pre-Design Investigation (PDI) Work Plan (WP) that governs all sampling and analysis activities for the WCPA. These plans ensure that data of appropriate quality are collected and meet the project-specific requirements.

This QAPP presents the QA/QC requirements designed to ensure that environmental data collected for the site and laboratory analyses are of the appropriate quality to achieve the project objectives defined in the PDI WP. These documents describe the background of the site, sources of contamination, information derived from previous investigations, and descriptions of field methods for each task. Additionally, the PDI WP and associated Field Sampling Plan (FSP) discusses the specific protocols for sample collection, equipment decontamination, handling of investigation-derived waste (IDW), sample handling and storage, and field QC. This performance based QAPP is intended to document laboratory quality requirements for the Portland Harbor Record of Decision (ROD) Table 17 and Table 21 parameters for soil and sediment. The PDI WP specifies the sampling and analytical schedule for a given phase of work, and the subset of the ROD Table 17 or Table 21 constituents needed will be dependent upon the objectives established in the PDI WP.

This QAPP specifies the requirements for laboratory analyses, data handling, data evaluation and assessment performance evaluations, chain-of-custody requirements, corrective actions, preventive maintenance of equipment, and additional information regarding sample handling and storage and field QC.

The elements included in this QAPP are consistent with those specified in the U.S. Environmental Protection Agency (EPA) *Requirements for Quality Assurance Project Plans, EPA QA/R-5* (EPA 2001). Table C-1-1 provides a crosswalk table of elements of the Uniform Federal Policy (UFP) QAPP (EPA 2005), EPA QA/R-5, and this QAPP. The objectives of the QAPP are to:

- Ensure that data collection and measurement procedures are standardized.
- Monitor the performance of the various measurement systems being used in the program to maintain statistical control and provide rapid feedback, so that corrective measures, if needed, can be taken before the data quality is compromised.
- Periodically assess the performance of these measurement systems and their components.
- Verify that reported data are sufficiently complete, comparable, representative, unbiased, and precise, so that they are suitable for their intended use.
- Define the following:
  - Who will use the data
  - What are the project's goals and objectives
  - What decisions will be made from the information obtained
  - How, when, and where will project information be acquired or generated
  - What possible problems may arise and what actions can be taken to mitigate their impact on the project
  - What type, quantity, and quality of data are specified
  - How "good" data have to be to support project decisions
  - How the data will be analyzed, assessed, and reported

This QAPP is intended for use by Jacobs Engineering Group Inc. (Jacobs) and its subcontractors who provide services associated with the environmental data collection effort. This QAPP supplements the PDI WP, FSP, and other project-specific documents.

This QAPP may be revised as necessary when guidelines and regulatory documents are revised, or when additional sampling or analysis methods are required. All contractors who might be affected by the revisions will be included in the review process and all revisions will be approved by EPA. Specific deviations or departures from the provisions in this QAPP may be called out in the PDI WP, approved through that review process, but must still meet QAPP requirements. In the case that specific revisions or deviations to this QAPP are called out and approved in the PDI WP, this document does not have to be revised.

Table C-1-1. Elements of the UFP QAPP and EPA QA/R-5 in Relation to This QAPP

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Optimized UFP QAPP Worksheets		EPA QA/R-5	Jacobs QAPP	Other Jacobs Project Documents
1 and 2	Title and Approval Page	A1 Title and Approval Sheet	Title Sheet	
3 and 5	Project Organization and QAPP Distribution	A3 Distribution List	Section 2	
4, 7, and 8	Personnel Qualifications and Sign-off Sheet	A4 Project/Task Organization A8 Special Training/Certification	Section 2	
6	Communication Pathways	--	Section 2	
9	Project Planning Session Summary	--	N/A	N/A
10	Conceptual Site Model	A5 Problem Definition/Background		FSP Section 1.1
11	Project/DQOs	A7 Quality Objectives and Criteria		PDI WP, Section 3 and Table 3-1 2
12	Measurement Performance Criteria	A7 Quality Objectives and Criteria	Section 5.1 and Section 5 Tables	
13	Secondary Data Uses and Limitations	B9 Non-Direct Measurements	Section 13	N/A
14 and 16	Project Tasks and Schedule	A6 Project/Task Description		PDI WP Section 7
15	Project Action Limits and Laboratory-Specific Detection /Quantitation Limits	--	Section 5 Tables	PDI WP Section 2 presents CULs and remedial action levels for PHSS
17	Sampling Design and Rationale	B1 Sampling Process Design (Experimental Design)		FSP Section 2
18	Sampling Locations and Methods	B2 Sampling Methods		FSP Section 3 and 4
19 and 30	Sample Containers, Preservation, and Hold Times	B4 Analytical Methods	Section 4.1	FSP Section 5

Table C-1-1. Elements of the UFP QAPP and EPA QA/R-5 in Relation to This QAPP

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Optimized UFP QAPP Worksheets		EPA QA/R-5	Jacobs QAPP	Other Jacobs Project Documents
20	Field QC	B5 Quality Control	Section 3.3	FSP Section 5
21	Field SOP	B2 Sampling Methods		FSP Sections 3 and 4
22	Field Equipment Calibration, Maintenance, Testing, and Inspection	B6 Instrument/Equipment Testing, Inspection, and Maintenance B7 Instrument/Equipment Calibration and Frequency B8 Inspection/Acceptance of Supplies and Consumables	Section 6.1 Section 5.4.5	FSP Section 6.3
23	Analytical SOP	B4 Analytical Methods	Section 5	FSP Section 5
24	Analytical Instrument Calibration	B7 Instrument/Equipment Calibration and Frequency	Section 6.2 and Section 5 Tables	
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection	B6 Instrument/Equipment Testing, Inspection, and Maintenance	Section 6.2 and Section 5 Tables	
26 and 27	Sample Handling, Custody, and Disposal	B3 Sample Handling and Custody	Sections 4.2 and 4.3	FSP Section 6
28	Analytical Quality Control and Corrective Action	B5 Quality Control	Sections 5.3 and 5.4 and Section 5 Tables	
29	Project Documents and Records	A9 Documents and Records B10 Documentation/Data Management	Section 12 Section 13	
31, 32 and 33	Assessments and Corrective Action	C1 Assessments and Response Actions C2 Reports to Management	Section 8 Section 11	
34	Data Verification and Validation Inputs	D1 Data Review, Verification, and Validation D2 Data Verification and Validation Methods	Section 7	
35	Data Verification Procedures	D1 Data Review, Verification, and Validation D2 Data Verification and Validation Methods	Section 7	

Table C-1-1. Elements of the UFP QAPP and EPA QA/R-5 in Relation to This QAPP

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Optimized UFP QAPP Worksheets		EPA QA/R-5	Jacobs QAPP	Other Jacobs Project Documents
36	Data Validation Procedures	D1 Data Review, Verification, and Validation D2 Data Verification and Validation Methods	Section 7	
37	Data Usability Assessment	D3 Reconciliation with User Requirements	Section 12	

Notes:

CUL = cleanup level

DQO = data quality objective

N/A = not applicable

SOP = standard operating procedure

2. Program Organization and Responsibility

This section identifies and describes the responsibilities of key project positions related to project management, field task management, QA/QC management, and data management. Contact information for the Environmental, Health and Safety (EHS) Support and Jacobs staff is provided in Table C-2-1.

The QAPP will be maintained by the Jacobs PDI Project Manager (PM). If updates are needed, the document will be updated and recirculated to the staff in Table C-2-1.

2.1 Project Staff and Responsibilities

Consultants and contractors retained by the Willbridge Cove Group will perform PDI and Remedial Design (RD) sampling and analysis activities. The consultant team includes EHS Support as the Project Coordinator (PC) and Jacobs as the supervising contractor. The consultant team is responsible for implementation of these tasks at the direction and oversight of the Willbridge Cove Group.

2.1.1 Willbridge Cove Area Project Coordinator

The EHS Support PC's responsibilities include:

- Serving as the primary point of contact responsible for the coordination and management of the work required in the Administrative Settlement Agreement and Order on Consent (ASAOC) for RD for the WCPA dated January 31, 2020
- Providing quarterly progress reports to the EPA
- Oversight of the supervising contractor
- Planning, facilitating, and leading meetings between the supervising contractor, the Willbridge Cove Group, and other agencies or stakeholders.

2.1.2 Project Manager

The Jacobs PM's responsibilities include:

- Development and implementation of the project
- Technical oversight of all investigative and routine monitoring and sampling
- Schedule, financial status, technical status, and contract management
- Overall project QA
- Interfacing with the PC, Task Manager, Field Task Manager (FTM), Project Chemist, and Data Manager
- After a QA review by an independent senior review team, the PM and senior review team will identify appropriate corrective action(s) to be initiated if QA problems or deficiencies requiring special action are discovered

2.1.3 Task Manager

- Technical oversight of all investigative and routine monitoring and sampling
- Overall project QA
- Management of project tasks associated with sampling, general QA, oversight of field personnel in sampling activities, coordination of sample collection, and coordination of sample submittal to the analytical lab

2.1.4 Field Task Manager

The FTM's responsibilities include:

- Coordinating field schedules
- Coordinating Jacobs field personnel and subcontractors at the site
- Maintaining communication with the laboratory regarding scheduled sampling events and coordinating delivery of samples to the laboratory
- Managing sample tracking, sample analysis, and data reporting from each laboratory
- Managing project tasks associated with sampling, providing general QA, overseeing field personnel in sampling activities, coordinating sample collection, and coordinating sample submittal to the analytical lab
- Collecting and reviewing all field task-related documents and archiving the documents in the project file

2.1.5 Project Chemist

The Project Chemist's responsibilities include:

- Approving and maintaining adherence to QA/QC requirements specified in this QAPP
- Providing guidance regarding environmental analytical chemistry methods and QC procedures applicable to environmental analytical chemistry
- Coordinating or performing validation of the analytical data
- Performing quality audits and surveillance, preparing QA reports, implementing QC activities, and suggesting corrective actions, as necessary
- Communicating QA/QC issues to the PM, Task Manager, FTM, and Project Data Manager
- Recommending resolution for any anomalies or out-of-control events that arise during the analysis of samples

2.1.6 Data Manager

The Data Manager's responsibilities include:

- Maintaining overall management and control of all analytical and field data that will be used for decision making and project reporting purposes

2.1.7 Site Safety Coordinator

The Site Safety Coordinator's responsibilities include:

- Site safety for Jacobs and subcontracted personnel working on the project
- Implementation of the Health and Safety Plan (HSP), contractor safety, and training

2.1.8 Quality Assurance Manager

The Quality Assurance Manager (QAM) will oversee all aspects of project QA/QC, responsibilities, to include:

- Review and approval of this QAPP
- Planning for audits or inspections of field or laboratory work
- Oversight of quality implementation and/or need for corrective actions, including review of field and laboratory non-conformance and corrective actions during data collection and generation

- Review of field and laboratory reports
- Communicates with project team on accuracy and completeness of deliverables, including results of audits, evaluation of subcontractor Statement of Work (SOW), and WP
- Data validation oversight
- Assessing final data usability, limitations, and completeness
- Document conformance with the project objectives and requirements.

Table C-2-1. Contact Information for Project Staff

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Title	Name	Phone	Email
PC	John Lang	(513) 325-2732	john.lang@ehs-support.com
PM	Marilyn Gauthier	(425) 894-6464	Marilyn.Gauthier@jacobs.com
Task Manager	Keith Sheets	(510) 541-8542	Keith.Sheets@jacobs.com
FTM	TBD	TBD	TBD
Jacobs Project Chemists/Data Validators	Berney Kidd David Summerville	(530) 229-3203 (907) 762-1106	Bernice.Kidd@jacobs.com David.Summerville@jacobs.com
Jacobs Data Manager	Leslie Karlewicz	(206) 910-0860	Leslie.Karlewicz@jacobs.com
Jacobs QA Manager	Kris Ivarson	(509) 302-3472	Kris.Ivarson@jacobs.com
Site Safety Liaison	TBD	TBD	TBD
Laboratory Contacts	Todd Poyfair/ALS Project Manager Howard Boorse/ALS Senior Chemist	360-218-8601 360-430-7733	Todd.Poyfair@alsglobal.com Howard.Boorse@alsglobal.com

Note:

TBD = to be determined

Table C-2-2. Communication Pathways

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Communication Drivers	Responsible Entity	Name	Phone	Procedure (Timing, Pathways, etc.)
Communication with EPA (via the Willbridge Cove Group PC)	PC	John Lang	(513) 325-2732	Pathway: Act as liaison between PRPs and U.S. government agency. Documentation: Review and approve necessary documents associated with sampling and results. Provide written notice to proceed and approval of reports. Mode: email, phone Timing: as necessary

Table C-2-2. Communication Pathways

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Communication Drivers	Responsible Entity	Name	Phone	Procedure (Timing, Pathways, etc.)
Communication with project team	Jacobs PM	Marilyn Gauthier	(425) 894-6464	Pathway: Primary point of contact for team; can delegate communication to other internal or external points of contact; notifies Jacobs, PRPs, and EPA of project-related problems and issues Documentation: Provides direction to team; authorizes real-time changes to plan and can stop work if needed. Mode: email, phone Timing: as necessary
Technical Approach and Data Evaluation Review	Jacobs Subject matter Expert and Senior Technical Consultant	Jeff Gentry Theresa Himmer Patricia White	(503) 329-7738 (207) 407-3086 (617) 721-2527	Pathway: Communicate with Jacobs PM and Jacobs project team. Documentation: Provide input on strategy and technical approach; responds to technical or field questions; reviews data and technical deliverables as necessary; provides verbal or written comments to Jacobs PM on technical deliverables. Mode: email, phone, meetings Timing: as necessary
QA Management	Jacobs QAM	Kris Ivarson	(509) 302-3472	Pathway: Communicates with Jacobs PM and Jacobs project team Documentation: Responsible for implementation of the quality management system for all project work; communicates with Jacobs PM and project team on accuracy and completeness on major deliverables, including results of internal project and records audits; and evaluation of subcontractor SOW and WP document for conformance with the project objectives and requirements. Mode: email, phone, meetings Timing: as necessary
Progress of Field Program	Jacobs Task Manager	Keith Sheets	(510) 541-8542	Pathway: Communicates with Jacobs PM and Jacobs project team Documentation: Documents and conveys progress of field activities, including deviations from the FSP; directs field team; can stop work in the field. Any corrective actions for field issues will be determined by a Task Manager. Ensure information is captured in reports/deliverables accurately for field activities. Mode: email, phone, meetings Timing: as necessary; corrective actions for field issues will be reported to the Jacobs PM and/or QAM within 4 hours

Table C-2-2. Communication Pathways

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Communication Drivers	Responsible Entity	Name	Phone	Procedure (Timing, Pathways, etc.)
Health and Safety Issues	Jacobs Responsible Health and Safety Manager	John Culley/ Jacobs	(509) 464-7228	Pathway: Communicates with Jacobs PM and Jacobs QAM Documentation: Responsible for supporting the team by developing health and safety requirements; approves HSP; conducts field safety audits. Mode: email, phone. Timing: as necessary; health and safety issues will be reported to the Jacobs Project Manager and QAM immediately
Provide daily progress reports on field effort and Health and Safety concerns	Jacobs FTM and Site Safety Liaison	TBD	TBD	Pathway: Communicates with Jacobs PM, Jacobs Task Manager, Jacobs Project Chemists, Jacobs QAM, and Jacobs Responsible Health and Safety Manager Documentation: The FTM will provide the Jacobs PM and task manager with written daily progress reports, including field records, sampling logs, chain-of-custody records, and any other pertinent information. The Site Safety Liaison is responsible for the adherence of team members and subcontractors to the site safety requirements described in the HSP. Will provide daily safety briefings and directs onsite safety activities and report health and safety incidents and near misses to the PM and health and safety manager. Timing: Daily field progress reports within 2 days; site safety briefings are daily, report health and safety incidents and near misses within 4 hours
Communication with Laboratory and Release of Analytical Data	Jacobs Senior Project Chemist	Berney Kidd David Summerville	(530) 229-3203 (907) 762-1106	Pathway: Communicates with Jacobs PM, Jacobs Task Manager, Jacobs QAM, and project laboratories Documentation: Ensures that the laboratory meets QAPP requirements; provides direction on corrective action requirements for analytical issues. No analytical data can be released until data validation is completed and has been approved by the Project Chemists. Mode: email, phone, meetings Timing: data validation completed within 5 business days for receipt of data; analytical issues communicated to the project laboratory within 24 hours of identification

Note:

PRP = Potentially Responsible Parties

2.2 Training and Certification Requirements

All personnel engaged in field activities will have completed health and safety training that meets the requirements of the project-specific HSP. All subcontracted project personnel will read the project-specific HSP. Documentation will be maintained to demonstrate that all requirements of the plan are followed.

All laboratories providing analytical services for the site will be certified by the State of Oregon for the project-specific methods. Each Laboratory Manager will be responsible for ensuring that all personnel have been properly trained and are qualified to perform their assigned tasks.

2.3 Coordination with EPA

EPA is the lead oversight agency for all Portland Harbor in-water PDI activities. EPA will oversee the Willbridge Cove Group activities as described in the PDI WP. EPA will coordinate all state, tribal, and agency partner participation per Section 7 of the ASAOC SOW. The EPA PC is Hunter Young.

The PM or PC will notify the EPA PC at least 14 days prior to beginning any field activities so that EPA can schedule any oversight activities required. The PM or PC will also notify the EPA PC once field activities have been completed.

Split samples for chemical analyses can be provided to EPA upon request. The EPA PC should contact the PM to coordinate this activity and determine appropriate logistics. If EPA elects to collect split samples, collection at stations where field duplicates are taken is recommended so that the EPA comparison samples can be evaluated relative to the field and analytical variability measured by the project team.

3. Sampling Procedures

This section provides an overview as to how environmental samples will be collected and tracked. Field sampling forms and relevant field SOPs are included in the FSP.

3.1 Sampling Design

The number and location of samples for the site are discussed in the PDI WP (Section 3) and the FSP (Section 2). The rationale for the sampling design is also described in the PDI WP. The sampling design is a function of the medium sampled, information about the sampling site, the type of data to be collected, how the data are to be used, and existing Portland Harbor-specific sampling guidance (EPA 2017c, 2020). The specific protocols for sampling, equipment decontamination, handling of IDW, and field QC are discussed in the FSP.

3.2 Sampling Method Requirements

The FSP and associated SOPs address sampling methods.

3.3 Field Quality Control Samples

QC samples will be collected to monitor accuracy, precision, and the presence of field contamination for analytical methods to be performed in the offsite laboratory. Section 5.3.2 contains a description of field QC samples.

3.4 Sample Documentation and Tracking

Sample containers should be received from the laboratory pre-labeled with the preservative. The sample identification and date and time of sampling are entered on the label immediately after collection. The labels must be secured using clear tape to maintain the identification of each sample.

Vital information regarding the collection of each sample will be recorded in a field logbook. A separate logbook will be used for this site. It will be bound with consecutively numbered pages. All entries will be legibly written in black ink and signed and dated by the individual making the entries. Factual and objective language will be used. All entries will be complete and accurate enough to allow reconstruction of each field activity. Information recorded during the collection of each sample will include:

- Sample location and description (sketches and measured distances from reference points will be recorded if no established identification for sample location)
- Sample identification
- Sampler's name
- Date and time of sampling
- Sample designation as composite or grab
- Sample matrix
- Type and identification of sampling equipment used
- Field measurement data (e.g., pH, temperature, and conductivity)
- Field observations that may be relevant to the analysis or sample integrity (odor, color, and weather conditions)
- Associated QC blanks

- Preservative used
- Lot numbers of sample containers, chain-of-custody number, and custody seal number
- Shipping arrangement
- Destination laboratory

4. Sample Handling and Custody

This section summarizes sampling handling and custody protocols.

4.1 Containers and Preservatives

Laboratories will provide the required sample containers for all environmental and associated QC samples. All containers will be certified free of the analytes of concern for this project. No sample containers will be reused. The contracted laboratory will add preservatives, if required, prior to shipping the sample containers to the field. The laboratory, upon receipt of the samples, will verify the adequacy of preservation and will add additional preservative, if necessary. Samples requesting volatile organic compound (VOC) analyses should not undergo preservation verification until the time of analysis. The containers, minimum sample quantities, required preservatives, and maximum holding times are shown in Tables C-4-1 through C-4-5.

Table C-4-1. Sample Containers, Preservation, and Holding Times – Aqueous
Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	Method	Container and Minimum Quantity	Preservation	Holding Time
Metals	SW6020B/ SW7470A	1 × 500 mL, P	Nitric acid to pH < 2; chill to ≤ 6°C (field filter prior to acid preservation if dissolved metals required)	28 days for mercury; 180 days for all other metals
TPH-DRO	NWTPH-Dx	2 × 250-mL, G-TLC	Hydrochloric acid to pH < 2; chill to ≤ 6°C	14 days to extraction; 40 days to analysis
PAH	SW8270D-SIM	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	7 days to extraction; 40 days to analysis
Low-level bis(2-ethylhexyl)phthalate	SW8270D-LL	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	7 days to extraction; 40 days to analysis
Low-Level Pesticides	E1699	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	7 days to extraction; 40 days to analysis
Low-Level Pesticides (DDx only)	E1699M	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	7 days to extraction; 40 days to analysis
PCB Aroclors	SW8082-LL	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	1 year to extraction; 1 year to analysis

Table C-4-1. Sample Containers, Preservation, and Holding Times – Aqueous

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	Method	Container and Minimum Quantity	Preservation	Holding Time
PCB Congeners	E1668	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	1 year to extraction; 1 year to analysis
Dioxins/Furans	E1613B	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C; store in dark	1 year to extraction; 1 year to analysis

Notes:

Bottle types may vary based on availability or sharing.

< = less than

≤ = less than or equal to

°C = degree(s) Celsius

AG = amber glass

G = glass

G-TLC = glass with Teflon-lined cap

mL = milliliter(s)

P = polyethylene

PAH = polycyclic aromatic hydrocarbon

PCB = polychlorinated biphenyl

TPH-DRO = total petroleum hydrocarbons – diesel-range organics

Table C-4-2. Sample Containers, Preservation, and Holding Times – Solid

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	Method	Container and Minimum Quantity	Preservation	Holding Time
Metals	SW6020B/ SW7471	4-oz., G	None	28 days for mercury; 180 days for all other metals
TPH-DRO	NWTPH-Dx	4-oz., G	Cool ≤ 6°C	14 days to extraction; 40 days to analysis
PAH	SW8270D-SIM	8-oz., G	Cool ≤ 6°C	14 days to extraction; 40 days to analysis
Low-level bis(2-ethylhexyl)phthalate	SW8270D-LL	4-oz., G	Cool ≤ 6°C	14 days to extraction; 40 days to analysis
Low-Level Pesticide	E1699	4-oz., AG	Cool ≤ 6°C	14 days to extraction; 40 days to analysis
PCB Congeners	E1668	4-oz., AG	Cool ≤ 6°C	1 year to extraction; 1 year to analysis
Low-Level Pesticide (DDx only)	E1699M	8-oz., AG	Cool ≤ 6°C	14 days to extraction; 40 days to analysis
PCB Aroclors	SW8082-LL		Cool ≤ 6°C	1 year to extraction; 1 year to analysis
Total Organic Carbon	SW9060		Cool ≤ 6°C; store in dark	28 days
Dioxins/Furans	E1613B	8-oz., AG	Cool ≤ 6°C; store in dark	1 year to extraction; 1 year to analysis
Grain Size	ASTM D6913	8-oz., G	None	N/A
Grain Size with hydrometer	ASTM D7928			

Notes:

Bottle types may vary based on laboratory requirement or potential to take multiple analyses from a single container.

ASTM = ASTM International

DI = Deionized

oz. = ounce(s)

VOA = volatile organic analysis

Table C-4-3. Sample Containers, Preservation, and Holding Times – Archived Solid

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	Method	Container and Minimum Quantity	Preservation	Holding Time
Metals	SW6010B or C/ SW7471A or B	4-oz., G	Freeze ≤-10°C	1 year for mercury; 2 years for all other metals
TPH-DRO	NWTPH-Dx	4-oz., G	Freeze ≤-10°C	1 year to extraction; 40 days to analysis
PAH	SW8270 D-SIM	8-oz., G	Freeze ≤-10°C	1 year to extraction; 40 days to analysis
Low-level bis(2-ethylhexyl)phthalate	SW8270 D-LL	4-oz., G	Freeze ≤-10°C	1 year to extraction; 40 days to analysis
Low-Level Pesticide	E1699	4-oz., AG	Freeze ≤-10°C	1 year to extraction; 40 days to analysis
Low-Level Pesticides (DDx only)	E1699m	4-oz., AG	Freeze ≤-10°C; store in dark	1 year to extraction; 40 days to analysis
PCB Aroclors	SW8082-LL			1 year to extraction; 1 year to analysis
Total Organic Carbon	SW9060			1 year to analysis
PCB Congeners	E1668	4-oz., AG	Freeze ≤-10°C	1 year to extraction; 1 year to analysis
Dioxins/Furans	E1613B	8-oz., AG	Freeze ≤-10°C; store in dark	1 year to extraction; 1 year to analysis
Grain Size (sieve and hydrometer)	ASTM D6913 / ASTM D7928	8-oz., G	None	NA

Notes:

Bottle types may vary based on availability or sharing

Table C-4-4. Sample Containers, Preservation, and Holding Times – Aqueous Waste Characterization

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	Method	Container and Minimum Quantity	Preservation	Holding Time
Metals	SW6010B or C/ SW7470A	1 × 500 mL, P	Nitric acid to pH < 2; chill to ≤ 6°C (field filter prior to acid preservation if dissolved metals required)	28 days for mercury; 180 days for all other metals
TPH-DRO/RRO	SW8015B	2 × 250-mL, G-TLC	Cool ≤ 6°C	7 days to extraction; 40 days to analysis

Table C-4-4. Sample Containers, Preservation, and Holding Times – Aqueous Waste Characterization

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	Method	Container and Minimum Quantity	Preservation	Holding Time
SVOCs	SW8270C or D	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	7 days to extraction; 40 days to analysis
VOCs	SW8260B or C	3 × 40-mL, G-TLC	Hydrochloric acid to pH < 2; chill to ≤ 6°C; no headspace	14 days (preserved); 7 days (unpreserved)
TPH-GRO	SW8015B	3 × 40-mL, G-TLC	Hydrochloric acid to pH < 2; chill to ≤ 6°C; no headspace	14 days (preserved); 7 days (unpreserved)
Pesticides	SW8081A	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	7 days to extraction; 40 days to analysis
Herbicides	SW8151A	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	7 days to extraction; 40 days to analysis
PCBs	SW8082	2 × 1,000-mL, AG-TLC	Cool ≤ 6°C	1 year to extraction; 1 year to analysis
Corrosivity	SW9045C	1 × 500-mL, P	Cool ≤ 6°C	Immediately
Ignitability	SW1010	1 × 500-mL, P	None	N/A

Notes:

Waste samples are not validated, methods listed above may not be included in QA/QC tables

Bottle types may vary based on availability or sharing

Waste samples will be analyzed for Resource Conservation and Recovery Act of 1976 and/or State of Oregon required analytes

AG = amber glass

G = glass

G-TLC = glass with Teflon-lined cap

RRO = residual-range organics

SVOC = semi-volatile organic compound

TPH-GRO = total petroleum hydrocarbons gasoline-range organics

Table C-4-5. Sample Containers, Preservation, and Holding Times – Solid Waste Characterization

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	Method	Container and Minimum Quantity	Preservation	Holding Time
TCLP Metals	SW1311/SW6010B/ SW7470A	4-oz., G	None	180 days to leaching (28 days for mercury); then 28 days for mercury; 180 days for all other metals
TPH-DRO	SW8015	4-oz., G	Cool ≤ 6°C	14 days to extraction; 40 days to analysis
TCLP SVOC	SW1311/SW8270C	4-oz., G	Cool ≤ 6°C	14 days to leaching, then 7 days to extraction; 40 days to analysis
TCLP VOCs	SW1311/SW8260B	4-oz., G	Cool ≤ 6°C; no headspace	48 hours to leaching, then 7 days to analysis (unpreserved) or 14 days to analysis (preserved)
TPH-GRO	SW8015	4-oz., G	Cool ≤ 6°C; no headspace	14 days (preserved in methanol); 48 hours (unpreserved)
TCLP Pesticides	SW1311/SW8081A	4-oz., G	Cool ≤ 6°C	14 days to leaching, then 7 days to extraction; 40 days to analysis
TCLP Herbicides	SW1311/SW8151A	4-oz., G	Cool ≤ 6°C	14 days to leaching, then 7 days to extraction; 40 days to analysis
PCBs	SW8082	4-oz., G	Cool ≤ 6°C	1 year to extraction; 1 year to analysis
Corrosivity	SW9045C	4-oz., G	Cool ≤ 6°C	28 days
Ignitability	SW1030	4-oz., G	None	N/A

Notes:

Waste samples are not validated, methods listed above may not be included in QA/QC tables

Bottle types may vary based on availability or sharing

Waste samples will be analyzed for Resource Conservation and Recovery Act of 1976 and/or State of Oregon required analytes

TCLP = toxicity characteristic leaching procedure

4.2 Chain-of-Custody

Collecting data of known quality begins at the point of sample collection. Legally defensible data are generated by adhering to proven evidentiary procedures. These procedures are outlined in the following subsections and must be followed to preserve and ensure the integrity of all samples from the time of collection through analysis. Sample custody records must be maintained both in the field and in the subcontractor laboratory. A sample is in someone's custody if it is either in their physical possession or view, locked up, or kept in a secured and restricted area. Until shipment, sample custody will be the responsibility of the sampling team leader.

4.2.1 Chain-of-Custody Process

Chain-of-custody records document sample collection and shipment to the laboratory. A chain-of-custody form will be completed for each sampling event. The original copy will be provided to the laboratory with the sample shipping cooler, and a copy will be retained in the field documentation files and sent to the Project Chemist at the end of each day sampling is conducted. The chain-of-custody form will identify the contents of each shipment and maintain the custodial integrity of the samples. All chain-of-custody forms will be signed and dated by the responsible sampling team personnel. The "relinquished by" box will be signed by the responsible sampling team personnel, and the date, time, and air bill number will be noted on the chain-of-custody form. The laboratory will return the executed copy of the chain-of-custody with the hard copy report.

The shipping coolers containing the samples will be sealed with a custody seal any time the coolers are not in an individual's possession or view before shipping. All custody seals will be signed and dated by the responsible sampling team personnel.

At a minimum, the chain-of-custody form must contain:

- Site name
- PM, Project Chemist, and Data Manager names, telephone numbers, and email addresses
- Unique sample identification
- Date and time of sample collection
- Source of sample (including name, location, sample type, and matrix)
- Number of containers
- Designation of matrix spike/matrix spike duplicate (MS/MSD)
- Preservative used
- Analyses required
- Name of sampler
- Custody transfer signatures and dates and times of sample transfer from the field to transporters and to the laboratories
- Bill of lading or transporter tracking number (if applicable)
- Turnaround time
- Lab name, address, and contact information
- Laboratory purchase order number
- Project number
- Any special instructions

Erroneous entries on chain-of-custody records will be corrected by drawing a line through the error and entering the corrected information. The person performing the correction will date and initial each change made on the chain-of-custody form.

4.2.2 Laboratory Responsibilities

Once the samples reach the laboratory, they will be checked against information on the chain-of-custody form for anomalies. The condition, temperature, and appropriate preservation of samples will be checked and documented on the chain-of-custody form. Checking an aliquot of the sample using pH paper is an acceptable procedure (precautions must be taken to avoid contamination of the sample). Samples requesting VOC analyses will not undergo preservation verification until the time of analysis. The occurrence of any anomalies in the received samples and their resolution will be documented in laboratory records. All sample information will then be entered into a tracking system, and unique analytical sample identifiers will be assigned. A copy of this information will be reviewed by the laboratory for accuracy. Sample holding time tracking begins with the collection of samples and continues until the analysis is complete. Laboratory analyses will be documented on the chain-of-custody form. Procedures ensuring internal laboratory chain-of-custody will also be implemented and documented by the laboratory. Sample custody must be maintained using an internal custody system that requires samples to be kept in a secured and restricted area when not in use and to be checked out and checked back in by the analysts who use the samples. Internal custody records must be maintained by the laboratory as part of the documentation file for each sample. Specific instructions concerning the analysis specified for each sample will be communicated to the analysts. Analytical batches will be created, and laboratory QC samples will be introduced into each batch.

While samples are stored in the laboratory, samples will be stored in limited-access, temperature-controlled areas. Refrigerators, coolers, and freezers will be monitored for temperature 7 days a week. Acceptance criterion for the temperatures of the refrigerators and coolers is 0 to 6°C, but do not freeze. Acceptance criterion for the temperatures of the freezers will be less than -10°C. All cold-storage areas will be monitored by thermometers that have been calibrated with a NIST-traceable thermometer. As indicated by the findings of the calibration, correction factors will be applied to each thermometer. Records that include acceptance criteria will be maintained. Samples for volatile organics determination will be stored separately from other samples, standards, and sample extracts. Samples will be stored after analysis until disposed of in accordance with applicable local, state, and federal regulations. The laboratory will maintain disposal records.

Along with sample receipt documentation, the following information will be documented on sample receipt forms by the sample custodian:

- Date samples received
- Sample identification number
- Laboratory sample identification number
- Analytical tests requested for the sample batch
- Sample matrix
- Number of samples in the batch
- Container description and location in the laboratory
- Verification of sample preservation

SOPs describing sample control and custody will be maintained by the laboratory.

Samples designated as “HOLD” on the chain-of-custody form will be logged in upon receipt and held under chain-of-custody. Samples on “HOLD” are released for analysis by the Project Chemist via email

notification. The chain-of-custody form will be resubmitted to the Data Manager with relevant release notification.

Samples designated as "ARCHIVE" on the chain-of-custody form will be logged in upon receipt and stored under chain-of-custody. All "ARCHIVE" sediment samples submitted to the analytical laboratory will be archived at less than -10°C and all "ARCHIVE" water samples submitted to the analytical laboratory will be stored at 0 to 6°C. The extracts for water samples will be archived at less than -10°C. The laboratories will maintain proper storage conditions for the entire time that the samples are in their possession. All laboratories for this project will store the "ARCHIVE" samples for up to 12 months. The laboratories will not dispose of the samples for this project until they are authorized to do so by the Project QA Manager and/or EPA.

4.3 Sample Packaging and Transport

The following subsections contain guidelines for sample packaging and transport that may be superseded, amended, or replaced in the FSP.

4.3.1 Sample Container Preparation

- The labels will be secured to each container with clear tape, if not previously done.
- Container lids will be checked for tightness.
- Sample bottles will be double bagged in heavy-duty plastic. Glass containers will be covered with bubble wrap to prevent breakage, if being shipped.

4.3.2 Shipping Cooler Preparation

- All previous labels used on the sample shipping cooler will be removed.
- The drain plugs will be sealed with fiberglass tape (outside and inside) to prevent melting ice from leaking.
- A cushioning layer of packing material such as bubble wrap will be placed at the bottom of the cooler (approximately 1-inch thick) to prevent breakage during shipment.
- All ice will be either double bagged in a zip-locked plastic bag, or if all samples are double bagged, loose ice is acceptable inside of large plastic bag.

4.3.3 Placing Samples in the Cooler

- The chain-of-custody form will be placed in a zip-locked bag.
- Samples will be placed in an upright position in the cooler.
- Ice will be placed on top of samples and between samples. Ideally, ice will be placed in resealable plastic bags in duplicate to minimize leakage of ice melt into the cooler.
- Substantial void space between samples will be filled with packing material.

4.3.4 Closing the Cooler

- The cooler lid will be taped with strapping tape, encircling the cooler several times.
- Custody seals will be affixed to each cooler lid to further ensure the integrity of the samples. Custody seals will be used for every sample shipment.

4.3.5 Transport

- Sample coolers will be transported to the laboratory (an overnight courier may be used) as soon as practicable after sample collection. Intermediate stops will be avoided, except for emergencies only, in which case, the situation will be noted in the field notebooks.
- The laboratory will be notified that samples are being shipped.

5. Data Quality Objectives and Quality Assurance Program

The DQOs for each individual project were established based on the EPA document *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA 2006) and are specified in the PDI WP and FSP. They are the basis for the design of the data collection plan and, as such, these DQOs specify the type, quality, and quantity of data to be collected and how the data are to be used to make the appropriate decisions for the project.

The DQOs are developed through a seven-step process, each step of which derives valuable criteria that are used to establish the final data collection design. The first five steps of the process identify mostly qualitative criteria, such as what problem has initiated the project and what decision it attempts to resolve. These steps also define the type of data to be collected, where and when the data will be collected, and a decision rule that defines how the decision will be made; the DQO process and the first five steps are presented in the PDI WP. The sixth step defines quantitative criteria expressed as limits on decision errors that can be tolerated by the decisionmaker. Step 6 consists of the measurement indicators presented in this QAPP (see Section 5.1). The final step is the development of the data collection design using the criteria developed in the previous six steps. The final output of the process is a data collection design that meets the qualitative and quantitative needs of the project.

5.1 Precision, Accuracy, Representativeness, Completeness, Comparability and Sensitivity

Data quality will be evaluated based on the measurement performance criteria associated with specific analyses and include precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS). To meet PARCCS requirements, QC criteria are provided in the standard field and laboratory methods, plus laboratory-specific protocols. Performance criteria are listed in Tables C-5-2 through C-5-24 for each analyte group.

5.1.1 Precision

Precision is a measure of reproducibility of analytical results. It can be defined as the degree of mutual agreement among individual measurements obtained under similar conditions. Total precision is a function of the variability associated with both sampling and analysis. Precision will be evaluated as the relative percent difference (RPD) between field duplicate sample results and laboratory sample duplicates, between the laboratory control sample (LCS) and laboratory control sample duplicate (LCSD), or between the MS and MSD results. Field duplicates will comprise 5 percent of the sampling effort. Field duplicates will be split in the field from a homogenized sample. No field replicates (two unique samples) will be collected. MS/MSD samples will be field-designated at a 5-percent frequency.

Precision is evaluated by calculating the RPD using the following equation:

$$RPD = \frac{(D_1 - D_2) \times 100}{\overline{D}}$$

Where:

D_1 = original sample concentration
D_2 = replicate sample concentration
$\overline{D}$ = the mean of D_1 and D_2
RPD = relative percent difference

Precision of three comparable results is evaluated by calculating the relative standard deviation (RSD). The acceptance criteria for analyte-specific RSDs are developed during the DQO development process on a site-specific basis. Precision of replicate samples is evaluated by calculating the RSD using the following equation:

$$\%RSD = \frac{\sqrt{\frac{\sum_{i=1}^{i=n} \left| D_i - \overline{D} \right|^2}{n}}}{\overline{D}} \times 100$$

Where:

D_i → =→the individual sample concentrations
$\overline{D}$ → =→the mean of n values
n → =→the total number of values

5.1.2 Accuracy

Accuracy is the degree of agreement between a measured value and the "true" or expected value. It represents an estimate of total error from a single measurement, including both systematic errors or bias, and random errors that may reflect variability due to imprecision. Accuracy is evaluated in terms of percent recoveries determined from results of the MS/MSD, LCS/LCSD, and surrogates where applicable.

Accuracy is expressed in percent recovery (%R) using the following equation:

$$\%R = 100 \times \left[\frac{C_m}{C_{sm}} \right]$$

Where:

C_m → =→measured concentration
C_{sm} →=→actual concentration

5.1.3 Representativeness

Representativeness is the degree to which sample data accurately reflect the characteristics of a population of samples. It is achieved through a well-designed sampling program and by using standardized sampling strategies and techniques and analytical procedures. Factors that can affect representativeness include site homogeneity, sample homogeneity at a single point, and available information around which the sampling program is designed. Using multiple methods to measure an analyte can also result in non-representativeness of sample data.

5.1.4 Completeness

Analytical completeness is a measure of the amount of valid data obtained compared with the amount that was expected to be obtained under correct, normal conditions. Completeness is calculated for the aggregation of data for each analyte measured for any particular sampling event or other defined set of samples (such as by site) as set out in the DQOs. Completeness is calculated and reported for each method, matrix, and analyte combination. The goal for this program is 90 percent for each method, matrix, and analyte combination.

The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the dataset. For completeness requirements, valid results are all results not qualified with an R-flag after a usability assessment has been performed.

Analytical completeness is calculated and reported for each method, matrix, and analyte combination using the following formula:

$$\%\cdot Completeness = 100 \times \left(\frac{V}{n}\right)$$

Where:

V → =→number·of·measurements·judged·valid
n → =→total·number·of·measurements

5.1.5 Comparability

Comparability is the confidence with which one data set can be compared to another. It is achieved by maintaining standard techniques and procedures for collecting and analyzing samples and reporting the analytical results in standard units. Results of performance evaluation samples and systems audits will provide additional information for assessing comparability of data among participating subcontractor laboratories.

5.1.6 Sensitivity

Sensitivity refers to the minimum magnitude at which analytical methods can resolve quantitative differences among sample concentrations. If the minimum magnitude for a particular analytical method is sufficiently below an action level or risk screening criterion, then the method sensitivity is deemed sufficient to fully evaluate the dataset with respect to the desired reference values. Frequently, risk-based screening levels fall below the sensitivity of even the most sensitive analytical methods. In such cases, it is necessary to review the qualifications of several laboratories, both from the standpoint of sensitivity as well as other data quality indicators, to select the best laboratory for the project. Method detection limits (MDLs) and reporting limits (RLs) are discussed in the following section.

5.2 Method Detection Limits, Reporting Limits, and Instrument Calibration Requirements

5.2.1 Method Detection Limits

The MDL is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. Each participating laboratory will determine the MDL for each method, matrix, and analyte for each instrument that will be used to analyze samples. The MDLs will be initially determined before analyzing samples and will be redetermined at least once every 12 months.

1) Estimate the MDL using one of the following:
   a) The mean determined concentration plus three times the standard deviation of a set of method blanks
   b) The concentration value that corresponds to an instrument signal/noise ratio in the range of 3 to 5

c) The concentration equivalent of three times the standard deviation of replicate instrumental measurement of spiked blanks

d) The region of the standard curve where there is a significant change in sensitivity (that is, a break in the slope of the standard curve)

e) Instrumental limitations

f) Previously determined MDL

2) Determine the initial MDL (**Note:** The initial MDL is used when the laboratory does not have adequate data to perform the Ongoing Annual Verification, typically when a new method is implemented or if a method was rarely used in the last 24 months):

a) Select a spiking level, typically 2 to 10 times the estimated MDL in Step 1. Spiking levels in excess of 10 times the estimated detection limit may be required for analytes with very poor recovery (for example, for an analyte with 10 percent recovery, spiked at 100 micrograms per liter [µg/L], with mean recovery of 10 µg/L; the calculated MDL may be around 3 µg/L. Therefore, in this example, the spiking level would be 33 times the MDL, but spiking lower may result in no recovery at all).

b) Process a minimum of seven spiked samples and seven method blank samples through all steps of the method. The samples used for the MDL must be prepared in at least three batches on three separate calendar dates and analyzed on three separate calendar dates. (Preparation and analysis may be on the same day.) Existing data may be used, if compliant with the requirements for at least three batches, and generated within the last 24 months. The most recent available data for method blanks and spiked samples must be used. Statistical outlier removal procedures should not be used to remove data for the initial MDL determination, because the total number of observations is small, and the purpose of the MDL procedure is to capture routine method variability. However, documented instances of gross failures (for example, instrument malfunctions, mislabeled samples, and cracked vials) may be excluded from the calculations, provided that at least seven spiked samples and seven method blanks are available. (The rationale for removal of specific outliers must be documented and maintained on file with the results of the MDL determination.)

i) If multiple instruments will be assigned the same MDL, then the sample analyses must be distributed across all instruments.

ii) A minimum of two spiked samples and two method blank samples prepared and analyzed on different calendar dates is required for each instrument. Each analytical batch may contain one spiked sample and one method blank sample run together. A spiked sample and a method blank sample may be analyzed in the same batch but are not required to be.

iii) The same prepared extract may be analyzed on multiple instruments so long as the minimum requirement of seven preparations in at least three separate batches is maintained.

c) Evaluate the spiking level: If any result for any individual analyte from the spiked samples does not meet the method qualitative identification criteria or does not provide a numerical result greater than zero, repeat the spiked samples at a higher concentration. (Qualitative identification criteria are a set of rules or guidelines for establishing the identification or presence of an analyte using a measurement system. Qualitative identification does not ensure that quantitative results for the analyte can be obtained.)

d) Make all computations as specified in the analytical method and express the final results in the method-specified reporting units:

i) Calculate the sample standard deviation (S) of the replicate spiked sample measurements and the sample standard deviation of the replicate method blank measurements from all instruments to which the MDL will be applied.

ii) Compute the MDLs (the MDL based on spiked samples) as follows:

$$MDL_S = t(n-1, 1-\alpha = 99) S_S$$

Where:

MDL_S → = the method detection limit based on spiked samples

$t(n-1, 1-\alpha = 99)$ → = the Student's *t*-value appropriate for a single-tailed 99th percentile *t* statistic and a standard deviation estimate with n-1 degrees of freedom

S_S → = → sample standard deviation of the replicate spiked sample analyses

iii) Compute the MDL_b (the MDL based on method blanks) as follows:

a. If no method blanks give numerical results for an individual analyte, the MDL_b does not apply. A numerical result includes both positive and negative results, including results below the current MDL, but not results of not detected commonly observed when a peak is not present in chromatographic analysis.

b. If some (but not all) of the method blanks for an individual analyte give numerical results, set the MDL_b equal to the highest method blank result. If more than 100 method blanks are available, set MDL_b to the level that is no less than the 99th percentile of the method blank results. For "n" method blanks where nis greater than or equal to (≥) 100, sort the method blanks in rank order. The (n * 0.99) ranked method blank result (round to the nearest whole number) is the MDL_b. For example, to find MDL_b from a set of 164 method blanks where the highest ranked method blank results are … 1.5, 1.7, 1.9, 5.0, and 10, then 164 × 0.99 = 162.36, which rounds to the 162nd method blank result. Therefore, MDL_b is 1.9 for n =164 (10 is the 164th result, 5.0 is the 163rd result, and 1.9 is the 162nd result). Alternatively, spreadsheet algorithms can be used to calculate the 99th percentile to interpolate between the ranks more precisely.

c. If all method blanks for an individual analyte give numerical results, then calculate the MDL_b as:

$$MDL_b = X + t(n-1, 1-\alpha = 99) S_b$$

Where:

MDL_b → =the MDL based on method blanks

X → =mean of the method blank results (use zero in place of the mean if the mean is negative)

$t(n-1, 1-\alpha = 99)$ =the Student's t-value appropriate for the single-tailed 99th percentile t statistic and a standard deviation estimate with n-1 degrees of freedom.

S_b → =sample standard deviation of the replicate method blank sample analyses

Note: If 100 or more method blanks are available, as an option, MDL_b may be set to the concentration that is greater than or equal to the 99th percentile of the method blank results, as described in Section (2)(d)(iii)(B).

e) Select the greater of MDLs or MDL_b as the initial MDL.

5.2.2 Reporting Limits

Table C-5-1 contains a list of the analytical laboratories that will be used and the associated analytical and preparatory methods to be performed. To allow for matrix interferences and variability of instrument controls, the RLs will typically be three to eight greater than two times the laboratory calculated MDL. RLs used by the laboratory should not be greater than the detection limit objectives listed in Tables C-5-2 through C-5-10.

When calibrating instruments, a standard at a concentration equal to or less than the RL must be included. Reporting requirements are as follows:

- Analytes at concentrations greater than the laboratory's MDL but less than the RL will be flagged as estimated with a "J" qualifier and reported. Analytes that are not detected at or above the laboratory's MDL will be reported as not detected at the RL and flagged "U."
- RLs and sample results will be reported to two significant figures if less than 10 and to 3 significant figures if 10 or greater. RLs will be reported on a dry-weight basis for soil and sediment samples. All QC will be reported to three significant figures.

Table C-5-1. Analytical and Preparatory Methods

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analytical Method	Parameter	Preparatory Methods	Analytical Laboratory
SW6020B	Trace metals by ICP-MS	SW3005A, SW3010A, SW3015, SW3050B, SW3051, SW3052	ALS-Kelso
SW7470A/SW7471B	Mercury by cold vapor atomic adsorption	See analytical method	ALS-Kelso
NWTPH-Dx	TPH extractable	See analytical method	ALS-Kelso
E1668	PCB Congeners	See analytical method	ALS-Burlington
SW8270D-SIM	PAHs	SW3510C, SW3520C, SW3535A, SW3540C, SW3541, SW3546	ALS-Kelso
E1613B	Dioxins and Furans	See analytical method	ALS-Burlington
SW8270D-LL	Low-level bis(2-ethylhexyl)phthalate	SW3510C, SW3520C, SW3535A, SW3540C, SW3541, SW3546	ALS-Kelso
E1699m	Low-Level Pesticides (DDx only)	See analytical method	ALS-Kelso
SW8082-LL	PCB Aroclors	SW3510C, SW3520C, SW3665A SW3550B, SW3665A	ALS-Kelso
E1699	Low-Level Pesticides	See analytical method	ALS-Burlington

Note:

ICP-AES = inductively coupled plasma-atomic emission spectrometry

Table C-5-2. Reporting Limit Objectives for Metals by Method SW6020B/SW7470A/SW7471B

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	MDLs -Reporting Limits - Water (µg/L)	MDLs -Soil/ Sediment (µg/kg)	Reporting Limits - Soil/ Sediment (µg/kg)	CUL Soil/ Sediment (µg/kg)
Arsenic	0.09 / 0.5	60	500	3,000
Cadmium	0.008 / 0.02	7	20	510
Copper	0.05 / 0.1	40	100	359,000
Lead	0.006 / 0.02	20	50	196,000
Mercury (SW7470A/SW7471B)	0.02 / 0.02	2	20	85

Table C-5-2. Reporting Limit Objectives for Metals by Method SW6020B/SW7470A/SW7471B

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	MDLs -Reporting Limits - Water (µg/L)	MDLs -Soil/ Sediment (µg/kg)	Reporting Limits - Soil/ Sediment (µg/kg)	CUL Soil/ Sediment (µg/kg)
Zinc	0.5 / 2	200	500	459,000

Table C-5-3. Reporting Limit Objectives for Total Petroleum Hydrocarbons by Method NWTPH-Dx

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

TPH	MDLs - Reporting Limits - Water (µg/L)	MDLs - Soil/ Sediment (µg/kg)	Reporting Limits -Soil/ Sediment (µg/kg)	CUL Soil/ Sediment (µg/kg)
TPH-Diesel (C10-C24)	11 / 250	790	25,000	91,000
TPH-Motor oil (C24-C35)	29 / 500	2,900	100,000	NA

Note:

µg/kg = microgram(s) per kilogram

Table C-5-4. Reporting Limit Objectives for Low-Level Pesticides by Method E1699

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	MDLs/Reporting Limits - Water (µg/L)	MDLs - Soil/Sediment (µg/kg)	Reporting Limits – Soil/Sediment (µg/kg)	CUL Soil/Sediment (µg/kg)
2,4'-DDD	0.070 / 0.04	0.032	0.04	6.1*
2,4'-DDE	0.032 / 0.04	0.070	0.04	6.1*
2,4'-DDT	0.085 / 0.04	0.085	0.04	6.1*
4,4'-DDD	0.029 / 0.04	0.015	0.04	6.1*
4,4'-DDE	0.015 / 0.04	0.029	0.04	6.1*
4,4'-DDT	0.020 / 0.04	0.020	0.04	6.1*
Aldrin	0.024 / 0.04	0.024	0.04	2
alpha-Chlordane	0.012 / 0.04	0.012	0.04	1.4**
cis-Nonachlor	0.013 / 0.04	0.013	0.04	1.4**
Dieldrin	0.018 / 0.04	0.018	0.04	0.07
gamma-BHC (Lindane)	0.017 / 0.04	0.017	0.04	5
gamma-Chlordane	0.021 / 0.04	0.021	0.04	1.4**
Oxychlordane	0.013 / 0.04	0.013	0.04	1.4**
trans-Nonachlor	0.024 / 0.04	0.024	0.04	1.4**

* CUL is for total DDx (sum of six isomers associated with DDT, DDD, and DDE)

** CUL is for sum of sum of cis-chlordane, trans-chlordane, oxychlordane, cis-nonachlor, and trans-nonachlor

Notes:

DDD = dichlorodiphenyldichloroethane

DDE = dichlorodiphenyldichloroethane

DDT = dichlorodiphenyltrichloroethane

Table C-5-5. Reporting Limit Objectives for Low-Level Pesticides by Method E1699m

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	MDLs/Reporting Limits - Water (µg/L)	MDLs - Soil/Sediment (µg/kg)	Reporting Limits – Soil/Sediment (µg/kg)	CUL Soil/Sediment (µg/kg)
2,4'-DDD	0.00011 / 0.0005	0.063	0.1	6.1*
2,4'-DDE	0.00011 / 0.0005	0.079	0.1	6.1*
2,4'-DDT	0.000087 / 0.0005	0.094	0.1	6.1*
4,4'-DDD	0.0001 / 0.0005	0.035	0.1	6.1*
4,4'-DDE	0.000075 / 0.0005	0.07	0.1	6.1*
4,4'-DDT	0.000088 / 0.0005	0.047	0.1	6.1*

* CUL is for total DDx (sum of six isomers associated with DDT, DDD, and DDE)

Notes:

DDD = dichlorodiphenyldichloroethane

DDE = dichlorodiphenyldichloroethane

DDT = dichlorodiphenyltrichloroethane

Table C-5-6. Reporting Limit Objectives for PAHs by Method SW8270D-SIM

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	MDLs/Reporting Limits - Water (µg/L)	MDLs - Soil/Sediment (µg/kg)	Reporting Limits – Soil/Sediment (µg/kg)	CUL Soil/Sediment (µg/kg)
1-Methylnaphthalene	0.0035 / 0.02	0.51	5	85*
2-Methylnaphthalene	0.0023 / 0.02	0.39	5	85*
Acenaphthene	0.0044 / 0.02	0.76	5	85*
Acenaphthylene	0.0034 / 0.02	0.59	5	85*
Anthracene	0.0036 / 0.02	0.58	5	85*
Benzo(a)anthracene	0.0026 / 0.02	0.72	5	85*
Benzo(a)pyrene	0.0043 / 0.02	0.76	5	85*
Benzo(b)fluoranthene	0.0041 / 0.02	0.92	5	85*
Benzo(g,h,i)perylene	0.0029 / 0.02	0.85	5	85*
Benzo(k)fluoranthene	0.003 / 0.02	0.87	5	85*
Chrysene	0.0034 / 0.02	0.8	5	85*
Dibenz(a,h)anthracene	0.0025 / 0.02	0.8	5	85*
Fluoranthene	0.01 / 0.02	0.98	5	85*

Table C-5-6. Reporting Limit Objectives for PAHs by Method SW8270D-SIM

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	MDLs/Reporting Limits - Water (µg/L)	MDLs - Soil/Sediment (µg/kg)	Reporting Limits – Soil/Sediment (µg/kg)	CUL Soil/Sediment (µg/kg)
Fluorene	0.0038 / 0.02	0.61	5	85*
Indeno(1,2,3-c,d)pyrene	0.0026 / 0.02	0.87	5	85*
Naphthalene	0.0038 / 0.02	0.6	5	85*
Phenanthrene	0.005 / 0.02	1.4	5	85*
Pyrene	0.0053 / 0.02	0.76	5	85*

* CUL is for carcinogenic PAHs as benzo(a)pyrene equivalents

Table C-5-7. Reporting Limit Objective for Low-Level bis(2-ethylhexyl)phthalate by SW8270D-LL

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	MDLs/Reporting Limits - Water (µg/L)	MDLs - Soil/Sediment (µg/kg)	Reporting Limits – Soil/Sediment (µg/kg)	CUL Soil/Sediment (µg/kg)
bis(2-Ethylhexyl)phthalate	0.13 / 1	8.9	100	135

Table C-5-8. Reporting Limit Objectives for Dioxins/Furans by Method E1613B

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	MDLs/Reporting Limits - Water (µg/L)	MDLs - Soil/Sediment (µg/kg)	Reporting Limits – Soil/Sediment (µg/kg)	CUL Soil/Sediment (µg/kg)
2,3,7,8-TCDF	0.00014 / 0.002	0.00002	0.0002	0.00040658
2,3,7,8-TCDD	0.00012 / 0.002	0.00004	0.0002	0.0002
1,2,3,7,8-PeCDF	0.00043 / 0.010	0.00005	0.001	NA
2,3,4,7,8-PeCDF	0.00035 / 0.010	0.00005	0.001	0.0003
1,2,3,7,8-PeCDD	0.00059 / 0.010	0.00004	0.001	0.0002
1,2,3,4,7,8-HxCDF	0.00032 / 0.010	0.00004	0.001	0.0004
1,2,3,6,7,8-HxCDF	0.00042 / 0.010	0.00003	0.001	NA
2,3,4,6,7,8-HxCDF	0.00040 / 0.010	0.00004	0.001	NA
1,2,3,7,8,9-HxCDF	0.00037 / 0.010	0.00004	0.001	NA
1,2,3,4,7,8-HxCDD	0.00037 / 0.010	0.00004	0.001	NA

Table C-5-8. Reporting Limit Objectives for Dioxins/Furans by Method E1613B

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	MDLs/Reporting Limits - Water (µg/L)	MDLs - Soil/Sediment (µg/kg)	Reporting Limits – Soil/Sediment (µg/kg)	CUL Soil/Sediment (µg/kg)
1,2,3,6,7,8-HxCDD	0.00062 / 0.010	0.00006	0.001	NA
1,2,3,7,8,9-HxCDD	0.00062 / 0.010	0.00007	0.001	NA
1,2,3,4,6,7,8-HpCDF	0.00048 / 0.010	0.00005	0.001	NA
1,2,3,4,7,8,9-HpCDF	0.00027 / 0.010	0.00003	0.001	NA
1,2,3,4,6,7,8-HpCDD	0.00048 / 0.010	0.00007	0.001	NA
OCDF	0.00075 / 0.020	0.00008	0.005	NA
OCDD	0.00098 / 0.020	0.00011	0.005	NA
Total 2,3,7,8-TCDD Equivalents	NA	NA	NA	NA

- Data may be reported to the MDL where the RL is greater than the CUL

Table C-5-9. Reporting Limit Objectives for PCB Congeners by Method E1668

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	Target MDLs/Reporting Limits - Water (µg/L)	Target MDLs - Soil/Sediment (µg/kg)	Target Reporting Limits – Soil/Sediment (µg/kg)	CUL Soil/Sediment (µg/kg)
PCB-1	0.000002 / 0.000025	0.00006	0.00125	9*
PCB-2	0.000002 / 0.000025	0.00014	0.00125	9*
PCB-3	0.0000022 / 0.000025	0.00011	0.00125	9*
PCB-4	0.0000091 / 0.000025	0.00007	0.00125	9*
PCB-10	0.000007 / 0.000025	0.00005	0.00125	9*
PCB-9	0.0000075 / 0.000025	0.00008	0.00125	9*
PCB-7	0.000025 / 0.000025	0.00009	0.00125	9*
PCB-6	0.0000101 / 0.000025	0.00012	0.00125	9*
PCB-5	0.0000078 / 0.000025	0.00005	0.00125	9*
PCB-8	0.0000053 / 0.000025	0.00026	0.00125	9*
PCB-14	0.0000059 / 0.000025	0.00009	0.00125	9*
PCB-11	0.0000035 / 0.000025	0.00387	0.00125	9*
PCB-(13/12)	0.000004 / 0.000025	0.00010	0.00125	9*
PCB-15	0.000007 / 0.000025	0.00020	0.00125	9*

Table C-5-9. Reporting Limit Objectives for PCB Congeners by Method E1668

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	Target MDLs/Reporting Limits - Water (µg/L)	Target MDLs - Soil/Sediment (µg/kg)	Target Reporting Limits – Soil/Sediment (µg/kg)	CUL Soil/Sediment (µg/kg)
PCB-19	0.0000021 / 0.000025	0.00005	0.00125	9*
PCB-(30/18)	0.000005 / 0.000025	0.00022	0.00125	9*
PCB-17	0.0000027 / 0.000025	0.00016	0.00125	9*
PCB-27	0.0000021 / 0.000025	0.00005	0.00125	9*
PCB-24	0.0000017 / 0.000025	0.00007	0.00125	9*
PCB-16	0.000002 / 0.000025	0.00016	0.00125	9*
PCB-32	0.0000014 / 0.000025	0.00012	0.00125	9*
PCB-34	0.0000019 / 0.000025	0.00005	0.00125	9*
PCB-23	0.0000022 / 0.000025	0.00005	0.00125	9*
PCB-(26/29)	0.0000023 / 0.000025	0.00020	0.00125	9*
PCB-25	0.0000034 / 0.000025	0.00005	0.00125	9*
PCB-31	0.0000033 / 0.000025	0.00022	0.00125	9*
PCB-(28/20)	0.0000019 / 0.000025	0.00016	0.00125	9*
PCB-(21/33)	0.0000024 / 0.000025	0.00005	0.00125	9*
PCB-22	0.0000014 / 0.000025	0.00007	0.00125	9*
PCB-36	0.0000019 / 0.000025	0.00016	0.00125	9*
PCB-39	0.0000018 / 0.000025	0.00012	0.00125	9*
PCB-38	0.0000013 / 0.000025	0.00005	0.00125	9*
PCB-35	0.0000018 / 0.000025	0.00005	0.00125	9*
PCB-37	0.0000016 / 0.000025	0.00022	0.00125	9*
PCB-54	0.000002 / 0.000025	0.00006	0.00125	9*
PCB-(50/53)	0.0000036 / 0.000025	0.00022	0.00125	9*
PCB-(45/51)	0.0000052 / 0.000025	0.00013	0.00125	9*
PCB-46	0.0000028 / 0.000025	0.00010	0.00125	9*
PCB-52	0.0000022 / 0.000025	0.00053	0.00125	9*
PCB-(73/43)	0.0000039 / 0.000025	0.00010	0.00125	9*
PCB-(69/49)	0.0000027 / 0.000025	0.00019	0.00125	9*
PCB-48	0.0000052 / 0.000025	0.00029	0.00125	9*

Table C-5-9. Reporting Limit Objectives for PCB Congeners by Method E1668

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	Target MDLs/Reporting Limits - Water (µg/L)	Target MDLs - Soil/Sediment (µg/kg)	Target Reporting Limits – Soil/Sediment (µg/kg)	CUL Soil/Sediment (µg/kg)
PCB-(44/47/65)	0.0000029 / 0.000025	0.00012	0.00125	9*
PCB-(59/62/75)	0.0000041 / 0.000025	0.00097	0.00125	9*
PCB-42	0.0000045 / 0.000025	0.00024	0.00125	9*
PCB-(41/40/71)	0.0000016 / 0.000025	0.00014	0.00125	9*
PCB-64	0.0000048 / 0.000025	0.00024	0.00125	9*
PCB-72	0.0000026 / 0.000025	0.00026	0.00125	9*
PCB-68	0.0000018 / 0.000025	0.00007	0.00125	9*
PCB-57	0.0000035 / 0.000025	0.00016	0.00125	9*
PCB-58	0.0000038 / 0.000025	0.00009	0.00125	9*
PCB-67	0.0000036 / 0.000025	0.00012	0.00125	9*
PCB-63	0.0000018 / 0.000025	0.00012	0.00125	9*
PCB-(61/70/74/76)	0.0000009 / 0.000025	0.00008	0.00125	9*
PCB-66	0.0000055 / 0.000025	0.00069	0.00125	9*
PCB-55	0.000003 / 0.000025	0.00041	0.00125	9*
PCB-56	0.000003 / 0.000025	0.00014	0.00125	9*
PCB-60	0.0000012 / 0.000025	0.00010	0.00125	9*
PCB-80	0.0000024 / 0.000025	0.00012	0.00125	9*
PCB-79	0.0000032 / 0.000025	0.00011	0.00125	9*
PCB-78	0.0000016 / 0.000025	0.00011	0.00125	9*
PCB-81	0.0000014 / 0.000025	0.00011	0.00125	9*
PCB-77	0.000002 / 0.000025	0.00022	0.00125	9*
PCB-104	0.0000036 / 0.000025	0.00012	0.00125	9*
PCB-96	0.0000018 / 0.000025	0.00009	0.00125	9*
PCB-103	0.0000023 / 0.000025	0.00010	0.00125	9*
PCB-94	0.0000032 / 0.000025	0.00009	0.00125	9*
PCB-95	0.0000039 / 0.000025	0.00012	0.00125	9*
PCB-(100/93/102/98)	0.0000027 / 0.000025	0.00037	0.00125	9*
PCB-(88/91)	0.0000058 / 0.000025	0.00032	0.00125	9*

Table C-5-9. Reporting Limit Objectives for PCB Congeners by Method E1668

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	Target MDLs/Reporting Limits - Water (µg/L)	Target MDLs - Soil/Sediment (µg/kg)	Target Reporting Limits – Soil/Sediment (µg/kg)	CUL Soil/Sediment (µg/kg)
PCB-84	0.000007 / 0.000025	0.00048	0.00125	9*
PCB-89	0.0000025 / 0.000025	0.00010	0.00125	9*
PCB-121	0.0000043 / 0.000025	0.00013	0.00125	9*
PCB-92	0.0000026 / 0.000025	0.00012	0.00125	9*
PCB-(113/90/101)	0.0000026 / 0.000025	0.00015	0.00125	9*
PCB-83	0.0000043 / 0.000025	0.00064	0.00125	9*
PCB-99	0.0000052 / 0.000025	0.00051	0.00125	9*
PCB-112	0.0000032 / 0.000025	0.00009	0.00125	9*
PCB-(108/119/86/97/125/87)	0.0000065 / 0.000025	0.00053	0.00125	9*
PCB-(117/116/85)	0.0000065 / 0.000025	0.00149	0.00125	9*
PCB-(110/115)	0.0000041 / 0.000025	0.00008	0.00125	9*
PCB-82	0.0000038 / 0.000025	0.00007	0.00125	9*
PCB-111	0.0000025 / 0.000025	0.00012	0.00125	9*
PCB-120	0.0000036 / 0.000025	0.00019	0.00125	9*
PCB-(107/124)	0.0000047 / 0.000025	0.00012	0.00125	9*
PCB-109	0.0000038 / 0.000025	0.00011	0.00125	9*
PCB-123	0.0000038 / 0.000025	0.00008	0.00125	9*
PCB-106	0.0000019 / 0.000025	0.00101	0.00125	9*
PCB-118	0.0000017 / 0.000025	0.00012	0.00125	9*
PCB-122	0.0000022 / 0.000025	0.00008	0.00125	9*
PCB-114	0.0000028 / 0.000025	0.00039	0.00125	9*
PCB-105	0.000002 / 0.000025	0.00014	0.00125	9*
PCB-127	0.0000015 / 0.000025	0.00007	0.00125	9*
PCB-126	0.0000019 / 0.000025	0.00004	0.00125	9*
PCB-155	0.0000029 / 0.000025	0.00014	0.00125	9*
PCB-152	0.0000026 / 0.000025	0.00008	0.00125	9*
PCB-150	0.0000028 / 0.000025	0.00014	0.00125	9*

Table C-5-9. Reporting Limit Objectives for PCB Congeners by Method E1668

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	Target MDLs/Reporting Limits - Water (µg/L)	Target MDLs - Soil/Sediment (µg/kg)	Target Reporting Limits – Soil/Sediment (µg/kg)	CUL Soil/Sediment (µg/kg)
PCB-136	0.0000045 / 0.000025	0.00007	0.00125	9*
PCB-145	0.0000017 / 0.000025	0.00005	0.00125	9*
PCB-148	0.000004 / 0.000025	0.00016	0.00125	9*
PCB-(151/135)	0.0000019 / 0.000025	0.00008	0.00125	9*
PCB-154	0.0000019 / 0.000025	0.00005	0.00125	9*
PCB-144	0.0000052 / 0.000025	0.00149	0.00125	9*
PCB-(147/149)	0.000005 / 0.000025	0.00013	0.00125	9*
PCB-(134/143)	0.0000015 / 0.000025	0.00016	0.00125	9*
PCB-(139/140)	0.0000033 / 0.000025	0.00017	0.00125	9*
PCB-131	0.000004 / 0.000025	0.00005	0.00125	9*
PCB-142	0.0000025 / 0.000025	0.00026	0.00125	9*
PCB-132	0.0000031 / 0.000025	0.00007	0.00125	9*
PCB-133	0.0000034 / 0.000025	0.00011	0.00125	9*
PCB-165	0.000002 / 0.000025	0.00018	0.00125	9*
PCB-146	0.0000028 / 0.000025	0.00009	0.00125	9*
PCB-161	0.0000051 / 0.000025	0.00066	0.00125	9*
PCB-(153/168)	0.0000039 / 0.000025	0.00010	0.00125	9*
PCB-141	0.0000032 / 0.000025	0.00005	0.00125	9*
PCB-130	0.0000044 / 0.000025	0.00007	0.00125	9*
PCB-137	0.0000041 / 0.000025	0.00087	0.00125	9*
PCB-164	0.000005 / 0.000025	0.00006	0.00125	9*
PCB-(138/163/129)	0.000003 / 0.000025	0.00025	0.00125	9*
PCB-160	0.0000033 / 0.000025	0.00020	0.00125	9*
PCB-158	0.0000017 / 0.000025	0.00011	0.00125	9*
PCB-(128/166)	0.0000024 / 0.000025	0.00003	0.00125	9*
PCB-159	0.0000029 / 0.000025	0.00006	0.00125	9*
PCB-162	0.0000025 / 0.000025	0.00006	0.00125	9*
PCB-167	0.0000022 / 0.000025	0.00012	0.00125	9*

Table C-5-9. Reporting Limit Objectives for PCB Congeners by Method E1668

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	Target MDLs/Reporting Limits - Water (µg/L)	Target MDLs - Soil/Sediment (µg/kg)	Target Reporting Limits – Soil/Sediment (µg/kg)	CUL Soil/Sediment (µg/kg)
PCB-(156/157)	0.0000028 / 0.000025	0.00002	0.00125	9*
PCB-169	0.0000027 / 0.000025	0.00016	0.00125	9*
PCB-188	0.0000028 / 0.000025	0.00025	0.00125	9*
PCB-179	0.0000023 / 0.000025	0.00020	0.00125	9*
PCB-184	0.0000018 / 0.000025	0.00007	0.00125	9*
PCB-176	0.000003 / 0.000025	0.00016	0.00125	9*
PCB-178	0.0000054 / 0.000025	0.00023	0.00125	9*
PCB-175	0.0000032 / 0.000025	0.00014	0.00125	9*
PCB-187	0.0000023 / 0.000025	0.00019	0.00125	9*
PCB-182	0.0000036 / 0.000025	0.00013	0.00125	9*
PCB-186	0.0000053 / 0.000025	0.00016	0.00125	9*
PCB-(183/185)	0.0000028 / 0.000025	0.00024	0.00125	9*
PCB-174	0.0000022 / 0.000025	0.00017	0.00125	9*
PCB-177	0.0000038 / 0.000025	0.00023	0.00125	9*
PCB-181	0.0000044 / 0.000025	0.00033	0.00125	9*
PCB-(171/173)	0.0000041 / 0.000025	0.00018	0.00125	9*
PCB-172	0.0000039 / 0.000025	0.00012	0.00125	9*
PCB-192	0.0000052 / 0.000025	0.00041	0.00125	9*
PCB-(180/193)	0.0000031 / 0.000025	0.00020	0.00125	9*
PCB-191	0.0000028 / 0.000025	0.00013	0.00125	9*
PCB-170	0.0000033 / 0.000025	0.00024	0.00125	9*
PCB-190	0.0000034 / 0.000025	0.00014	0.00125	9*
PCB-189	0.0000028 / 0.000025	0.00007	0.00125	9*
PCB-202	0.0000037 / 0.000025	0.00020	0.00125	9*
PCB-201	0.0000052 / 0.000025	0.00007	0.00125	9*
PCB-204	0.0000019 / 0.000025	0.00009	0.00125	9*
PCB-(197/200)	0.0000025 / 0.000025	0.00010	0.00125	9*
PCB-(198/199)	0.0000129 / 0.000025	0.00027	0.00125	9*

Table C-5-9. Reporting Limit Objectives for PCB Congeners by Method E1668

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	Target MDLs/Reporting Limits - Water (µg/L)	Target MDLs - Soil/Sediment (µg/kg)	Target Reporting Limits – Soil/Sediment (µg/kg)	CUL Soil/Sediment (µg/kg)
PCB-196	0.0000052 / 0.000025	0.00009	0.00125	9*
PCB-203	0.0000047 / 0.000025	0.00008	0.00125	9*
PCB-195	0.0000031 / 0.000025	0.00050	0.00125	9*
PCB-194	0.0000034 / 0.000025	0.00013	0.00125	9*
PCB-205	0.0000032 / 0.000025	0.00014	0.00125	9*
PCB-208	0.0000057 / 0.000025	0.00016	0.00125	9*
PCB-207	0.0000047 / 0.000025	0.00016	0.00125	9*
PCB-206	0.0000026 / 0.000025	0.00014	0.00125	9*
PCB-209	0.0000027 / 0.000025	0.00015	0.00125	9*

*CUL is for total congeners

Table C-5-10 Reporting Limit Objectives for PCB Aroclors by Method SW8082-LL

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	MDLs/Reporting Limits - Water (µg/L)	MDLs -Soil/Sediment (µg/kg)	Reporting Limits – Soil/Sediment (µg/kg)	CUL Soil/Sediment (µg/kg)
Aroclor-1016	0.0028 / 0.005	0.5	2	9*
Aroclor-1221	0.0028 / 0.01	0.5	2	9*
Aroclor-1232	0.0028 / 0.005	0.5	2	9*
Aroclor-1242	0.0028 / 0.005	0.5	2	9*
Aroclor-1248	0.0028 / 0.005	0.5	2	9*
Aroclor-1254	0.0028 / 0.005	0.5	2	9*
Aroclor-1260	0.0028 / 0.005	0.5	2	9*
Aroclor-1262	0.0028 / 0.005	0.5	2	9*

* CUL is for total PCBs

5.2.3 Instrument Calibration

Laboratory instruments will be calibrated by qualified personnel before sample analysis according to the procedures specified in each method. Calibration will be verified at method-specified intervals throughout the analysis sequence. The frequency and acceptance criteria for calibration are specified for each analytical method with supplemental requirements defined below for organic methodologies. When multi-point calibration is specified, the concentrations of the calibration standards should bracket those expected in the samples. Samples will be diluted, if necessary, to bring analyte responses to within the calibration range. Data that exceed the calibration range will not be reported by the laboratory. The initial calibration (ICAL) curve will be verified as accurate with a standard purchased or prepared from an independent second source. The initial calibration verification (ICV) involves the analysis of a standard containing all the target analytes, typically in the middle of the calibration range, each time the ICAL is performed. Quantitation based on extrapolation is not allowed.

5.2.3.1 Initial Calibration Models

Organic and inorganic methodologies often provide multiple options for ICAL curve fits and associated acceptance criteria for use. The following subsections outline required "good laboratory practices" that will be employed by the laboratory. The hierarchy that the laboratory will use when selecting the calibration curve fit for use in quantitation of sample results is outlined below.

Calibration Techniques

- Verify that correct instrument operating conditions and routine maintenance as specified in the method and laboratory SOPs, are employed. All maintenance activities will be documented in a laboratory notebook for troubleshooting and scheduling of future routine, periodic maintenance.
- Ensure that the instrument is free of contamination prior to calibration. Do NOT perform any blank subtraction.
- Perform the entire ICAL before sample analyses. The calibration standards must be analyzed in a sequential order from the lowest to highest concentration. If **one** calibration standard fails to meet criteria, it may be reanalyzed at the end of the calibration sequence. Justification for removing a calibration point from the curve fit selected includes such items as improper purge, injection failure, non-spiked level, or other obvious failures. The failure of multiple standards suggests an instrument problem or operator error and corrective action is required.
- Only the lowest calibration point or the highest calibration point can be removed from the calibration curve without justification. If the lowest standard is removed, the RL for that compound increases to the level of the next lowest calibration standard. Approval to elevate RLs greater than project-specific objectives MUST be approved by the Project Chemist. If the highest standard is removed, the linear range is shortened for that compound.
- The lowest standard in the calibration curve must be at or below the required RL.
- The other standard concentrations must define the working range of the instrument or the expected range of concentrations found in the samples.
- Either external or internal calibration can be employed for methods not involving mass spectrometry detectors. Internal calibration must be used when a mass spectrometry detector is employed.
- A minimum of five calibration points must be used for the calibration curve for gas chromatography (GC)/mass spectrometry and high-pressure liquid chromatography methods.
- Most compounds tend to be linear and a linear approach will be favored when linearity is suggested by the calibration data. Non-linear calibration will be considered only when a linear approach cannot be applied. Prior to using a non-linear calibration approach, the Project Chemist must be notified and

provide approval. It is not acceptable to use an alternate calibration procedure when a compound fails to perform in the usual manner. When this occurs, it is indicative of instrument problem or operator error.

- If a non-linear calibration curve fit is employed, a minimum of six calibration levels must be used for second-order (quadratic) curves, and a third-order polynomial requires a minimum of seven calibration levels.
- When more than five levels of standards are analyzed in anticipation of using second- or third-order calibration curves, all calibration points MUST be used regardless of the calibration option employed. The highest or lowest calibration point may be excluded to narrow the calibration range and meet the requirements for a specific calibration option. Otherwise, unjustified exclusion of calibration data is expressly forbidden.

Calibration Options

The acceptable calibration options and the hierarchy that the laboratory should use when selecting a specific option are outlined below; the choice of calibration option may also be based on previous experience or a prior knowledge of detector response:

- **Linear calibration using average calibration or response factors (RFs)**. Calibration factors for external calibrations or RFs for internal calibrations must have a RSD not exceeding 20 percent or 15 percent, respectively, to be used for quantitation. (For dioxins and furans by GC/mass spectrometry, the maximum RSDs are 20 percent for unlabeled standards and 30 percent for labeled standards.) A minimum RFs of 0.05 for most target analytes and 0.01 for the least responsive target analytes must be achieved to ensure detectability.
- **Linear calibration using a linear regression equation (y = mx + b)**. The correlation coefficient (r) must equal 0.995 or better; coefficient of determination (r^2) must equal 0.990 or better. The line should NOT be forced through the origin. The equation and a plot of the linear regression must be included in the raw data generated by the laboratory and made available in the data package upon the client's request.
- **A non-linear calibration**. This model may be a second-order or third-order polynomial. The model must be continuous without a break in the function and should NOT be forced through the origin. The coefficient of determination of the non-linear regression must be 0.99 or better. The equation and a plot of the non-linear regression must be included in the raw data generated by the laboratory and made available in the data package upon the client's request.

5.2.3.2 Continuing Calibration

Periodic verification of the ICAL is essential in generating analytical data of known quality. The continuing calibration verification (CCV) analyses ensure that the instrument has not been adversely affected by the sample matrix or other instrument failures that would increase or decrease the sensitivity or accuracy of the method. The laboratory will perform continuing calibration for all methods according to the specific requirements in the method and laboratory SOPs.

5.3 Elements of Quality Control

Laboratory QC checks indicate the state of control that prevailed at the time of sample analysis. QC checks that involve field samples, such as matrix, surrogate spikes, and field duplicates, also indicate the presence of matrix effects. Field-originated blanks provide a way to monitor for potential contamination to which field samples are subjected. This QAPP specifies requirements for method blanks, LCS/LCSDs, surrogate spikes, and MS/MSDs that must be followed by laboratories participating in the data collection effort.

A laboratory QC batch is defined as a method blank, LCS/LCSD, MS/MSD, or a sample duplicate, depending on the method and 20 or fewer environmental samples of similar matrix that are extracted or analyzed together. For GC/mass spectrometry volatile analyses, a method blank, LCS/LCSD, and MS/MSD must be analyzed in each 12-hour tune period. The number of environmental samples allowed in the laboratory QC batch is defined by the remaining time in the method-prescribed 12-hour tune period divided by the analytical run time. Each preparation or analytical batch will be identified in such a way as to be able to associate environmental samples with the appropriate laboratory QC samples.

5.3.1 Quality Control Analyses/Parameters Originated by Laboratory

5.3.1.1 Method Blank

Blanks are used to monitor each preparation or analytical batch for interference and/or contamination from glassware, reagents, and other potential sources within the laboratory. A method blank is an analyte-free matrix (laboratory reagent water for aqueous samples or Ottawa sand, sodium sulfate, or glass beads [metals] for solid samples) to which all reagents are added in the same amount or proportions as are added to the samples. It is processed through the entire sample preparation and analytical procedures along with the samples in the batch. There will be at least one method blank per preparation or analytical batch. If a target analyte is found at a concentration that exceeds the RL, corrective action must be performed to identify and eliminate the contamination source. All associated samples must be re-prepared and reanalyzed after the contamination source has been eliminated. No analytical data may be corrected for the concentration found in the blank.

5.3.1.2 Laboratory Control Sample

The LCS will consist of an analyte-free matrix (laboratory reagent water for aqueous samples or Ottawa sand sodium sulfate, or glass beads [metals] for solid samples) spiked with known amounts of analytes that come from a source different than that used for calibration standards. Target analytes specified in the QAPP will be spiked into the LCS. The spike levels will be less than or equal to the midpoint of the calibration range. If LCS results are outside the specified control limits, corrective action must be taken, including sample re-preparation and reanalysis, if appropriate. If more than one LCS is analyzed in a preparation or analytical batch, the results of all LCSs must be reported. Any LCS recovery outside QC limits affects the accuracy for the entire batch and requires corrective action.

5.3.1.3 Surrogates

Surrogates are organic analytes that behave similarly to the analytes of interest but are not expected to occur naturally in the samples. They are spiked into the standards, samples, and QC samples prior to sample preparation. Recoveries of surrogates are used to indicate accuracy, method performance, and extraction efficiency. If surrogate recoveries are outside the specified control limits, corrective action must be taken, including sample re-preparation and reanalysis, if appropriate.

5.3.1.4 Matrix Spike/Matrix Spike Duplicate

MS/MSD are a duplicate pair of samples—collected along with an investigatory sample to which the laboratory adds a spike containing the analytes of concern at known concentrations. It is subjected to the same preparation and analytical procedures as the native sample. All target analytes specified in the QAPP are spiked into the sample. MS recoveries are used to evaluate the effect of the sample matrix on the recovery of the analytes of interest. An MSD is a second fortified sample matrix. The RPD between the results of the duplicate MS measures the precision of sample results. Only project-specific samples designated on the chain-of-custody form will be spiked. The spike levels will be less than or equal to the midpoint of the calibration range.

The additional volume for MS/MSDs collected by the field team will be collected at a frequency of 1 for every 20 field samples of each matrix collected from each site. The sample will be collected in triplicate volume for each analysis required and designated on the chain-of-custody form as an MS/MSD. The MS/MSD samples will be collected simultaneously or in immediate succession, using identical recovery techniques as the parent sample and treated in an identical manner during storage, transportation, and analysis. The sampling locations for the MS/MSD will be documented in the field logbook.

5.3.1.5 Internal Standards

Some methods require the use of internal standards to compensate for losses during injection or purging or losses due to viscosity. Internal standards are compounds with similar properties as the analytes of interest but are not expected to occur naturally in the samples. A measured amount of the internal standard is added to the standards, samples, and QC samples following preparation. When the internal standard results are outside the control limits, corrective action must be taken, including sample reanalysis, if appropriate.

5.3.1.6 Laboratory Sample Duplicate

A sample duplicate selected by the laboratory is called a laboratory sample duplicate. It is subjected to the same preparation and analytical procedures as the native sample. The RPD between the results of the native sample and laboratory sample duplicate measures the precision of sample results. The data collected may also yield information regarding whether the sample matrix is heterogeneous.

5.3.1.7 Interference Check Samples

The interference check samples are used in ICP analyses to verify background and inter-element correction factors. They consist of two solutions: A and AB. Solution A contains the interfering analytes, and Solution AB contains both the analytes of interest and the interfering analytes. Both solutions are analyzed at the beginning and at the end of each analytical sequence. When the interference check samples results are outside the control limits, corrective action must be taken, including sample reanalysis, if appropriate.

5.3.1.8 Retention Time Windows

Retention time windows for gas and liquid chromatographic analyses must be established by replicate injections of the calibration standard over multiple days, analytical method, or appropriate laboratory SOP. The absolute retention time of the calibration verification standard at the start of each analytical sequence will be used as the centerline of the window. For an analyte to be reported as positive, its elution time must be within the retention time window.

5.3.2 Quality Control Analyses Originated by the Field Team

5.3.2.1 Equipment Blank

Equipment rinsate blanks will be collected to evaluate field sampling and decontamination procedures by pouring deionized water over the decontaminated equipment. Equipment blanks will be collected on reusable sampling equipment for each matrix sampled and will be collected at a rate of one per week per matrix per equipment. Equipment blanks will not be collected on single-use or dedicated equipment. The equipment blanks will be analyzed for the same parameters specified for the corresponding matrix.

5.3.2.2 Trip Blank

Trip blanks are used to monitor for contamination during sample shipping and handling, and for cross-contamination through VOC migration among the collected samples. They are prepared in the laboratory by pouring ASTM International Type II or deionized water or methanol into a sample container for volatile analysis. They are then sealed, transported to the field, remain sealed while samples for volatile analysis are collected, and transported back to the laboratory in the same cooler as the samples for volatile analysis. One trip blank will accompany each cooler containing samples for volatile analysis, a separate trip blank is required for aqueous and solid matrices.

5.3.2.3 Field Duplicates

A field duplicate is an independent sample collected as close as possible to the original sample from the same source under identical conditions and is used to document sampling and analytical precision. Field duplicates will be collected at a minimum frequency of 5 percent or 1 per sampling event, whichever is more frequent, for each matrix and for each type of analysis. The sampling procedures described in Section 3 will be followed. The sampling locations for field duplicate samples will be recorded in the field logbook.

Duplicate samples will be collected simultaneously or in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis.

5.3.3 Temperature Blank

Temperature blanks will be used so that the laboratory can verify the temperature upon receipt of the samples. The temperature of the samples upon arrival will be annotated on the chain-of-custody form and also mentioned in the laboratory narrative that accompanies the analytical results. One temperature blank will accompany each sample cooler.

5.4 Additional Quality Control Requirements

5.4.1 Holding Time

The holding time requirements specified in this QAPP must be met. For methods requiring both sample preparation and analysis, the preparation holding time will be calculated from the time of sampling to the completion of preparation. The analysis holding time will be calculated from the time of completion of preparation to the time of completion of the analysis, including any required dilutions, confirmation analysis, and reanalysis. For methods requiring analysis only, the holding time is calculated from the time of sampling to completion of the analysis, including any required dilutions, confirmation analysis, and re-analysis.

5.4.2 Confirmation

Confirmation analysis must be carried out as specified for specific methods (SW8082 for this project) when the result is at or above the RL. The result designated as the primary result will be reported. All calibration and QC requirements must be met when confirmation analysis is carried out.

5.4.3 Cleanup Procedures to Minimize Matrix Effects

To maintain the lowest possible RLs, appropriate cleanup procedures will be employed when it is indicated by the method to remove or minimize matrix interference. Methods for sample cleanup include, but are not limited to, gel permeation chromatography, silica gel, alumna, Florisil, mercury (sulfur removal), sulfuric

acid, and acid/base partitioning. Method blanks, MS/MSDs, and LCSs must be subjected to the same cleanup procedures performed on the samples to monitor the efficiencies of these procedures.

5.4.4 Sample Dilution

Dilution of a sample results in elevated RLs and ultimately affects the usability of data related to potential actions at the sampling site. It is important to minimize dilutions and maintain the lowest possible RLs. When dilutions are necessary because of high concentrations of target analytes, lesser dilutions should also be reported to fully characterize the sample for each analyte. The level of the lesser dilution will be such that it will provide the lowest possible RLs without having a lasting deleterious effect on the analytical instrumentation.

When a sample exhibits characteristics of matrix interference that are identified through analytical measurement or visual observation, appropriate cleanup procedure(s) must be proven ineffective or inappropriate before proceeding with dilution and analysis.

5.4.5 Standard Materials and Other Supplies and Consumables

Standard materials must be of known high purity and traceable to an approved source. Pure standards must not exceed the manufacturer's expiration date or 1 year following receipt, whichever comes first. Solutions prepared by the laboratory from the pure standards must be used within the expiration date specified in the laboratory's SOP.

All other supplies and consumables must be inspected prior to use to ensure that they meet the requirements specified in the appropriate SOP. The laboratory's inventory and storage system should ensure their use within the manufacturer's expiration date and that the supplies are stored under proper conditions.

5.4.6 Manual Integration

The laboratory is required to provide all analysts performing methods that rely on interpretation of chromatographic data with training on appropriate software or manual-integration practices. The laboratory also will make every effort to minimize the use of manual integration of data. If the need arises to use manual integration to correct a software auto-integration error, the manual integration will be clearly identified in the instrument data. Before- and after-enlargements of the region of the chromatogram where the manual integration was performed will be provided on an appropriate scale to allow an independent reviewer to evaluate the need and quality of the manual integration. The analyst will also document the reason for the manual integration on the chromatogram along with their date and initials. The Laboratory Manager or designee will approve the manual integration by dating and initialing the chromatogram.

5.4.7 Laboratory Quality Assurance Program

The laboratory will maintain a QA manual or equivalent document. The QA manual will define the laboratory's internal QA/QC procedures including:

- QA policies, objectives, and requirements
- Organization and personnel
- Document control
- SOPs (analytical methods and administrative)
- Data generation
- Software verification
- QA

- QC
- Non-conformance/corrective action procedures
- Data review

5.4.7.1 Laboratory Standard Operating Procedures

The laboratory will maintain SOPs for all analytical methods and laboratory operations. The format for SOPs will conform to the following references:

- *Test Methods for Evaluating Solid Waste, Physical and Chemical Methods, SW-846, 3rd Edition, Update III, Section 1* (EPA 1996)
- *Good Laboratory Practices in Principles and Guidance to Regulations for Ensuring Data Integrity in Automated Laboratory Operations* (EPA 1995)

All SOPs must have a unique identification number that is traceable to previous revisions of the same document.

A copy of the laboratory SOPs will be provided as Attachment 1 to this QAPP once laboratory selection is complete.

5.4.7.2 Demonstration of Capability

Laboratory QA department personnel will maintain records documenting the ability of each analyst to perform applicable method protocols. Documentation will include annual checks for each method and analyst. In addition, internal, blind performance evaluation samples for each method and matrix demonstrating overall laboratory performance must be submitted annually. The laboratory may receive additional blind performance evaluation samples in conjunction with this program.

5.5 Reporting Limits and Analytical Requirements

Table C-5-1 contains a list of the analytical laboratories that will be used and the associated analytical and preparatory methods to be performed. Tables C-5-2 through C-5-10 contain lists of target analytes, the methods to be used, the RL objectives, and the project CULs specific to this project. Laboratory MDLs and RLs will be compared to target RLs and the project CULs prior to sample collection to ensure DQOs will be met.

The accuracy and precision limits are listed in Tables C-5-11 through C-5-18; the limits are only guidelines and data may be validated against laboratory-derived accuracy and precision limits if they are deemed appropriate. Calibration and QC requirements are specified in Tables C-5-19 through C-5-24. These tables are expectations for all laboratories and allow consistency amongst multiple laboratories.

Table C-5-11. Accuracy and Precision for Metals by Method SW6020B/SW7470A/SW7471B

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	LCS/LCSD/MS/MSD Accuracy (%R)	Precision (%RPD)
Arsenic	75 to 125	≤ 20
Cadmium	75 to 125	≤ 20
Copper	75 to 125	≤ 20
Lead	75 to 125	≤ 20
Mercury (SW7470A/SW7471B)	80 to 120	≤ 20
Zinc	75 to 125	≤ 20

Table C-5-12. Accuracy and Precision Limits for TPH by Method NWTPH-Dx

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	LCS/LCSD/MS/MSD Accuracy (%R)	Precision (%RPD)
TPH-DRO extractable	50 to 150	≤ 20
Surrogates		
Ortho-terphenyl	50 to 150	--

Table C-5-13. Accuracy and Precision Limits for Low-Level Pesticides by Method E1699 and E1699m

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	LCS/LCSD/MS/MSD Accuracy (%R)	Precision (%RPD)
2,4'-DDD	50 to 120	≤ 50
2,4'-DDE	50 to 120	≤ 50
2,4'-DDT	50 to 120	≤ 50
4,4'-DDD	50 to 120	≤ 50
4,4'-DDE	50 to 120	≤ 50
4,4'-DDT	50 to 120	≤ 50
Aldrin	50 to 120	≤ 50
alpha-Chlordane	50 to 120	≤ 50
cis-Nonachlor	50 to 120	≤ 50
Dieldrin	50 to 120	≤ 50
gamma-BHC (Lindane)	50 to 120	≤ 50
gamma-Chlordane	50 to 120	≤ 50
Oxychlordane	50 to 120	≤ 50
trans-Nonachlor	50 to 120	≤ 50

Table C-5-14. Accuracy and Precision Limits for PAHs by Method SW8270D-SIM

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	LCS/MS/MSD Accuracy – Solid (%R)	Precision (%RPD) – Water and Solid
1-Methylnaphthalene	40 to 120	≤25
2-Methylnaphthalene	40 to 120	≤25
Acenaphthene	40 to 120	≤ 25
Acenaphthylene	40 to 120	≤ 25
Anthracene	40 to 120	≤ 25
Benzo(a)anthracene	40 to 120	≤ 25
Benzo(a)pyrene	40 to 120	≤ 25
Benzo(b)fluoranthene	40 to 120	≤ 25
Benzo(k)fluoranthene	40 to 120	≤ 25
Benzo(g,h,i)perylene	40 to 120	≤ 25
Chrysene	40 to 120	≤ 25
Dibenzo(a,h)anthracene	40 to 120	≤ 25
Fluoranthene	40 to 120	≤ 25
Fluorene	40 to 120	≤ 25
Indeno(1,2,3-c,d)pyrene	40 to 120	≤ 25
Naphthalene	40 to 120	≤ 25
Phenanthrene	40 to 120	≤ 25
Pyrene	40 to 120	≤ 25
Surrogates		
2-Fluorobiphenyl	40 to 120	--
Terphenyl-d14	40 to 120	--

Table C-5-15. Accuracy and Precision Limits for bis(2-ethylhexyl)phthalate by SW8270D-LL
Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	LCS/LCSD/MS/MSD Accuracy (%R)	Precision (%RPD)
Bis(2-ethylhexyl)phthalate	47 to 110	≤ 40
Surrogates		
2,4,6-Tribromophenol	10 to 124	--
2-Fluorobiphenyl	22 to 85	--
2-Fluorophenol	10 to 84	--
Nitrobenzene-d5	14 to 85	--
Phenol-d6	10 to 88	--
Terphenyl-d14	30 to 102	--

Table C-5-16. Accuracy and Precision Limits for Dioxins and Furans by Method E1613B
Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	LCS/LCSD/MS/MSD Accuracy (%R) – Water and Solid	Precision (%RPD) – Water and Solid
2,3,7,8-TCDF	70 to 130	≤20
2,3,7,8-TCDD	70 to 130	≤20
1,2,3,7,8-PeCDF	70 to 130	≤20
2,3,4,7,8-PeCDF	70 to 130	≤20
1,2,3,7,8-PeCDD	70 to 130	≤20
1,2,3,4,7,8-HxCDF	70 to 130	≤20
1,2,3,6,7,8-HxCDF	70 to 130	≤20
2,3,4,6,7,8-HxCDF	70 to 130	≤20
1,2,3,7,8,9-HxCDF	70 to 130	≤20
1,2,3,4,7,8-HxCDD	70 to 130	≤20
1,2,3,6,7,8-HxCDD	70 to 130	≤20
1,2,3,7,8,9-HxCDD	70 to 130	≤20
1,2,3,4,6,7,8-HpCDF	70 to 130	≤20
1,2,3,4,7,8,9-HpCDF	70 to 130	≤20
1,2,3,4,6,7,8-HpCDD	70 to 130	≤20
OCDF	70 to 130	≤20
OCDD	70 to 130	≤20

Table C-5-16. Accuracy and Precision Limits for Dioxins and Furans by Method E1613B

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	LCS/LCSD/MS/MSD Accuracy (%R) – Water and Solid	Precision (%RPD) – Water and Solid
Surrogates		
$^{13}C_{12}$-2,3,7,8-TCDF	40 to 135	--
$^{13}C_{12}$-2,3,7,8-TCDD	40 to 135	--
$^{13}C_{12}$-1,2,3,7,8-PeCDF	40 to 135	--
$^{13}C_{12}$-2,3,4,7,8-PeCDF	40 to 135	--
$^{13}C_{12}$-1,2,3,7,8-PeCDD	40 to 135	--
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	40 to 135	--
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	40 to 135	--
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	40 to 135	--
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	40 to 135	--
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	40 to 135	--
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	40 to 135	--
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	40 to 135	--
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	40 to 135	--
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	40 to 135	--
$^{13}C_{12}$-OCDD	40 to 135	--
$^{37}Cl_4$-2,3,7,8-TCDD	40 to 135	--

Table C-5-17. Accuracy and Precision Limits for PCB Congeners by Method E1668

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	LCS/LCSD/MS/MSD Accuracy (%R) – Water and Solid	Precision (%RPD) – Water and Solid
PCB-1	50 to 150	≤20
PCB-2	50 to 150	≤20
PCB-3	50 to 150	≤20
PCB-4	50 to 150	≤20
PCB-10	50 to 150	≤20
PCB-9	50 to 150	≤20
PCB-7	50 to 150	≤20
PCB-6	50 to 150	≤20
PCB-5	50 to 150	≤20
PCB-8	50 to 150	≤20
PCB-14	50 to 150	≤20
PCB-11	50 to 150	≤20
PCB-(13/12)	50 to 150	≤20
PCB-15	50 to 150	≤20
PCB-19	50 to 150	≤20
PCB-(30/18)	50 to 150	≤20
PCB-17	50 to 150	≤20
PCB-27	50 to 150	≤20
PCB-24	50 to 150	≤20
PCB-16	50 to 150	≤20
PCB-32	50 to 150	≤20
PCB-34	50 to 150	≤20
PCB-23	50 to 150	≤20
PCB-(26/29)	50 to 150	≤20
PCB-25	50 to 150	≤20
PCB-31	50 to 150	≤20
PCB-(28/20)	50 to 150	≤20
PCB-(21/33)	50 to 150	≤20
PCB-22	50 to 150	≤20
PCB-36	50 to 150	≤20
PCB-39	50 to 150	≤20
PCB-38	50 to 150	≤20
PCB-35	50 to 150	≤20

Table C-5-17. Accuracy and Precision Limits for PCB Congeners by Method E1668

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	LCS/LCSD/MS/MSD Accuracy (%R) – Water and Solid	Precision (%RPD) – Water and Solid
PCB-37	50 to 150	≤20
PCB-54	50 to 150	≤20
PCB-(50/53)	50 to 150	≤20
PCB-(45/51)	50 to 150	≤20
PCB-46	50 to 150	≤20
PCB-52	50 to 150	≤20
PCB-(73/43)	50 to 150	≤20
PCB-(69/49)	50 to 150	≤20
PCB-48	50 to 150	≤20
PCB-(44/47/65)	50 to 150	≤20
PCB-(59/62/75)	50 to 150	≤20
PCB-42	50 to 150	≤20
PCB-(41/40/71)	50 to 150	≤20
PCB-64	50 to 150	≤20
PCB-72	50 to 150	≤20
PCB-68	50 to 150	≤20
PCB-57	50 to 150	≤20
PCB-58	50 to 150	≤20
PCB-67	50 to 150	≤20
PCB-63	50 to 150	≤20
PCB-(61/70/74/76)	50 to 150	≤20
PCB-66	50 to 150	≤20
PCB-55	50 to 150	≤20
PCB-56	50 to 150	≤20
PCB-60	50 to 150	≤20
PCB-80	50 to 150	≤20
PCB-79	50 to 150	≤20
PCB-78	50 to 150	≤20
PCB-81	50 to 150	≤20
PCB-77	50 to 150	≤20
PCB-104	50 to 150	≤20
PCB-96	50 to 150	≤20
PCB-103	50 to 150	≤20

Table C-5-17. Accuracy and Precision Limits for PCB Congeners by Method E1668

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	LCS/LCSD/MS/MSD Accuracy (%R) – Water and Solid	Precision (%RPD) – Water and Solid
PCB-94	50 to 150	≤20
PCB-95	50 to 150	≤20
PCB-(100/93/102/98)	50 to 150	≤20
PCB-(88/91)	50 to 150	≤20
PCB-84	50 to 150	≤20
PCB-89	50 to 150	≤20
PCB-121	50 to 150	≤20
PCB-92	50 to 150	≤20
PCB-(113/90/101)	50 to 150	≤20
PCB-83	50 to 150	≤20
PCB-99	50 to 150	≤20
PCB-112	50 to 150	≤20
PCB-(108/119/86/97/125/87)	50 to 150	≤20
PCB-(117/116/85)	50 to 150	≤20
PCB-(110/115)	50 to 150	≤20
PCB-82	50 to 150	≤20
PCB-111	50 to 150	≤20
PCB-120	50 to 150	≤20
PCB-(107/124)	50 to 150	≤20
PCB-109	50 to 150	≤20
PCB-123	50 to 150	≤20
PCB-106	50 to 150	≤20
PCB-118	50 to 150	≤20
PCB-122	50 to 150	≤20
PCB-114	50 to 150	≤20
PCB-105	50 to 150	≤20
PCB-127	50 to 150	≤20
PCB-126	50 to 150	≤20
PCB-155	50 to 150	≤20
PCB-152	50 to 150	≤20
PCB-150	50 to 150	≤20
PCB-136	50 to 150	≤20
PCB-145	50 to 150	≤20

Table C-5-17. Accuracy and Precision Limits for PCB Congeners by Method E1668

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	LCS/LCSD/MS/MSD Accuracy (%R) – Water and Solid	Precision (%RPD) – Water and Solid
PCB-148	50 to 150	≤20
PCB-(151/135)	50 to 150	≤20
PCB-154	50 to 150	≤20
PCB-144	50 to 150	≤20
PCB-(147/149)	50 to 150	≤20
PCB-(134/143)	50 to 150	≤20
PCB-(139/140)	50 to 150	≤20
PCB-131	50 to 150	≤20
PCB-142	50 to 150	≤20
PCB-132	50 to 150	≤20
PCB-133	50 to 150	≤20
PCB-165	50 to 150	≤20
PCB-146	50 to 150	≤20
PCB-161	50 to 150	≤20
PCB-(153/168)	50 to 150	≤20
PCB-141	50 to 150	≤20
PCB-130	50 to 150	≤20
PCB-137	50 to 150	≤20
PCB-164	50 to 150	≤20
PCB-(138/163/129)	50 to 150	≤20
PCB-160	50 to 150	≤20
PCB-158	50 to 150	≤20
PCB-(128/166)	50 to 150	≤20
PCB-159	50 to 150	≤20
PCB-162	50 to 150	≤20
PCB-167	50 to 150	≤20
PCB-(156/157)	50 to 150	≤20
PCB-169	50 to 150	≤20
PCB-188	50 to 150	≤20
PCB-179	50 to 150	≤20
PCB-184	50 to 150	≤20
PCB-176	50 to 150	≤20
PCB-178	50 to 150	≤20

Table C-5-17. Accuracy and Precision Limits for PCB Congeners by Method E1668

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	LCS/LCSD/MS/MSD Accuracy (%R) – Water and Solid	Precision (%RPD) – Water and Solid
PCB-175	50 to 150	≤20
PCB-187	50 to 150	≤20
PCB-182	50 to 150	≤20
PCB-186	50 to 150	≤20
PCB-(183/185)	50 to 150	≤20
PCB-174	50 to 150	≤20
PCB-177	50 to 150	≤20
PCB-181	50 to 150	≤20
PCB-(171/173)	50 to 150	≤20
PCB-172	50 to 150	≤20
PCB-192	50 to 150	≤20
PCB-(180/193)	50 to 150	≤20
PCB-191	50 to 150	≤20
PCB-170	50 to 150	≤20
PCB-190	50 to 150	≤20
PCB-189	50 to 150	≤20
PCB-202	50 to 150	≤20
PCB-201	50 to 150	≤20
PCB-204	50 to 150	≤20
PCB-(197/200)	50 to 150	≤20
PCB-(198/199)	50 to 150	≤20
PCB-196	50 to 150	≤20
PCB-203	50 to 150	≤20
PCB-195	50 to 150	≤20
PCB-194	50 to 150	≤20
PCB-205	50 to 150	≤20
PCB-208	50 to 150	≤20
PCB-207	50 to 150	≤20
PCB-206	50 to 150	≤20
PCB-209	50 to 150	≤20

Table C-5-18. Accuracy and Precision Limits for PCB Aroclors by Method SW8082-LL

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Analyte	LCS/LCSD/MS/MSD Accuracy (%R)	Precision (%RPD)
Aroclor-1016	70 to 130	≤ 25
Aroclor-1221	70 to 130	≤ 25
Aroclor-1232	70 to 130	≤ 25
Aroclor-1242	70 to 130	≤ 25
Aroclor-1248	70 to 130	≤ 25
Aroclor-1254	70 to 130	≤ 25
Aroclor-1260	70 to 130	≤ 25
Aroclor-1262	70 to 130	≤ 25

Table C-5-19. Calibration and Quality Control Requirements for Metals by Method SW6020B

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Quality Control Check	Frequency	Criteria	Corrective Action
Mass spectrometer tuning sample	Before initial calibration	Mass calibration ≤ 0.1 amu from the true value. Resolution < 0.9 amu full width at 10% peak height.	Retune instrument, then reanalyze tuning solution.
ICAL (a blank and at least one standard)	Before sample analysis, every 24 hours, whenever modifications are made to the system, or when CCV fails.	If more than one standard is used, correlation coefficient must be r ≥ 0.995.	N/A
Second-source calibration verification	Immediately following each ICAL.	All analytes within ±10% of expected value.	Correct problem and repeat ICAL.
Calibration blank	After every second-source or CCV analysis.	No analytes detected at or above the RL.	Correct the problem, then reanalyze previous 10 samples.
CCV	After every 10 samples and at the end of the analysis sequence.	All analytes within ±10% of expected value.	Recalibrate and reanalyze all samples since the last acceptable CCV.
Method blank	At least one per analytical batch.	No analytes detected at or above the RL.	Correct the problem and re-prep and reanalyze all associated samples.
Interference check standard (ICS-A and ICS-AB)	At the beginning of an analytical run or once during a 12-hour period, whichever is more frequent.	ICS-A: Absolute value of all non-spiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes). ICS-AB: Within ±20% of expected value.	Correct the problem, recalibrate, reanalyze ICS and all affected samples.
MS/MSD	One set per 20 project-specific samples. MSD is optional if a laboratory sample duplicate is performed.	All analytes within limits specified in accuracy and precision table.	None.
Laboratory sample duplicate	Once per analytical batch if MSD not performed.	All analytes within limits specified in accuracy and precision table.	None.
LCS/LCSD	At least one per analytical batch.	All analytes within limits specified in Accuracy and Precision table.	Correct the problem, and re-prep and reanalyze the LCS/LCSD and all samples in the analytical batch.
Dilution test	Each new sample matrix.	Result from 1:5 dilution must be within ±10% of the undiluted sample result (applies only if undiluted sample result is at least 25 times the RL).	Perform post-digestion spike addition.
Internal standard	Every field sample, standard and QC sample.	Internal standard intensity within 30 to 120% of intensity	If recoveries are acceptable for QC samples, but not

Table C-5-19. Calibration and Quality Control Requirements for Metals by Method SW6020B

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Quality Control Check	Frequency	Criteria	Corrective Action
		of the internal standard in the ICAL blank.	field samples, the field samples may be considered to suffer from a matrix effect. Reanalyze sample at 5-fold dilutions until criteria is met. For failed QC samples, correct problem, and rerun all associated failed field samples.
Linear range calibration check standard	Once per quarter.	All analytes within ±10% of expected value.	Correct problem then reanalyze or re-set linear range.
Post-digestion spike addition	When dilution test fails.	Recovery within 75 to 125% of expected value.	None.

Note:

ICS = interference check solution

Table C-5-20. Calibration and Quality Control Requirements for Metals by Method SW7470A/SW7471B

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Quality Control Check	Frequency	Criteria	Corrective Action
Multi-point ICAL (a blank and at least five standards)	Before initial sample analysis, every 24 hours, whenever modifications are made to the analytical system, or when CCV fails.	Correlation coefficient of linear regression is r ≥ 0.995	Correct the problem and repeat the ICAL.
Second-source calibration verification	Immediately following each ICAL.	All analytes within ±20% of expected value.	Correct the problem and repeat ICAL.
Calibration blank	After every second-source or CCV analysis.	No analytes detected at or above the RL.	Correct the problem, then reanalyze previous 10 samples.
CCV	After every 10 samples and at the end of the analysis sequence.	All analytes within ±20% of expected value.	Recalibrate and reanalyze all samples since the last acceptable CCV.
Method blank	At least one per analytical batch.	No analytes detected at or above the RL.	Correct the problem and re-prep and reanalyze all associated samples.
MS/MSD	One set per 20 project-specific samples. MSD is optional if a laboratory sample duplicate is performed.	All analytes within limits specified in accuracy and precision table.	None.

Table C-5-20. Calibration and Quality Control Requirements for Metals by Method SW7470A/SW7471B

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Quality Control Check	Frequency	Criteria	Corrective Action
Laboratory sample duplicate	Once per analytical batch if MSD not performed.	All analytes within limits specified in accuracy and precision table.	None.
LCS	At least one per analytical batch.	All analytes within limits specified in accuracy and precision table.	Correct the problem, and re-prep and reanalyze the LCS and all samples in the analytical batch.
Dilution test	Each new sample matrix.	Result from 1:5 dilution must be within ±10% of the undiluted sample result (applies only if undiluted sample result is at least 25 times the RL).	Perform recovery test.
Recovery test	When dilution test fails.	Recovery within 85 to 115% of expected value.	Analyze all samples by method of standard additions.

Table C-5-21. Calibration and Quality Control Requirements for TPH by Method NWTPH-Dx

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Quality Control Check	Frequency	Criteria	Corrective Action
Multi-point ICAL (minimum five points)	Prior to sample analysis, or when calibration verification fails.	If the %RSD is ≤ 20%, the average RRF may be used for quantitation; otherwise use calibration curve with coefficient of determination r2 ≥ 0.990.	Correct the problem and repeat the ICAL.
CCV	At the start of each analytical sequence and after every 10 samples, and at the end of the sequence.	Analytes within ±15% of expected value	Correct the problem, then recalibrate and reanalyze all samples since the last acceptable CCV.
Method Blank	At least one per analytical batch.	No analytes detected at or above the RL.	Correct the problem and re-prep and reanalyze all associated samples.
Surrogate spike	Every standard, sample, method blank, MS/MSD, and LCS.	All surrogates in samples, method blank, MS/MSD, and LCS within limits specified in Accuracy and Precision table.	Correct the problem and reanalyze (re-prep if necessary).
MS/MSD	One set per 20 samples.	Within limits specified in accuracy and precision table.	None.
LCS	At least one per analytical batch.	Within limits specified in Accuracy and Precision table.	Correct the problem, and re-prep and reanalyze the LCS and all samples in the analytical batch.

Note:

RRF = relative response factor

Table C-5-22. Calibration and Quality Control Requirements for PAHs by Method SW8270D-SIM and Low-Level bis(2-ethylhexyl)phthalate by Method SW8270D-LL

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Quality Control Check	Frequency	Criteria	Corrective Action
DFTPP Tuning	Prior to ICAL and calibration verification (every 12 hours).	Refer to criteria listed in the method.	Retune instrument and verify.
Multi-point ICAL (minimum five points)	Prior to sample analysis, or when calibration verification fails.	Option 1: Mean %RSD for all analytes ≤ 15% with no individual analyte RSD > 30% Option 2: Least squares regression $r^2 \geq 0.990$.	Correct the problem and repeat the ICAL.
Second-source calibration verification	Once for each multi-point ICAL.	All analytes within ±30% of expected value.	Correct the problem and repeat ICAL.

Table C-5-22. Calibration and Quality Control Requirements for PAHs by Method SW8270D-SIM and Low-Level bis(2-ethylhexyl)phthalate by Method SW8270D-LL

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Quality Control Check	Frequency	Criteria	Corrective Action
CCV	At the start of each analytical sequence and every 12 hours thereafter.	All compounds within 20%D of expected value.	Correct the problem, then recalibrate and reanalyze all samples since the last acceptable CCV.
Retention time window calculated for each analyte	Each analyte.	Relative retention time of each analyte within ± 0.06 relative retention time units of the CCV.	Not applicable (used for identification of analyte).
Internal standards	Each sample and quality control sample, method blank, MS/MSD, and LCS.	Retention time within ±30 seconds from retention time of the daily CCV standard. EICP area within –50% to +100% of the daily CCV standard.	Inspect mass spectrometer and GC for malfunctions; reanalyze all affected samples.
Method blank	At least one per analytical batch.	No analytes detected at or above the reporting limit.	Correct the problem, then re-prep and reanalyze all associated samples.
Surrogate spike	Every standard, sample, method blank, MS/MSD, and LCS.	At least two surrogates per fraction in samples, method blank and LCS within limits specified in accuracy and precision table.	Correct the problem and reanalyze (re-prep if necessary).
MS/MSD	One set per 20 project-specific samples.	Within limits specified in accuracy and precision table.	None.
LCS	At least one per analytical batch.	Within limits specified in accuracy and precision table.	Correct the problem, then re-prep and reanalyze the LCS and all samples in the analytical batch.

Note:

%D = percent drift

Table C-5-23. Calibration and Quality Control Requirements for Dioxins and Furans by E1613B, PCB Congeners by E1668 and Low-Level Pesticides by E1699 or E1699m

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Quality Control Check	Frequency	Criteria	Corrective Action
Performance check Use GC column performance check solution if the laboratory operates during consecutive 12-hour periods.	Prior to ICAL or calibration verification. At the beginning of each 12-hour period during which samples or calibration solutions are analyzed.	In accordance with the method.	Correct problem, then repeat column performance check.
Endrin/4,4'-DDT breakdown	Prior to ICAL or calibration verification.	In accordance with the method.	Correct problem, then repeat.
ICAL for all analytes identified in method	At instrument setup and after ICV or CCV failure, prior to sample analysis	Ion abundance ratios in accordance with the method. S/N ratio in accordance with the method.	Correct problem, then repeat ICAL.
Second-source calibration verification	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Ion abundance specified in the method must be met.	Correct the problem and repeat ICAL.
CCV	At the beginning of each 12-hour period, and at the end of each analytical sequence.	Ion abundance specified in the method must be met.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and recalibrate; then reanalyze all affected samples since the last acceptable CCV.
Internal standard	Every field sample, standard, and quality control sample.	%R for each IS in the original sample (prior to dilutions) must be within 40 to 135% of the ICAL average RF.	Correct problem, then re-prep and reanalyze the samples with failed IS.
Method blank	At least one per analytical batch.	No analytes detected at or above the reporting limit.	Correct the problem, then re-prep and reanalyze all associated samples.
MS/MSD	One set per 20 project-specific samples.	Within limits specified in accuracy and precision table.	None.

Table C-5-23. Calibration and Quality Control Requirements for Dioxins and Furans by E1613B, PCB Congeners by E1668 and Low-Level Pesticides by E1699 or E1699m

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Quality Control Check	Frequency	Criteria	Corrective Action
LCS	At least one per analytical batch.	Within limits specified in accuracy and precision table.	Correct the problem, then re-prep and reanalyze the LCS and all samples in the analytical batch.

Notes:

IS = internal standard

S/N = signal/noise ratio

Table C-5-24. Calibration and Quality Control Requirements for PCB Aroclors by Method SW8082-LL

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Quality Control Check	Frequency	Criteria	Corrective Action
Breakdown check (Endrin/DDT Method 8081 only)	Before sample analysis and at the beginning of each 12-hour shift.	Degradation of DDT and Endrin must each be ≤ 15%.	Correct problem, then repeat breakdown checks.
Multi-point ICAL (minimum five points)	Prior to sample analysis, or when calibration verification fails.	Option 1: %RSD for all analytes 20% if using average RRFs Option 2: Least squares regression r^2 0.990	Correct the problem and repeat the ICAL.
Second-source calibration verification	Once for each multi-point ICAL.	All analytes within 20% of expected value.	Correct the problem and repeat ICAL.
CCV	At the start of each analytical sequence and every 12 hours thereafter.	All analytes within + 20% of expected value,	Correct the problem, then recalibrate and reanalyze all samples since the last acceptable CCV.
Retention time window calculated for each analyte	Each analyte.	Relative retention time of each analyte within + 0.06 relative retention time units of the CCV.	Not applicable (used for identification of analyte).
Method blank	At least one per analytical batch.	No analytes detected at or above the reporting limit.	Correct the problem, then re-prep and reanalyze all associated samples.
Surrogate spike	Every standard, sample, method blank, MS/MSD, and LCS.	All analytes within limits specified in accuracy and precision table.	Correct the problem and reanalyze (re-prep if necessary).
MS/MSD	One set per 20 project-specific samples.	All analytes within limits specified in accuracy and precision table.	None.
LCS	At least one per analytical batch.	All analytes within limits specified in accuracy and precision table.	Correct the problem, then re-prep and reanalyze the LCS and all samples in the analytical batch.
Confirmation of positive results	All positive results must be confirmed	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%.	None.

6. Calibration Procedures and Frequency

6.1 Field Calibration Procedures

Field equipment will be calibrated before the start of work each sampling day. Any instrument drift from prior calibration will be recorded in the field notebook. Calibration will be in accordance with procedures and schedules outlined in the particular instrument's operations manual and the information included within the FSP.

Calibrated equipment will be uniquely identified by using either the manufacturer's serial number or other means. A label with the identification number and the date when the next calibration is due will be physically attached to the equipment. If this is not possible, records traceable to the equipment (for example, showing the equipment identification) will be readily available for reference. In addition, the results of calibrations and records of repairs will be recorded in the logbook.

Scheduled periodic calibration of testing equipment does not relieve field personnel of the responsibility of using properly functioning equipment. If an individual suspects an equipment malfunction, the device will be removed from service, tagged so that it is not inadvertently used, and the appropriate personnel notified so that a recalibration can be performed, or substitute equipment can be obtained.

Equipment that fails calibration or becomes inoperable during use will be removed from service and either segregated to prevent inadvertent use or tagged to indicate it is out of calibration. Such equipment will be repaired and satisfactorily recalibrated. Equipment that cannot be repaired will be replaced.

6.2 Laboratory Calibration Procedures

Qualified personnel will appropriately calibrate laboratory instruments prior to sample analysis. The requirements specified in each method and Section 5.2 will be followed. Only certified standards of known purity may be used for calibration. Calibration will be verified at specified intervals throughout the analysis. The frequency and acceptance criteria for calibration are specified for each analytical method in Tables C-5-19 through C-5-24. When multi-point calibration is specified, the concentrations of the calibration standards should bracket those expected in the samples. Samples must be diluted, if necessary, to bring analyte responses within the calibration range. The laboratory may only report those data that result from quantitation within the demonstrated working calibration range. Quantitation based on extrapolation is not acceptable. Section 5.2.3 addresses initial and continuing calibration requirements in greater detail.

7. Data Reduction, Validation, and Reporting

7.1 Laboratory Data Management

Data reduction will be performed manually or by using appropriate application software. Quantitation procedures specified for each method must be followed. If data reduction is performed manually, the documentation must include the formulas used. Any application software used for data reduction must have been verified previously by the laboratory for accuracy. Documentation of the software's verification must be maintained on file in the laboratory. All documentation of data reduction must allow re-creation of the calculations.

All data will undergo a minimum of three levels of review at the laboratory prior to release. The analyst performing the tests will initially review 100 percent of the data. After the analyst's review has been completed, 100 percent of the data will be reviewed independently by a senior analyst or by the section supervisor for accuracy; compliance with calibration, QC requirements, and holding times; and completeness. Analyte identification and quantitation must be verified. Calibration and QC results will be compared with the applicable control limits. RLs will be reviewed to make sure they meet the project objectives. Results of multiple dilutions will be reviewed for consistency. Any discrepancies must be resolved and corrected. Laboratory qualifiers will be applied when there are nonconformances that could potentially affect data usability. These qualifiers must be properly defined as part of the deliverables. All issues relevant to the quality of the data must be addressed in a case narrative. The laboratory QC Manager will review a minimum of 10 percent of data or deliverables generated for this program against the project-specific requirements. The Laboratory Manager or Client Service Representative will conduct a final data review to ensure that all required analyses were performed on all samples and that all documentation is complete.

The hard copy and electronic laboratory reports for all samples and analyses will contain the information necessary to perform data evaluation.

7.1.1 Content of Deliverables

There are four types of reporting deliverables, three types will be used for this project depending upon the DQOs. Following is a brief synopsis of when it is appropriate to use each deliverable:

- **Level 1**—Appropriate for screening sample results or for geotechnical data such as grain size. Non-critical project decisions are made using these data.
- **Level 2**—Appropriate for investigative samples results that will be replaced with confirmatory data or results used for disposal purposes. Less-critical project decisions are made using these data.
- **Level 4**—Appropriate for investigative, confirmatory, or closure results. Critical decisions may be made using these data and will be used for projects that require a high degree of confidence in the accuracy of the data.

Hard copy deliverables, in summary format, containing the necessary information to perform data evaluation/data validation are required. Reporting formats similar to those specified in the latest versions of the EPA *Contract Laboratory Program Statements of Work for Organics and Inorganics Analyses* are preferred. The laboratory data report will be organized in a format that facilitates data identification and retrieval. Alternate reporting formats require approval from the Project Chemist.

Level 1: Select methods such as grain size will be reported as a Level 1 report and will include that following at a minimum (when applicable):

- Cover letter complete with:
  - Title of report and laboratory unique report identification (sample delivery group number)
  - Project name and location
  - Name and location of laboratory and second-site or subcontracted laboratory
  - Client name and address
  - Statement of authenticity and official signature and title of person authorizing report release
- Table of contents
- Summary of samples received that correlates field sample IDs with the laboratory IDs
- Laboratory qualifier flags and definitions
- Field identification number
- Date received
- Date prepared
- Date analyzed (and time of analysis if the holding time is less than or equal to 48 hours)
- Preparation and analytical methods
- Result for each analyte (dry-weight basis for soil)
- Percent solids results for soil samples
- Dilution factor (provide both diluted and undiluted results when available)
- Sample-specific reporting limit adjusted for sample size, dilution/concentration
- Sample-specific MDL adjusted for sample size and dilution/concentration (when project objectives require reporting less than the reporting limit)
- Units

Level 2: Ninety percent of the laboratory reports provided will be Level 2 reports and will consist of all elements included in a Level 1 deliverable plus:

- Case narrative that addresses the following information, at a minimum:
  - Sample receipt discrepancies, such as bubbles in VOA samples and temperature exceedances
  - Descriptions of all nonconformances in the sample receipt, handling, preparation, analytical and reporting processes, and corrective action taken in each occurrence (including internal standard, ICAL, continuing calibration, and retention time exceedances)
  - Identification and justification for sample dilution
- Surrogate percent recoveries
- MS/MSD and LCS spike concentrations, native sample results, spiked sample results, percent recoveries, and RPDs between the MS and MSD results. Associated QC limits must also be provided
- Method blank results
- Analytical batch reference number that cross references samples to QC sample analyses
- Executed chain-of-custody and sample receipt checklist

Level 4: Ten percent of the laboratory reports provided will be Level 4 and will include all elements outlined above for the Levels 1 and 2 reports plus the additional items listed below and all associated raw data. Up to 100 percent of dioxins and furans data may require a Level 4 data report. It is imperative that

the chromatographic and other instrument data be supplied in a scale that facilitates review from hard copy. Sufficient "blow ups" of complex areas of sample chromatograms will be provided. Additional information to be supplied will include:

- Analytical sequence or laboratory run log that contains sufficient information to correlate samples reported in the summary results to the associated method QC information, such as initial and continuing calibration analyses
- Confirmation results
- Calibration blank results for inorganic analyses (required in hard copy format only)
- ICP interference check sample true and measured concentrations and percent recoveries (required in hard copy format only)
- Method of standard addition results (if applicable; required in hard copy format only)
- Post-digestion spike recoveries (if applicable; required in hard copy format only)
- Internal standard recovery and retention time information, as applicable
- ICAL summary, including standard concentrations, RFs, average RFs, RSDs or correlation coefficients, and calibration plots or equations, if applicable (required in hard copy format only)
- CCV summary, including expected and recovered concentrations and percent differences (required in hard copy format only)
- Instrument tuning and mass calibration information for GC/mass spectrometry and ICP/mass spectrometry analyses
- Any other method-specific QC sample results
- Sample preparation logs that include the following information:
  - Preparation start and end times
  - Beginning and ending temperatures of water baths and digestion blocks
- Example calculation for obtaining numerical results from at least one sample for each matrix analyzed (provide algorithm)
- Reconstructed total ion chromatograms or selected ion current profiles for each sample (or blank) analyzed and mass spectra(s) for each compound identified, including:
  - Raw compound spectra
  - Enhanced or background spectra
  - Laboratory-generated library spectra (for tentatively identified compounds, provide the reference mass spectra(s) from software spectra library)
- Ion ratio information for dioxin/furan methods

7.1.2 Hard Copy and Electronic Deliverables

The laboratory will deliver electronic data deliverables as specified in the laboratory SOW within the timeframe specified in the laboratory SOW, contract, or purchase order from sample receipt.

All electronic data files will match the final hard copy results.

All raw data will be maintained on file in the laboratory and will be available on request by project management. Complete documentation of sample preparation and analysis and associated QC information will be maintained in a manner that allows easy retrieval in the event that additional validation or information is required. All data generated in the laboratory must be maintained and backed

up and will be made available upon request. All documentation must be retained for a minimum of 10 years after data acquisition.

The primary responsibility for the implementation of these procedures within the laboratory will reside with the Laboratory Manager or equivalent. The Laboratory Manager will approve laboratory reports before transferring the information to the client.

7.2 Data Validation and Verification

Laboratory analytical data acquired during this investigation will be reviewed and validated by the Project Chemist or equally qualified chemist to assure the data quality criteria set forth are met and that the data are usable for their intended purpose.

Validation will be performed in a manner consistent with the 2018-2019 Pre-RD Investigation (AECOM and Geosyntec 2019). EPA Stage 2A data validation will be conducted on 90 percent of the laboratory data. Where data are recorded for purposes of calculation or data reduction to a reported result (for example, total solids or grain size), 100 percent of the data per analytical test and per matrix will be subjected to EPA Stage 1 validation only. Stage 4 validation will be conducted on 10 percent of the data per analytical test and per matrix where instrument outputs are generated during analysis (for example, high-resolution mass spectrometry, GC, and ICP mass spectrometry). Up to 100 percent of dioxins and furans data may require Stage 4 validation based on review of summary data against project DQOs. If systematic data quality issues are identified with the analytical data during validation, the laboratory will be contacted, and data will be validated using Stage 4 protocols until the data quality issues are resolved.

The validated analytical data will be compiled into a Scribe database and an electronic data deliverable will be prepared and provided to EPA in the standard regional format per the ASAOC SOW (Section 5.4(a)) and the *Program Data Management Plan, Portland Harbor Remedial Design Investigation Portland Harbor Superfund Site* (EPA 2020).

Data validation will use guidance from the EPA *National Functional Guidelines for Inorganic Superfund Methods Data Review* (2017a) and *National Functional Guidelines for Organic Superfund Methods Data Review* (2017b), substituting the calibration and QC requirements specified in QAPP Tables C-5-19 through C-5-24 for those specified in the guidelines. The general flagging criteria defined in Table C-7-1 will be used; the qualifier flags are defined in Table C-7-2. Attachment 2 of this QAPP includes the Data Quality Evaluation Report Template.

7.2.1 Data Validation Levels

Personnel involved in the data validation function will be independent of any data generation effort. There will be three levels of data validation: Stage 1, Stage 2A, and Stage 4. The Project Chemist will oversee the data validation effort. Data validation will be carried out when the data packages are received from the laboratory. The validation will be performed on an analytical batch basis using the summary results of calibration and laboratory QC, as well as those of the associated field samples. Data packages will be reviewed for all constituents of concern.

7.2.1.1 Stage 1 Data Validation

Stage 1 validation of the laboratory analytical data package consists of verification and validation checks for the compliance of sample receipt conditions, sample characteristics (for example, percent moisture), and analytical results (with associated information). This level of validation will be performed on the Level 1 report deliverables outlined in Section 7.1.1 and is applicable only to data such as total solids, grain size, or IDW samples that are not critical to project decisions or used for comparison to project

screening levels. It is recommended that the following minimum baseline checks (as relevant) be performed on the laboratory analytical data package received for a Stage 1 validation label:

- Documentation identifies the laboratory receiving and conducting analyses and includes documentation for all samples submitted by the project or requester for analyses.
- Requested analytical methods were performed and the analysis dates are present.
- Requested target analyte results are reported along with the original laboratory data qualifiers and data qualifier definitions for each reported result.
- Requested target analyte result units are reported.
- Requested RLs for all samples are present and results at and below the requested RLs are clearly identified.
- Sampling dates and times, date and time of laboratory receipt of samples, and sample conditions upon receipt at the laboratory (including preservation, pH, and temperature) are documented.
- Sample results are evaluated by comparing sample conditions upon receipt at the laboratory (for example, preservation checks) and sample characteristics (for example, percent moisture) to the requirements and guidelines.

7.2.1.2 Stage 2A Data Validation

Stage 2A validation builds on the validation conducted in Stage 1. Stage 2A validation of the laboratory analytical data package consists of the Stage 1 validation plus the verification and validation checks for the compliance of sample-related QC. This level of validation is performed on the Level 2 reports outlined in Section 7.1.1 and applies to all methods used for critical project decisions, compliance with DQOs, or comparison to project screening levels. It is recommended that the following additional minimum baseline checks be performed on the laboratory analytical data package received for a Stage 2A validation:

- Requested methods (handling, preparation, cleanup, and analytical) are performed.
- Method dates for handling, preparation, cleanup, and analysis are present, as appropriate.
- Sample-related QC data and QC acceptance criteria (e.g., method blanks, surrogate recoveries, deuterated monitoring compound [DMC] recoveries, LCS recoveries, duplicate analyses, MS/MSD recoveries, serial dilutions, post-digestion spikes, and standard reference materials) are provided and linked to the reported field samples (including the field QC samples such as trip and equipment blanks).
- Requested spike analytes or compounds (for example, surrogate, DMCs, LCS spikes, and post-digestion spikes) have been added, as appropriate.
- Sample holding times (from sampling date to preparation and preparation to analysis) are evaluated.
- Frequency of QC samples is checked for appropriateness (for example, one LCS per 20 samples in a preparation batch).
- Sample results are evaluated by comparing holding times and sample-related QC data to the requirements and guidelines present in national or regional data validation documents, analytical method(s), or contract.

7.2.1.3 Stage 4 Data Validation

Stage 4 validation builds on the validation conducted in Stage 2A. Stage 4 validation of the laboratory analytical data package consists of the Stage 2A validation plus the evaluation of instrument outputs. This level of validation is performed on the Level 4 reports outlined in Section 7.1.1 and applies to all methods

used for critical project decisions, compliance with DQOs or comparison to project screening levels. It is recommended that the following additional minimum baseline checks (as relevant) be performed on the laboratory analytical data package received for a Stage 4 validation:

- ICAL data (for example, ICAL standards, ICV standards, ICAL blanks) are provided for all requested analytes and linked to field samples reported. For each ICAL, the calibration type used is present along with the ICAL equation used including any weighting factor(s) applied and the associated correlation coefficients, as appropriate. Recalculations of the standard concentrations using the ICAL curve are present, along with their associated percent recoveries. For the ICV standard, the associated percent recovery (or percent difference, as appropriate) is present.
- Appropriate number and concentration of ICAL standards are present.
- Continuing calibration data (for example, CCV standards and continuing calibration blanks [CCBs]) are provided for all requested analytes and linked to field samples reported, as appropriate. For the CCV standard(s), the associated percent recoveries (or percent differences, as appropriate) are present.
- Reported samples are bracketed by CCV standards and CCBs standards as appropriate.
- Method-specific instrument performance checks are present as appropriate (e.g., tunes for mass spectrometry methods, DDT/Endrin breakdown checks for pesticides and Aroclors, and instrument blanks and interference checks for ICP methods).
- Frequency of instrument QC samples is checked for appropriateness (e.g., GC-mass spectroscopy tunes have been run every 12 hours).
- Sample results are evaluated by comparing instrument-related QC data to the requirements and guidelines present in national or regional data validation documents, analytical method(s), or contract.
- Instrument response data (e.g., GC peak areas and ICP corrected intensities) are reported for requested analytes, surrogates, internal standards, and DMCs for all requested field samples, MSs, MSDs, LCS, and method blanks as well as calibration data and instrument QC checks (e.g., DDT/Endrin breakdowns, inter-element correction factors, and Florisil cartridge checks).
- Reported target analyte instrument responses are associated with appropriate internal standard analyte for each analyte (for methods using internal standard for calibration).
- Fit and appropriateness of the ICAL curve used or required (e.g., mean calibration factor, regression analysis [linear or non-linear, with or without weighting factors, with or without forcing]) is checked with recalculation of the ICAL curve for each (or selected) analyte(s) from the instrument response.
- Comparison of instrument response to the minimum response requirements for each analyte.
- Recalculation of each opening and closing CCV (and CCB) response from the peak data reported for each analyte from the instrument response, as appropriate.
- Compliance check of recalculated opening and/or closing CCV (and CCB) response to recalculated ICAL response for each analyte.
- Recalculation of percent ratios for each tune from the instrument response, as appropriate.
- Compliance check of recalculated percent ratio for each tune from the instrument response.
- Recalculation of each instrument performance check (for example, DDT/Endrin breakdown for pesticide analysis, instrument blanks, and interference checks) from the instrument response.
- Recalculation and compliance check of retention time windows (for chromatographic methods) for each analyte from the laboratory reported retention times.
- Recalculation of reported results for each reported target analyte from the instrument response.

- Recalculation of each reported spike recovery (surrogate recoveries, DMC recoveries, LCS recoveries, duplicate analyses, MS/MSD recoveries, serial dilutions, post-digestion spikes, and standard reference materials) from the instrument response.
- Each sample result and spike recovery are evaluated by comparing the recalculated numbers to the laboratory reported numbers according to the requirements and guidelines present in national or regional data validation documents, analytical method(s), or contract.
- All required instrument outputs (for example, chromatograms, mass spectra, atomic emission spectra, instrument background corrections, and interference corrections) for evaluating sample and instrument performance are present.
- Sample results are evaluated by checking each instrument output (e.g., chromatograms, mass spectra, atomic emission spectra data, instrument background corrections, and interference corrections) for correct identification and quantitation of analytes (e.g., peak integrations, use of appropriate internal standards for quantitation, elution order of analytes, and interferences).
- Each instrument's output is evaluated for confirmation of nondetect or tentatively identified analytes.

7.2.2 Data Validation Qualifiers and Reasons

Qualifier flags, if required, will be applied to the electronic sample results. If multiple flags are required for a result, the most severe flag will be applied to the electronic result. The hierarchy of flags from the most severe to the least severe will be as follows: R, UJ, U, J-, J+ and J.

Table C-7-1. General Flagging Conventions

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

QC Requirement	Criteria	Flag	Flag Applied to
Holding time	Time exceeded for extraction or analysis Time exceeded for extraction or analysis by a factor of > 2	J- for positive results; UJ for NDs J- for positive results; R for NDs	All analytes in sample (use professional judgment for PCBs and dioxins/furans)
Sample preservation	Sample not preserved; however, if sample preservation was not done in the field but was performed at the laboratory upon sample receipt, no flagging is required	J for positive results; UJ for NDs	Sample
	Sample receipt temperature > 6°C No flagging required if samples received the same day as collection and samples were on ice	J- for positive results; UJ for NDs	
Sample integrity (SW8260B/ SW8015)	Bubbles in VOA vial > 0.25 inch used for analysis	J- for positive results; UJ for NDs	Sample
Instrument tuning	Ion abundance method-specific criteria not met	R for all results	All associated samples in analytical batch
ICAL	All analytes must be within method-specified criteria	J for positive results; UJ for NDs	All associated samples in analytical batch
Second source check or continuing calibration	All analytes must be within method-specified criteria	High bias: J+ for positive results Low bias: J- for positive results; UJ for NDs	All associated samples in analytical batch
Low-level calibration check	All analytes must be within 50 percent of expected value	High bias: J+ for positive results Low bias: J- for positive results; UJ for NDs	All associated samples in analytical batch
ICS check for metals	All analytes must be within 20 percent of expected value	High bias: J+ for positive results Low bias: J- for positive results; UJ for NDs	All associated samples in analytical batch

Table C-7-1. General Flagging Conventions

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

QC Requirement	Criteria	Flag	Flag Applied to
LCS/LCSD	%R > UCL %R < LCL %R < LCL and < 10 percent LCS/LCSD RPD > CL	J+ for positive results J- for positive results; UJ for NDs J- for positive results; R for NDs J for positive results	The specific analyte(s) in all samples in associated analytical batch
Internal Standards (organic methods)	Area > UCL Area < LCL	J- for positive results; UJ for NDs J+ for positive results	Associated analytes in sample
Internal Standards (inorganic methods)	Area > UCL Area < LCL	J for positive results; UJ for NDs J for positive results; UJ for NDs	Associated analytes in sample
Surrogate spikes	%R > UCL %R < LCL and > 10 percent %R <10 percent	J+ for positive results J- for positive results; UJ for NDs J- for positive results; R for NDs	Associated analytes in sample
	Dilution > 10 times	No flagging required for failed surrogates	
Blanks (method, equipment, or trip)	Analyte(s) detected	U for positive sample results ≤ 5 times highest blank concentration	All samples in preparation, field or analytical batch, whichever applies
FDs	RPD > CL and FDs > RLs RPD>CL and one FD > RL, one ND or <RL	J for positive results J for positive results; UJ for NDs	The specific analyte(s) in FD pair Note: No flagging is required for RPDs based on both concentrations less than the RLs or where one result is ND and one is less than the RL
Laboratory sample duplicates	RPD>CL and sample concentration >RL	J for positive results	The specific analyte(s) in parent sample and FD only.
Estimated Maximum Possible Concentrations	Analyte is flagged as estimated maximum possible concentrations by the laboratory	J for positive results	Associated analytes in sample

Table C-7-1. General Flagging Conventions

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

QC Requirement	Criteria	Flag	Flag Applied to
MS/MSD	MS or MSD %R > UCL MS or MSD %R < LCL MS or MSD %R < LCL and <10 percent MS/MSD RPD > CL Sample concentration > 4 times spike concentration; excessive dilution[a]	J+ for positive results J- for positive results; UJ for NDs J- for positive results; R for NDs J for positive results No flag required	The specific analyte(s) in parent sample and its FD only, if FD collected from the same location and depth as the parent sample.
Post-digestion spike	All analytes must be within 25 percent of expected value Sample concentration > 4 times spike concentration; excessive dilution[a]	High bias: J+ for positive results Low bias: J- for positive results; UJ for NDs No flag required	The specific analyte(s) in parent sample
Serial dilutions	All analytes must be within 10 percent of expected value Sample concentration < 50 times MDL	If Post Spike not analyzed; J for positive results No flag required	The specific analyte(s) in parent sample
Confirmation	RPD between primary and confirmation results > 40 percent	J for positive results	The specific analyte(s) in the sample
RT window	Analyte retention time outside of established window	R for all results	The specific analyte(s) in the sample
Result greater than ICAL range	Analyte reported greater than ICAL range	J for positive result	The specific analyte(s) in the sample

[a] Spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more."

Notes:

> = greater than

%R = percent recovery

CL = control limit

FD = field duplicate

ND = nondetect

RT = retention time

UCL = upper control limit

Table C-7-2. Qualifier Flag Definitions

Quality Assurance Project Plan, Willbridge Cove Project Area, Portland, Oregon

Flag	Definition
J	Analyte was present but reported value may not be accurate or precise.
J+	Analyte was present but reported value may not be accurate or precise, high bias.
J-	Analyte was present but reported value may not be accurate or precise, low bias.
R	This result has been rejected.
U	This analyte was analyzed for but not detected at the specified reporting limit.
UJ	The analyte was not detected above the detection limit objective; however, the reported detection limit is approximate and may or may not represent the actual limit of quantitation necessary to measure the analyte accurately and precisely in the sample.

8. Performance Evaluations

To assess sample and data collection procedures, performance evaluations will be conducted and will consist of technical systems audits and performance audits. Assessment information will be reported to the PDI PM and Project QAM.

8.1 Technical Systems Audits

This section defines technical audits for the laboratories performing analyses and of field activities.

8.1.1 Laboratory Audits

The laboratories participating in the data collection effort may undergo an audit. Audits may also be undertaken by the regulatory agencies. Laboratory audits may be performed as either onsite audits, desk audits (review of sample receipt and analysis against laboratory QAM and SOPs), or a combination of both.

8.1.2 Field Audits

Field audits will be performed once a year, or as necessary, by an independent auditor, who is not part of the field team, to verify the execution of field procedures. An example of a filed audit checklist is included as Attachment 3, and general procedures to be evaluated include the following:

- Sample containers and preservatives
- Sample collection and identification procedures
- Sample custody, handling, and shipping procedures
- Equipment decontamination procedures
- Calibration of field instruments and performance of field tests
- Documentation of field activities, maintenance of field records, and document control

8.2 Performance Audits

This section defines performance audits.

8.2.1 Performance Evaluations

Laboratories are required to participate in a performance evaluation program. Any method or analyte failure in a performance evaluation program that affects the certification status of the laboratory must be immediately communicated to the Project Chemist.

8.2.2 External Audits

Announced and unannounced audits of the field operations and of the laboratories may be conducted during any stage of the project.

8.2.3 Internal Audits

The laboratory's QA Officer will conduct annual audits of the laboratory. The audits will verify, at a minimum, that written SOPs are being followed; standards are traceable to certified sources; documentation is complete; data review is being performed effectively and is properly documented; and data reporting, including electronic and manual data transfer, is accurate and complete. All audit findings will be

documented in QA reports to management. Necessary corrective actions will be taken within a reasonable timeframe. The QA Officer will verify that such actions are effective and complete and will document their implementation in an audit closeout report to laboratory management. The internal audit reports will be made available to the Jacobs PM and QAM, as well as to the EPA PC within 10 days of receipt by Jacobs.

9. Preventive Maintenance

The primary objective of a preventive maintenance program is to promote the timely and effective completion of a measurement effort. The maintenance program will be designed to minimize the downtime of crucial sampling and/or analytical equipment from expected or unexpected component failure. In implementing this program, efforts will be focused on:

- Establishing maintenance responsibilities
- Establishing maintenance schedules for major and/or critical instrumentation and apparatus
- Establishing an adequate inventory of critical spare parts and equipment

9.1 Maintenance Responsibilities

Laboratory instrument maintenance is the responsibility of the participating laboratory. Generally, the Laboratory Manager or Supervisor is responsible for the instruments in their work area. This responsible person will establish maintenance procedures and schedules for each instrument.

Maintenance responsibilities for field equipment are assigned to the FTL for specific sampling tasks. However, the field team using the equipment is responsible for checking the status of the equipment prior to use and reporting any problems encountered. The field team is also responsible for ensuring that critical spare parts are included as part of the field equipment checklist. Non-operational field equipment will be removed from service, and a replacement will be obtained. All field instruments will be properly protected against inclement weather conditions during the field investigation.

9.2 Maintenance Schedules

The effectiveness of any maintenance program depends, to a large extent, on adherence to specific maintenance schedules for each piece of equipment. Other maintenance activities are conducted as needed. Manufacturers' recommendations should provide the primary basis for establishing maintenance schedules. Manufacturers' service contracts may be used for implementing the scheduled maintenance.

Each laboratory analytical instrument will be assigned an instrument logbook. All maintenance activities will be documented in this logbook. The logbook should contain:

- Date of service
- Person performing service
- Type of service performed and reason for service
- Replacement parts installed (if appropriate)
- Date of next scheduled service
- Any other useful information

Each field instrument will be maintained as outlined in Section 6 of the FSP.

9.3 Spare Parts

In addition to a schedule for maintenance activities, an adequate inventory of spare parts is required to minimize equipment down time. The inventory includes those parts and supplies that:

- Are subject to frequent failure.
- Have limited useful lifetimes.
- Cannot be obtained in a timely manner should failure occur.

Field managers and the respective laboratory managers are responsible for maintaining an adequate inventory of spare parts. In addition to spare parts and supply inventories, an in-house source of backup equipment and instrumentation will be available.

10. Data Assessment

All data generated for this project will be evaluated according to the QA acceptance criteria specified in Tables C-5-11 through C-5-18. Limitations on data usability will be assigned, if appropriate, as a result of the validation process described in Section 8.

A usability assessment is performed to determine how well the data collected for a particular project support the project objectives. Decisions can then be made by the PM.

Data gaps may be present if (1) a sample is not collected, (2) a sample is not analyzed for the requested parameters, or (3) the data are determined to be unusable. The need for further investigation will be determined on a case-by-case basis. This determination will depend on whether data can be extrapolated from adjacent sample locations and whether the data are needed based on the results from adjacent sample locations.

The Project Chemist and the analytical laboratory subcontractor will ensure that the collected data meet the MDLs and RLs specified in the laboratory statements of work and the laboratory QC limits specified in this QAPP. During the data validation assessment, nonconformances will be documented, and the Project Chemist will apply validation qualifiers in accordance with the validation procedures outlined in this QAPP. The Project Chemist will then evaluate potential effects on data usability and work with the project team to determine whether the data are usable within the context of project objectives.

With the exception of rejected data, all data are usable as qualified by the Project Chemist. Estimated and/or biased results are usable. Outliers, if present, can be addressed on a case-by-case basis. There is no generic formula for determining whether a result is an outlier. Potential outliers will be referred to a statistician and/or senior consultant, who will determine which formulas are appropriate for classifying data points in a statistically appropriate and defensible manner. Data will not be eliminated from consideration on the basis of statistical outlier testing.

11. Corrective Action

Corrective action may be required as a result of deviations from field or analytical procedures. Deficiencies identified in audits and data quality evaluations may also call for corrective action. All project personnel have the responsibility, as part of the normal work duties, to identify, report, and solicit approval of corrective actions for conditions adverse to data quality.

This QAPP has specified the corrective action to be taken when deviations from calibration and QC acceptance criteria occur. These are listed in Tables C-5-19 through C-5-24. Field and laboratory staff may encounter conditions that require immediate corrective action that are not addressed in the monitoring plan or QAPP. These personnel will document conditions and the results of corrective actions in a field logbook or laboratory non-conformance report and communicate their actions as soon as feasible to the FTL, Laboratory Supervisor, and if necessary, the Project Chemist, for immediate input. A mechanism must be established to allow for supervisory review and/or client input for all deviations or deficiencies. A corrective action reporting system that requires immediate documentation of deviations or deficiencies and for supervisory review of the actions taken to correct them will be established. At a minimum, the corrective action report should include:

- The type of deviation or deficiency
- The date of occurrence
- The impact of the deviation or deficiency, such as samples affected
- The corrective action taken
- Documentation that the process has been returned to control

The only time that a corrective action report may be waived is when a deviation or deficiency is immediately corrected, and its impact is precluded. An example would be an unacceptable ICAL that is correctly calibrated before samples are analyzed.

Each corrective action report must be reviewed and approved by a person of authority, such as the FTL or Laboratory Supervisor. The ultimate responsibility for the laboratory corrective action process is the QC Manager, who must ensure the performance of proper documentation, approval, and close out of all out-of-control or non-conformance events. A non-conformance report will summarize each non-conformance condition. Corrective action reports that could potentially affect data quality must be brought to the attention of the Project Chemist. Report disposition will be the responsibility of the Project Chemist. The PM may be notified about a particular report at the Project Chemist's discretion. Copies of corrective action reports must be maintained in the laboratory or field project files.

12. Quality Assurance Reports

The Project Chemist will submit a QA report to the PM at the end of each sampling interval. The report will summarize the results of the data validation and the data assessment. The results will be presented in a manner that facilitates decision making. For example, temporal data may be more effectively presented if supplemented by a time plot. Any significant quality problems and recommended solutions will be included in the report. Limitations on data usability that were identified during data validation will be highlighted.

The usability of the data collected during field activities will be assessed in several ways:

- Any deviations from proposed field activities and sampling and handling procedures will be reviewed and their effect on data usability evaluated.
- Analytical results, including nondetect values, will be compared against the project CULs identified in the laboratory statements of work.
- Data verification and validation will be completed following the procedures in Section 7 of this QAPP.
- Based on the results of data verification and validation, the usability of the data will be evaluated in the context of project-specific DQOs following the procedures in Section 10.

13. Data Management

The electronic data will be used to generate validation reports, risk assessment calculations, modeling results, data summary tables, maps, and other figures. This program will follow standard procedures for environmental data collection. These protocols give data users simple procedures to rapidly access stored data; ensure consistency among all field activities; provide methods of data entry with known accuracy and efficiency; apply well-documented validation procedures to an electronic database; manage sample data using unique sample identification numbers; establish a sample inventory of new data collected and provide methods of sample inventory reconciliation; store and provide sample-specific attributes, including location identifiers, sample type and media, and sample date; and provide reporting and delivery formats to support data analysis and reduction.

Validated data will be loaded to the Portland Harbor database where historical data for the site are maintained.

13.1 Archiving

Hard copy and electronic versions will be archived in project files and on electronic archive tapes for the duration of the project, 5 years, or as specified in contractual agreements.

13.2 Data Flow and Transfer

The data flow from the laboratory and field to the project staff and data users will be sufficiently documented to ensure that data are properly tracked, reviewed, and validated before use.

13.3 Record Keeping

In addition to the data management procedures outlined in Section 7.1 for analytical data, the laboratory will ensure that they maintain electronic and hard copy records sufficient to recreate each analytical event. The minimum records the laboratory will keep contain the following:

- Raw data, including instrument printouts, bench work sheets, and/or chromatograms with compound identification and quantitation reports.
- Laboratory-specific written SOPs for each analytical method and QA/QC function in place at the time of analysis of project samples. Laboratory SOPs will be included as Attachment 1 to the final QAPP once the laboratory is selected.

13.4 Existing Data and Non-Direct Measurements

Existing sediment chemistry data from previous investigations within the PHSS are expected to be used for RD. These data sets include currently EPA-approved Feasibility Study data and the data from the 2018 Pre-RD study that are posted to the PHSS data portal. Additional data that may be used include data collected by to support maintenance dredging in the vicinity of the project area. The limitations of these data sets, if present, will be discussed as necessary during the resulting RD evaluations.

14. References

AECOM and Geosyntec. 2019. *PDI Evaluation Report – Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling*. Portland Harbor Superfund Site, Portland, Oregon. June 17.

U.S. Environmental Protection Agency (EPA). 1995. *Good Laboratory Practices in Principles and Guidance to Regulations for Ensuring Data Integrity in Automated Laboratory Operations*.

U.S. Environmental Protection Agency (EPA). 1996. *Test Methods for Evaluating Solid Waste, Physical and Chemical Methods, SW-846, 3rd Edition, Update III, Section 1*.

U.S. Environmental Protection Agency (EPA). 2001. *Requirements for Quality Assurance Project Plans, EPA QA/R-5.* March.

U.S. Environmental Protection Agency (EPA). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Parts 1-3, EPA/505/B-04/900A through 900*. March.

U.S. Environmental Protection Agency (EPA). 2006. *Guidance on Systematic Planning Using the Data Quality Objectives Process*. EPA QA/G-4. February.

U.S. Environmental Protection Agency (EPA). 2017a. *National Functional Guidelines for Inorganic Superfund Methods Data Review*. January.

U.S. Environmental Protection Agency (EPA). 2017b. *National Functional Guidelines for Organic Superfund Methods Data Review.* January.

U.S. Environmental Protection Agency (EPA). 2017c. *Portland Harbor Superfund Site Sampling Plan for Pre-Remedial Design, Baseline, and Long-Term Monitoring – Revised Working Draft.* June 6.

U.S. Environmental Protection Agency (EPA). 2020. *Program Data Management Plan, Portland Harbor Remedial Design Investigation Portland Harbor Superfund Site*. Region 10. August.

U.S. Environmental Protection Agency (EPA). 2020. *Remedial Design Guidelines and Considerations – Portland Harbor Superfund Site.* Region 10. Seattle, Washington. February.

Attachment 1
Lab Standard Operating Procedures

OCP Instrumental Method - HRMS

Document Authorization

Authorized By: _____ Date: _____
Quality Authorization

Authorized By: _____ Date: _____
Technical Authorization

Implementation Authorization

Authorized By: _____ Effective Date: _____
Laboratory Manager

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1.0 SCOPE AND APPLICATION

1.1 Scope

This method provides procedures for the instrumental detection and quantitative measurement of organochlorine pesticides (OCP) and related chlorinated compounds via isotope dilution and internal standard high resolution GC/MS. For preparation procedures refer to 'BU-TP-2103 OCP & Toxaphene Prep'.

This method covers the ultra trace analysis (ppq to ppt) of extracts prepared from most standard environmental samples including matrices such as air (stack and ambient), soils, sediments, other solids, potable water, non-potable water, tissues, and serums. This is based upon the following reference method:

US EPA Office of Water, Method 1699

The test codes that this method applies to are:

OCPEST-1699-HRMS-BU

1.2 Parameters and Limits of Reporting

The following specific organochlorine compounds can be determined by this method:

Parameter	LOR Solid (45) ng/g	LOR Tissue (20) ng/g	Synonym	CAS #
Hexachlorobutadiene	2.0	2.0		87-68-3
1,2,4,5-Tetrachlorobenzene	2.0	2.0		95-94-3
1,2,3,4-Tetrachlorobenzene	2.0	2.0		634-66-2
Pentachlorobenzene	5.0	5.0		608-93-5
Hexachlorobenzene	5.0	5.0		118-74-1
3,4,5,6-Tetrachloroveratrole	1.0	1.0		944-61-6
Pentachloroanisole	1.0	1.0		1825-21-4
α-Hexachlorocyclohexane	5.0	1.0	alpha-BHC	319-84-6
β-Hexachlorocyclohexane	5.0	1.0	beta-BHC	319-85-7
γ-Hexachlorocyclohexane	5.0	1.0	gamma-BHC, Lindane	58-89-9
δ-Hexachlorocyclohexane	5.0	1.0	delta-BHC	319-86-8
Pentachloronitrobenzene	1.0	1.0		82-68-8
Heptachlor	1.0	1.0		76-44-8
4,4'-DDNU	1.0	1.0	1,1-Bis(4-chlorophenyl)ethene	2642-81-1
Aldrin	1.0	1.0		309-00-2
Isodrin	1.0	1.0		465-73-6
Chlorpyrifos	1.0	5.0	Dursbane	2921-88-2
Dacthal	1.0	1.0		1861-32-1
Octachlorostyrene	1.0	1.0		29082-74-4
Heptachlor Epoxide B	1.0	1.0		28044-83-9
Heptachlor Epoxide A	1.0	1.0		1024-57-3
Oxychlordane	1.0	1.0		27304-13-8
trans-Chlordane	1.0	1.0	γ-Chlordane	5103-74-2
4,4'-DDMU	1.0	1.0	2-Chloro-1,1-bis(4-chlorophenyl)-ethene	1022-22-6
2,4'-DDE	1.0	1.0	o,p'-DDE	3424-82-6
cis-Chlordane	1.0	1.0	α-Chlordane	5103-71-9

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trans-nonachlor	1.0	1.0		39765-80-5
Endosulphan I	1.0	1.0	Thiodan sulphate I, alpha endosulfan	958-98-8
Dieldrin	1.0	1.0		60-57-1
4,4'-DDE	1.0	1.0	p,p'-DDE	72-55-9
2,4'-DDD	1.0	1.0	o,p'- DDD	53-19-0
Endrin	1.0	1.0		72-20-8
Toxaphene Congener Parlar-26	1.0	1.0	Parlar 26	142534-71-2
Endosulphan II	1.0	1.0	Thiodan sulphate II, beta endosulfan	33213-65-9
cis-Nonachlor	1.0	1.0		5103-73-1
4,4'-DDD	1.0	1.0	p,p'- DDD	72-54-8
2,4'-DDT	1.0	1.0	o,p'- DDT	789-02-6
Endosulphan Sulfate	1.0	1.0		1031-07-8
Endrin Aldehyde	1.0	1.0		7421-93-4
4,4'-DDT	1.0	1.0	p,p'- DDT	50-29-3
Endrin Ketone	1.0	1.0		53494-70-5
Methoxychlor	10.0	10.0	DMDT	72-43-5
Mirex	1.0	1.0		2385-85-5
Dicofol	100.0	100.0		115-32-2
Toxaphene Congener Parlar-50	1.0	1.0	Parlar 50	66860-80-8
Toxaphene Congener Parlar-62	1.0	1.0	Parlar 62	154159-06-5

These reflect EVOLIMS posted reporting limits based upon NA-SP-0102 Method Revalidation and MU - Chemistry.

The most common analyte lists and Limits of Reporting for ALS Canada labs are listed. All Limits of Reporting (LOR) are subject to validation by each lab. Other lists and LORs may be offered where validated.

Method detection limits (MDLs) are determined via 40CFR Part 136 Appendix B. These MDL are located within the Method Validation Files and in control of the Quality Scientist.

For OCP analyses as defined by this method, the MDLs are not normally used to define the reporting limits. The most common reporting approach is to report each congener or isomer group to 2.5:1 signal to noise (an estimated detection limit; EDL) as described in OSW SW846 Method 8290A.

1.3 Method Performance Data and Measurement Uncertainty

Refer to NA-SP-0100 Initial Method Validation – Chemistry and NA-SP-0102 Method Revalidation and MU – Chemistry for policies on determination and storage of method performance data.

2.0 PRINCIPLE OF METHOD

Extracts are analyzed via HRMS at resolution greater than 1 in 8000 and quantified via isotope dilution technique for analytes that have an exact isotopically labelled analogue, and internal standard technique for analytes which do not have a labelled analogue.

3.0 DEFINITIONS

Refer to NA-SP-0001 Definitions of Key Terms for definitions of common terms.

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For isotope dilution analytical methods, there is inconsistent use of terms within the literature and reference methods. This inconsistency is avoided herein by applying terms that are defined by the timing of their addition rather than by any other the use in the calculation of the data.

Extraction Standards: are the labelled analytes added just prior to extraction. For the data calculations, these are true internal standards - used for determining analyte concentration.

Clean-up Standard(s): is (are) the labelled analyte(s) added just prior to clean-up. Recoveries of the clean-up standard(s) provide an indication on the losses that occur during the extract clean-up and preparation for instrumental analysis.

Injection Standards: are the labelled analytes added just prior to instrumental analysis. These are used as an internal standard reference to determine the recoveries of the extraction and clean-up standards.

Sampling Standard: (also called field spikes) are the labelled analytes added, where applicable, prior to sampling onto sorbant media used for analyte capture during the sampling event.

4.0 INTERFERENCES

Prudent application of appropriate clean-ups generally provides extracts that are interference free. Occasionally, despite reasonable efforts during clean-up interferences can be observed.

5.0 SAMPLE HANDLING AND STORAGE

This method is for the analysis of extracts. For information on sample handling prior to extraction, please refer to the extract preparation method 'BU-TP-2103 OCP & Toxaphene Prep'.

Organic solutions of OCP are assumed to be as stable as PCB and PCDD/PCDF homologues. Indeed, these are very stable and not generally subject to degradation when protected from light and loss of solvent. Recommended hold times of samples and extracts are variable dependent upon the method. Below are the hold times specified for samples as defined by the individual related methods.

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Parameter(s) and Matrix: All Parameters - Water	
Container	1 litre amber bottle with Teflon lined lid pre-cleaned, Reference: EPA 1699 Section 6.1.1.
Preservative	If residual chlorine is present, add 80 mg sodium thiosulfate per liter of water. EPA 1699 Section 8.0
Min. Sample Amount	1 liter
Sample Pre-treatment	N/A
Lab Storage Conditions	>0 - 6˚C Ref: US EPA 40CFR Part 136 2012 If sample will be frozen allow room for expansion. Ref: EPA 1699 Section 8.0
Maximum Sample Holding Time (Provinces / Reference)	7 days from collection EPA 1699 Section 8.5.1
Maximum Extract / Digest Holding Time (Provinces / References)	40 days Reference: EPA 1699 Section 8.5.1

Parameter(s) and Matrix: All Parameters - Tissue	
Container	250 mL amber jar with Teflon lined lid Reference: EPA1699 Section 6.1.1
Preservative	N/A
Min. Sample Amount	20g
Sample Pre-treatment	N/A
Lab Storage Conditions	Freeze tissue samples upon receipt at the laboratory and maintain in the dark at less than -10˚C until prepared. Maintain unused sample in the dark at less than -10˚C. Reference EPA 1699 Section 8.4.3 Extracts are kept in fridge at less than 10˚C this is a variance from EPA 1699 Section 8.4.4 backed by historical data.
Maximum Sample Holding Time (Provinces / Reference)	1 year Reference EPA 1699 Section 8.5.2
Maximum Extract / Digest Holding Time (Provinces / References)	1 year Reference: EPA 1699 Section 8.5.2

Parameter(s) and Matrix: All Parameters – Soil	
Container	250 mL amber jar with Teflon lined lid Reference: EPA1699 Section 6.1.1
Preservative	N/A
Min. Sample Amount	20g
Sample Pre-treatment	N/A
Lab Storage Conditions	Store solid, semi-solid, oily, and mixed-phase samples in the dark at less than -10˚C Reference EPA 1699 Section 8.3.2 Extracts are kept in fridge at less than 10˚C this is a variance from EPA 1699 Section 8.4.4 backed by historical data.
Maximum Sample Holding Time (Provinces / References)	1 year Reference EPA 1699 Section 8.5.2
Maximum Extract / Digest	1 year

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Parameter(s) and Matrix: All Parameters – Soil	
Holding Time (Provinces / References)	Reference: EPA 1699 Section 8.5.2

Parameter(s) and Matrix: All parameters - Serum	
Container	Sample containers must be able to withstand freeze/thaw cycles and temperatures down to -80˚C without compromise to the samples. Options include: (a) amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE) or (b) polypropylene vials.
Preservative	None
Min. Sample Amount	Minimum sample amount required to meet the contract required RL
Sample Pre-treatment	None
Lab Storage Conditions	In the dark at <-70˚C for long term storage In the dark at <-10˚C for storage of up to 1 year Reference: Stats/Health Canada Standing Offer 4600000355
Maximum Sample Holding Time	Indefinite at <-70˚C [likely several years]; 1 year at <-10˚C. Reference: Stats/Health Canada Standing Offer 4600000355
Storage of Leftover Sample	In the dark at <-70˚C for long term storage In the dark at <-10˚C for storage of up to 1 year Reference: Stats/Health Canada Standing Offer 4600000355
Sample Disposal	Treatment with bleach (hypochlorite) and then rinsed down the sink

5.1 Sample Handling and Transportation

Sample collection is not conducted by the laboratory.

Recommended bottles for storage are pre-cleaned amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE). Solid tissues such as whole fish can be wrapped in solvent-cleaned aluminium foil.

All water sample bottles destined for OCP analysis are not adjusted in pH.

6.0 EQUIPMENT AND SUPPLIES

6.1 Primary Equipment

6.1.1 Autosamplers

Capable of dependable automated injection of liquid extracts down to 1uL, including pre- and post-injection automated syringe rinsing. Examples:

- CTC PAL GC-xt Auto-Sampler or functionally equivalent or superior, as provided with, or upgraded on, Waters Autospec HRMS systems.

6.1.2 Gas Chromatograph

Capable of temperature programming with isothermal hold, and temperature controlled injection capability. Electronic Pressure Control recommended but not essential. Examples:

- Agilent 7890B GC or functionally equivalent or superior, as provided with, or upgraded on, Waters Autospec HRMS systems, fitted with a Gerstel CIS-4

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injector or equivalent.

6.1.3 High Resolution Mass Spectrometer

Capable of repetitive and selective monitoring of at least 12 exact m/z's at high resolution (8,000) during a period of approximately one second. Micromass (now Waters) Autospec Premier series HRMS or functionally equivalent or superior.

6.1.4 GC column

HP5ms or equivalent.

6.1.4.1 Column specifications
The column must uniquely resolve 4,4'-DDD from 2,4'-DDT. Unique resolution means a valley height less than 60 percent of the shorter of the two peaks in the daily calibration. The column must be serviced or replaced when the above criterion is not met.

6.1.4.2 Suggested column—30±3-m long × 0.25±0.02-mm ID; 0.25-mm film HP5ms (or equivalent) with 1-2m of deactived silica guard column. This column is capable of meeting the requirements in Section 6.1.4.1.
Supplier: Agilent, Supplier #19091S-433; MEOL # 4320190

6.2 Support Equipment

1. Rotovap
2. Centrifuge
3. N-Evap

For glassware cleaning, please refer to BU-WI-3000 Organic Glassware/Equipment Cleaning, Proofing, and Maintenance

6.3 Software

Waters MassLynx for instrument control and data acquisition. Waters TargetLynx for data quantification and processing.

6.4 Supplies

6.4.1 Sample Prep Supplies

Refer to BU-TP-2103 OCP & Toxaphene prep

6.4.2 Instrument Supplies

1. Baffled liner, Gerstel p/n 6492-U, MEOL# 3290896
2. Syringe: 23 gauge, Chromatographic Specialties, MEOL#4320721

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3. HP5ms 30mx0.25mmx0.25µm column, Agilent, MEOL# 4320190
4. Siltek deactivated guard column 10mx0.25mm, Chromatographic Specialties, MEOL# 3080542

6.4.3 Standard Preparation Supplies

1. 10 µL-5000 µL syringes, Hamilton, MEOL # for various syringes can be found in G:\Enviro\Purchasing\Organic Prep\Approved Supplies.xls (Syringes Tab)
2. 2 mL Robovials (MEOL# 4001399) and Lids (MEOL# 4000796) with septa (MEOL# 3300552)
3. 5" pipettes (MEOL# 4430061)
4. 15 mL Centrifuge tubes (C-tubes) MEOL# 4000187
5. 50 mL Centrifuge tubes (C-tubes) MEOL# 4000202
6. 7 mL Graduated screw cap vials with solid PTFE lined caps MEOL# 4000687 (vial), MEOL# 4010593 (cap)
7. 22 mL Graduated screw cap vials with solid PTFE lined caps MEOL# 4000738 (vial), MEOL# 4010609 (cap)
8. Class "A" volumetric flasks of appropriate size

7.0 REAGENTS AND STANDARDS

The following reagents and standards have been shown to meet the performance specifications of this test method. Alternate sources may be substituted where equivalent performance and suitability has been verified. Refer to site-specific reagent and standard preparation logs and analytical records to identify the reagents and standards in current use at each lab. Preparation procedures below are for suggested nominal batch sizes of standard materials. These batches may be scaled up or down as needed to adjust for production demands.

Refer to BU-SP-0250 Control of Reagents and Reference Materials.

7.1 Reagents

All solvents must be Analytical Grade or better.

1. Toluene, Glass Distilled, Stored at ambient temperature. Stored in flammable materials storage cabinet. Expiry date: 1 year following date of receipt. Supplier: Caledon, Supplier # 9201-2-40; MEOL # 6010007

2. Perfluorokerosene (PFK), Either high-boiling or low-boiling may be used. Stored at ambient temperature. Expiry date: 5 years following date of receipt. Supplier: Cambridge Isotope Lab, Supplier # PFK-HIGH-1, MEOL # 5005748

3. Helium, UHP, Expiry date: indefinite. Supplier: Praxair, Supplier # HE 5.0UH-T; MEOL # n/a (ordered on the Praxair website)

7.2 Standards

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All standards are stored in the fridge in screw cap vials with PTFE lids. Unless otherwise indicated, the default expiry date is 1 year, or longer if revalidation is done. Refer to 'BU-WI-3002 Standards Prep, Spiking and Witnessing' for required protocols for standard preparation.

7.2.1 Extraction Standards

7.2.1.1 Extraction Standard Supplier Information

Expiry dates are stated on the supplier certificate of analysis.

7.2.1.1.1 Individual Extraction Standard Stock Solutions

Compound	Supplier	Catalogue #	MEOL #
13C6-Pentachorobenzene	Wellington	MCBZ-12345	5003929
13C6-Hexachorobenzene	CIL	CLM-351-1.2	5003340
13C10-Heptachlor	CIL	CLM-4759-1.2	5004212
13C10-Oxychlordane	CIL	CLM-4729-1.2	5004215
13C10-trans-Nonachlor	CIL	CLM-4735-1.2	5004214
13C12-Dieldrin	CIL	CLM-4726-1.2	5004210
13C12-Endrin	CIL	CLM-4782-1.2	5004211
13C9-Endosulfan II	CIL	CLM-6026-1.2	5006113
13C12-4,4'-DDD	CIL	CLM-7100-1.2	5006111
13C10-Mirex	CIL	CLM-4813-1.2	5005594
13C6-delta-BHC	CIL	CLM-3648-1.2	5008047
13C6-beta-BHC	CIL	CLM-3623-1.2	5008046
13C6-alpha-BHC	CIL	CLM-2482-1.2	5008045
13C6-gamma-BHC	CIL	CLM-1282-1.2	5008048
13C12-4,4'-DDT	CIL	CLM-1281-1.2	5001220
13C12-4,4'-DDE	CIL	CLM-1627-1.2	5008042
13C12-2,4'-DDE	CIL	CLM-4693-1.2	5008043
13C12-Methoxychlor	CIL	CLM-4683-1.2	5008044

7.2.1.1.2 Extraction Working Standard Solution Preparation (HROCP-ES#2-xxx)

Standard	Concentration (ng/mL)	Amt Added (µl)	Final Conc. (ng/mL)
MCBZ-12345	100000	250	2500
CLM-351-1.2	100000	250	2500
CLM-4759-1.2	100000	500	5000
CLM-4729-1.2	100000	500	5000
CLM-4735-1.2	100000	500	5000
CLM-4726-1.2	100000	250	2500
CLM-4782-1.2	100000	500	5000
CLM-6026-1.2	100000	500	5000
CLM-7100-1.2	100000	250	2500
CLM-4813-1.2	100000	250	2500
CLM-3648-1.2	100000	500	5000
CLM-3623-1.2	50000	1000	5000
CLM-2482-1.2	100000	500	5000

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CLM-1282-1.2	100000	500	5000
CLM-1281-1.2	100000	250	2500
CLM-1627-1.2	100000	250	2500
CLM-4693-1.2	100000	250	2500
CLM-4683-1.2	100000	250	2500

1. Using syringes of appropriate size, add the amount shown of each compound to a clean 10 mL Class A volumetric flask.
2. Add toluene to the 10 mL line of the flask. The final concentration for each target is listed.
3. Aliquot into labeled robovials.
4. Name the standard HROCP-ES#2-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.1.1.5 Low Final Volume Extraction Working Standard Solution Preparation (HROCP-ES2_LFV-xxx)

Standard	Concentration (ng/mL)	Amt Added (µl)	Final Conc. (ng/mL)
HROCP-ES#2-xxx	Per table7.2.1.1.2	500	125/250

1. Using a syringe of appropriate size, add the amount shown for HROCP-ES#2-xxx to a clean 10 mL Class A volumetric flask.
2. Add toluene to the 10 mL line of the flask. The final concentration for each target is listed.
3. Aliquot into labeled robovials.
4. Name the standard HROCP-ES2_LFV-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.2 Cleanup Standards

7.2.2.1 Cleanup Standard Supplier Information

Expiry dates are stated on the supplier certificate of analysis.

7.2.2.1.1 Individual Cleanup Standard Stock Solutions

Compound	Supplier	Catalogue #	MEOL #
13C12-PCB-133	Wellington	MBP-133	5004976

7.2.2.1.2 Cleanup Working Standard Solution Preparation (HROCP-GPC#1-xxx)

Standard	Concentration (ng/mL)	Amt Added (µl)	Final Conc. (ng/mL)
MBP-133	50000	280	5000

1. Using a syringe, add 2520 µl of toluene to a c-tube.
2. Using a syringe of appropriate size, add the amount shown of MBP-133 to the toluene.

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3. The final volume should be 2.8 mL and the final concentration is listed.
4. Aliquot into labeled robovials.
5. Name the standard HROCP-GPC#1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.3 Injection Standards

7.2.3.1 Injection Standard Supplier Information

Expiry dates are stated on the supplier certificate of analysis.

7.2.3.2 Individual Injection Standard Stock Solutions

Compound	Supplier	Catalogue #	MEOL #
13C12-PCB-9	Wellington	MBP-9	5005092
13C12-PCB-52	Wellington	MBP-52	5004572
13C12-PCB-101	Wellington	MBP-101	5004571

7.2.3.3 Injection Working Standard Solution Preparation (HROCP-IS#1-xxx)

Standard	Concentration (ng/mL)	Amt Added (µl)	Final Conc. (ng/mL)
MBP-9	50000	500	2500
MBP-52	50000	500	2500
MBP-101	50000	500	2500

1. Using syringes of appropriate size, add the amount shown of each compound to a clean 10 mL Class A volumetric flask.
2. Add toluene to the 10 mL line of the flask. The final concentration for each target is listed.
3. Aliquot into labeled robovials.
4. Name the standard HROCP-IS#1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.3.4 Low Final Volume Injection Working Standard Solution Preparation (HROCP-IS_LFV-xxx)

Standard	Concentration (ng/mL)	Amt Added (µl)	Final Conc. (ng/mL)
HROCP-IS#1-xxx	2500	400	500

1. Using a syringe, add 1200 µl of toluene to a c-tube.
2. Using a syringe of appropriate size, add the amount shown for HROCP-IS#1-xxx to the toluene.
3. The final volume should be 2 mL and the final concentration for each extraction standard is listed.
4. Aliquot into labeled robovials.
5. Name the standard HROCP-IS_LFV-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

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7.2.4 Native Standards

7.2.4.1 Native Standard Supplier Information

Expiry dates are stated on the supplier certificate of analysis.

7.2.4.2 Individual Neat Native Standards

Neat Compound	Supplier	Catalogue #	MEOL #	Individual Stock Solution
1,2,4,5-Tetrachlorobenzene	Supelco	442219	5006632	1,2,4,5-Tetrachlorobenzene-S$1-xxx
1,2,3,4-Tetrachlorobenzene	Sigma Aldrich	46014-100MG	5006803	1,2,3,4-Tetrachlorobenzene-S$1-xxx
Pentachlorobenzene	Sigma Aldrich	35886	n/a	Pentachlorobenzene-S$2-xxx
3,4,5,6-Tetrachloroveratrole	Orchid Cellmark	C011	n/a	3,4,5,6-Tetrachloroveratrole-S$1-xxx
Endosulphan Sulfate	Chem Service	N-11851-50MG	5004677	Endosulphan Sulfate-S$1-xxx
Dicofol	Fluka	36677-100MG-R	n/a	Dicofol-S$1-xxx
4,4'-DDNU	AccuStandard	P-1054N	n/a	4,4'-DDNU-S$1-xxx
4,4'-DDMU	AccuStandard	P-424N	n/a	4,4'-DDMU-S$1-xxx

Individual stock solutions are made by weighing out approximately 5mg (0.005g) of neat standards and diluting with toluene to a volume that gives a concentration of 1000ug/mL (approx 5mL). Examples of the standard names are shown in the table above where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.4.3 Individual Native Stock Solutions

Compound	Supplier	Catalogue #	MEOL #
Octachlorostyrene	AccuStandard	PC-001S	5003335
Pentachloronitrobenzene	AccuStandard	AS-E0156	5005363
Hexachlorobutadiene	AccuStandard	M-502-36	n/a
trans-nonachlor	AccuStandard	P-184S	5004031
Dacthal	AccuStandard	P-196S	5005364
Pentachloroanisole	AccuStandard	P-199S	n/a
cis-Nonachlor	AccuStandard	P-297S	5004039
Chlorpyrifos	AccuStandard	M-8140-03	5005498
Endrin Aldehyde	AccuStandard	P-046S	6031557
Endrin Ketone	AccuStandard	P-146S	5006802

7.2.4.4 Native Stock Standard Solution Preparation (HROCP-NS$1-xxx)

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Standard	Concentration (ng/mL)	Amt Added (µl)	Final Conc. (ng/mL)
PC-001S	35000	571.4	8000
AS-E0156	1000000	20.0	8000
M-502-36	200000	100.0	8000
P-184S	100000	200.0	8000
P-196S	100000	200.0	8000
P-199S	100000	200.0	8000
P-297S	100000	200.0	8000
1,2,4,5-Tetrachlorobenzene-S$1-xxx	1000000	20.0	8000
1,2,3,4-Tetrachlorobenzene-S$1-xxx	1000000	20.0	8000
Pentachlorobenzene	1000000	20.0	8000
3,4,5,6-Tetrachloroveratrole-S$1-xxx	1000000	20.0	8000
Endosulphan Sulfate-S$1-xxx	1000000	20.0	8000
Dicofol-S$1-xxx	1000000	200.0	80000
4,4'-DDNU-S$1-xxx	1000000	20.0	8000
4,4'-DDMU-S$1-xxx	1000000	20.0	8000
M-8140-03	1000000	100.0	40000
P-046S	100000	200.0	8000
P-146S	100000	200.0	8000

1. Using a syringe, add 168.8 µL of toluene to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each compound to the toluene.
3. The final volume should be 2.5 mL and the final concentration for each target is listed.
4. Aliquot into labeled robovials.
5. Name the standard HROCP-NS$1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.4.5 Individual Native Stock Solutions

Compound	Supplier	Catalogue #	MEOL #
Parlar-26	CIL	ULM-7828-1.2	5004216
Parlar-50	CIL	ULM-7829-S	5004217
Parlar-62	CIL	ULM-7830-1.2	5004218

7.2.4.6 AccuStandard Native Standard Mix Catalogue #AE-00010, MEOL# 5004038

Compound	Concentration (ng/mL)
Hexachlorobenzene	10000
α-Hexachlorocyclohexane	10000
β-Hexachlorocyclohexane	10000
γ-Hexachlorocyclohexane	10000
d-Hexachlorocyclohexane	10000
Heptachlor	10000
Aldrin	10000
Isodrin	10000
Heptachlor Epoxide B	10000
Heptachlor Epoxide A	10000

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Oxychlordane	10000
trans-Chlordane	10000
2,4'-DDE	10000
cis-Chlordane	10000
Endosulphan I	10000
Dieldrin	10000
4,4'-DDE	10000
2,4'-DDD	10000
Endrin	10000
Endosulphan II	10000
4,4'-DDD	10000
2,4'-DDT	10000
4,4'-DDT	10000
Methoxychlor	10000
Mirex	10000

7.2.4.7 Native Working Standard Solution Preparation (HROCP-NS#1-xxx)

Standard	Concentration (ng/mL)	Amt Added (µl)	Final Conc. (ng/mL)
AE-0010	10000	350	1250
HROCP-NS$1-xxx	8000-80000	437.5	1250-12500
ULM-7828-1.2	10000	350	1250
ULM-7829-S	10000	350	1250
ULM-7830-1.2	10000	350	1250

1. Using a syringe, add 962.5 µl of toluene to a c-tube.
2. Using syringes of appropriate size, add the amount shown of each compound to the toluene.
3. The final volume should be 2.8 mL and the final concentration for each target is listed.
4. Aliquot into labeled robovials.
5. Name the standard HROCP-NS#1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.4.8 Low Final Volume Native Working Standard Solution Preparation (HROCP-NS_LFV-xxx)

Standard	Concentration (ng/mL)	Amt Added (µl)	Final Conc. (ng/mL)
HROCP-NS#1-xxx	1250-12500	150	62.5-625

1. Using a syringe, add 2850 µl of toluene to a c-tube.
2. Using a syringe of appropriate size, add the amount shown for HROCP-NS#1-xxxto the toluene.
3. The final volume should be 3.0 mL and the final concentration for each extraction standard is listed.
4. Aliquot into labeled robovials.
5. Name the standard HROCP-NS_LFV-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

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7.2.4.9 Field/Lab Surrogate Standard

Some clients require their field and lab surrogate standards to be spiked in before extraction occurs. A standard is made in house for use in the calibration set.

7.2.4.10 Field/Lab Surrogate Standard Supplier Information

Expiry dates are stated on the supplier certificate of analysis.

7.2.4.11 Individual Neat Field/Lab Surrogate Standards

Neat Compound	Supplier	Catalogue #	MEOL #	Individual Stock Solution
1,3-Dibromobenzene	Sigma-Aldrich	194395-5G	n/a	13-Dibromobenzene-S$1-xxx
1,3,5-Tribromobenzene	Sigma-Aldrich	140066-25G	n/a	135-Tribromobenzene-S$1-xxx
1,2,4,5-Tetrabromobenzene	Sigma-Aldrich	278343-25G	n/a	1245-Tetrabromobenzene-S$1-xxx

Individual stock solutions are made by weighing out approximately 5mg (0.005g) of neat standards and diluting with toluene to a volume that gives a concentration of 1000ug/mL (approx 5mL). Examples of the standard names are shown in the table above where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.4.12 Field/Lab Surrogate Stock Standard Solution Preparation (HROCP-EC_FS_LS$1-xxx)

Standard	Concentration (ng/mL)	Amt Added (µl)	Final Conc. (ng/mL)
13-Dibromobenzene-S$1-xxx	1000000	1.5	1000
135-Tribromobenzene-S$1-xxx	1000000	1.5	1000
1245-Tetrabromobenzene-S$1-xxx	1000000	1.5	1000

1. Using a syringe, add 1495.5 µL of toluene to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each compound to the toluene.
3. The final volume should be 1.5 mL and the final concentration for each target is listed.
4. Name the standard HROCP-EC_FS_LS$1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.5 Calibration Standards

7.2.5.1 Calibration Standard Supplier Information

All stock solutions for the calibration standards are highlighted in sections 7.2.1 to 7.2.4.

7.2.5.2 Calibration CS1 Standard Preparation (HROCP-CS#1-xxx)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)

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Native	HROCP-NS#1	2.4	2-20
Extraction	HROCP-ES#2	60	100-200
Injection	HROCP-IS#1	60	100
Cleanup	HROCP-GPC#1	30	100
Field/Lab Surrogate	HROCP-EC_LS_FS$1	30	20

1. Using a syringe, add 1317.6 µl of toluene to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard source to the toluene.
3. The final volume should be 1.5 mL and the final concentration is listed.
4. Name the standard HROCP-CS#1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.5.3 Calibration CS2 Standard Preparation (HROCP-CS#2-xxx)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	HROCP-NS#1	9	7.5-75
Extraction	HROCP-ES#2	60	100-200
Injection	HROCP-IS#1	60	100
Cleanup	HROCP-GPC#1	30	100
Field/Lab Surrogate	HROCP-EC_LS_FS$1	30	20

1. Using a syringe, add 1311µl of toluene to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard source to the toluene.
3. The final volume should be 1.5 mL and the final concentration is listed.
4. Name the standard HROCP-CS#2-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.5.4 Calibration CS3 Standard Preparation (HROCP-CS#3-xxx)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	HROCP-NS#1	24	20-200
Extraction	HROCP-ES#2	60	100-200
Injection	HROCP-IS#1	60	100
Cleanup	HROCP-GPC#1	30	100
Field/Lab Surrogate	HROCP-EC_LS_FS$1	30	20

1. Using a syringe, add 1296 µl of toluene to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard source to the toluene.
3. The final volume should be 1.5 mL and the final concentration is listed.
4. Name the standard HROCP-CS#3-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

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7.2.5.5 Calibration CS4 Standard Preparation (HROCP-CS#4-xxx)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	HROCP-NS#1	60	50-500
Extraction	HROCP-ES#2	60	100-200
Injection	HROCP-IS#1	60	100
Cleanup	HROCP-GPC#1	30	100
Field/Lab Surrogate	HROCP-EC_LS_FS$1	30	20

1. Using a syringe, add 1260 µl of toluene to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard source to the toluene.
3. The final volume should be 1.5 mL and the final concentration is listed.
4. Name the standard HROCP-CS#4-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.5.6 Calibration CS5 Standard Preparation (HROCP-CS#5-xxx)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	HROCP-NS#1	180	150-1500
Extraction	HROCP-ES#2	60	100-200
Injection	HROCP-IS#1	60	100
Cleanup	HROCP-GPC#1	30	100
Field/Lab Surrogate	HROCP-EC_LS_FS$1	30	20

1. Using a syringe, add 1140 µl of toluene to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard source to the toluene.
3. The final volume should be 1.5 mL and the final concentration is listed.
4. Name the standard HROCP-CS#5-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.5.7 Calibration CS6 Standard Preparation (HROCP-CS#6-xxx)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	AE-00010	60	400
	HROCP-NS$1	75	400-4000
	ULM-7828-S	60	400
	ULM-7829-S	60	400
	ULM-7830-S	60	400
Extraction	HROCP-ES#2	60	100-200
Injection	HROCP-IS#1	60	100
Cleanup	HROCP-GPC#1	30	100
Field/Lab Surrogate	HROCP-EC_LS_FS$1	30	20

1. Using a syringe, add 1005 µl of toluene to a labeled robovial.

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2. Using syringes of appropriate size, add the amount shown of each standard source to the toluene.
3. The final volume should be 1.5 mL and the final concentration is listed.
4. Name the standard HROCP-CS#6-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.5.8 Calibration CS7 Standard Preparation (HROCP-CS#7-xxx)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	AE-00010	180	1200
	HROCP-NS$1	225	1200-12000
	ULM-7828-S	180	1200
	ULM-7829-S	180	1200
	ULM-7830-S	180	1200
Extraction	HROCP-ES#2	60	100-200
Injection	HROCP-IS#1	60	100
Cleanup	HROCP-GPC#1	30	100
Field/Lab Surrogate	HROCP-EC_LS_FS$1	30	20

1. Using a syringe, add 375 µl of toluene to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard source to the toluene.
3. The final volume should be 1.5 mL and the final concentration is listed.
4. Name the standard HROCP-CS#7-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.6 Reference Standards

7.2.6.1 Reference Standard Supplier Information

Expiry dates are stated on the supplier certificate of analysis.

7.2.6.2 Individual Neat Reference Standards

Neat Compound	Supplier	Catalogue #	MEOL #	Individual Stock Solution
Hexachlorobenzene	Sigma Aldrich	45522-250MG	5007699	Hexachlorobenzene-S$1-xxx

Individual stock solutions are made by weighing out approximately 5mg (0.005g) of neat standards and diluting with toluene to a volume that gives a concentration of 1000ug/mL (approx 5mL). Examples of the standard names are shown in the table above where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.6.3 Individual Reference Standard Stock Solutions

Compound	Supplier	Catalogue #	MEOL #
trans-nonachlor	CIL	ULM-7229-1.2	5005530
cis-nonachlor	CIL	ULM-7445-1.2	5005531

7.2.6.4 Supelco EPA CLP Organochlorine Pesticide Mix # CRM47426, MEOL# 5002039

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Compound	Concentration (ng/mL)
Aldrin	2000000
Alpha-BHC	2000000
Alpha chlordane	2000000
Beta-BHC	2000000
Delta-BHC	2000000
Dieldrin	2000000
Endosulfan I (Alpha)	2000000
Endosulfan II (Beta)	2000000
Endosulfan Sulfate	2000000
Endrin	2000000
Endrin Aldehyde	2000000
Endrin Ketone	2000000
Gamma-BHC	2000000
Gamma-Chlordane	2000000
Heptachlor	2000000
Heptachlor Epoxide Isomer B	2000000
Methoxychlor	2000000
4,4'-DDD	2000000
4,4'-DDE	2000000
4,4'-DDT	2000000

7.2.6.5 Native Stock Standard Solution Preparation (HROCP-NS$1-xxx)

Standard	Concentration (ng/mL)	Amt Added (µl)	Final Conc. (ng/mL)
CLP OC Pesticide Mix	2000000	4.5	6000
ULM-7229-1.2	100000	90.0	6000
ULM-7445-1.2	100000	90.0	6000
Hexachlorobenzene-S$1-xxx	1000000	9.0	6000

1. Using a syringe, add 1261.5 µL of toluene to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each compound (listed above) to the toluene.
3. The final volume should be 1.5 mL and the final concentration for each target is listed.

4. Name the standard HROCP-RS$1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.6.6 Reference Standard Preparation (HROCP-RS#1-xxx)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	HROCP-RS$1	37.5	150-750
Extraction	HROCP-ES#1	60	250
Injection	HROCP-IS#1	60	250
Cleanup	HROCP-GPC#1	30	100
Field/Lab Surrogate	HROCP_EC_FS_LS$1	30	20

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1. Using a syringe, add 1282.5 µl of toluene to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard source to the toluene.
3. The final volume should be 1.5 mL and the final concentration is listed.
4. Name the standard HROCP-RS#1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.7 Breakdown Standards

7.2.7.1 Breakdown Standard Supplier Information

Expiry dates are stated on the supplier certificate of analysis.

7.2.7.2 Individual Neat Native Standards

Neat Compound	Supplier	Catalogue #	MEOL #	Individual Stock Solution
Endrin	Chem Service	N-11854-100MG	5005297	Endrin-S$1-xxx

Individual stock solutions is made by weighing out approximately 5mg (0.005g) of neat standard and diluting with toluene to a volume that gives a concentration of 1000ug/mL (approx 5mL). Example of the standard names are shown in the table above where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.7.3 Breakdown Standard Preparation (HROCP-GC_BD#1-xxx)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	Endrin-S$1	3.6	1200
Extraction	13C12-44'-DDT	1.5	500
	CLM-4782-S	15	500
Injection	MBP-101	6.0	100

1. Using a syringe, add 2973.9 µl of toluene to a c-tube.
2. Using syringes of appropriate size, add the amount shown of each standard source to the toluene.
3. The final volume should be 3.0 mL and the final concentration is listed.
4. Aliquot into labeled robovials.
5. Name the standard HROCP-GC_BD#1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

8.0 QUALITY CONTROL

8.1 Method Quality Control

Maximum (Sample Preparation) Batch Size =	**20 client samples**

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Method QC	Min. Frequency	Data Quality Objective (DQO)
Method Blank (MB)	1 per batch	< Instrument response of lowest calibration standard applicable to the project (i.e. below CS1 or CS2 dependent upon the nominal response curve of an individual target)
Laboratory Control Sample (LCS)	1 per batch	See OPR in Table 1.
Reference Material (RM)	Upon client request	Project and RM specific
Matrix Spike (MS)	Upon client request	As per LCS provided spiking levels are at least 10-fold above the levels in the sample.
Matrix Spike Duplicate (MSD)	Upon client request	< 50% RSD; If the spiking levels are less than 10-fold above the native levels in the sample, then this criteria is not valid.
Sample Duplicate	Upon client request	< 50% RSD, where the analyte levels are within quantifiable range of the method
Procedural Blank (PB)	1 per batch*	Visual Assessment of Contamination

*If all samples from a batch require further cleanup, the QC samples must be included. If only selected samples require further cleanup, a procedural blank must be created which will receive the prescribed cleanup to ensure no contamination has been acquired through the additional steps.

8.2 Instrument Quality Control

Instrument QC	Min. Frequency	Min. Acceptance Criteria (units)
Initial calibration (IC)	As needed to re-establish calibration control and/or after major maintenance	• < 20% RSD or coefficient of variation (native targets that have their own corresponding labelled analogue) • <35% RSD or coefficient of variation (labelled analytes and native analytes that do not have a corresponding labelled analogue)
Calibration Verification Standard (CVS)	With each initial cal.	See ccv limits in Table 1
Continuing Calibration Verification (CCV)	Min. beginning of every 12 hour shift in which samples are run	• Ion abundance rations are within their respective theoretical limits (see Table 1) • All targets have a s/n ratio of at least 10:1 • The RT of each analyte is within 15 sec of that in the initial calibration • The % Diff is within the CCV limits (see Table 1)
Breakdown Standard (GC_BD)	Min. beginning of every 12 hour shift in which samples are run, suggested after every CCV	• Endrin and DDT breakdown is less than 20 %
LOR (or Detection Limit) check	Beginning of each initial calibration	s/n ≥ 10:1 on the daily ccv
Instrument Blank	Every analytical batch. Run before Method Blank.	Visually assessed to be free of carry-over contamination, < the response of the low calibration point for each target.
Chromatographic Performance	Every 12 hours	The CCV is also assessed for minimum chromatographic separation criteria (See section 6.1.4.1)
MS resolution (HRMS option only)	Start and end of each shift	8,000 throughout the mass range

Refer to Quality Control procedures for policies and guidance on data acceptance and qualification.

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9.0 Test Procedures

9.1 Sample Preparation

See BU-TP-2103 OCP & Toxaphene Prep.

9.2 Dilution Procedure

1) The target analyte concentrations in the sample extract must be within the calibrated range of the instrument. See BU-SP-0263 for Dilution policy and general procedures.

2) If a sample is diluted 100x or greater, then spike with ES and IS at the regular amount. The results will no longer be recovery corrected; therefore, a comment will need to be made on the report.

3) If the sample is in a microvial and the final volume will be 500 µL or less, then a measured amount of solvent (toluene, DCM, or hexane) is added directly to the uncapped microvial. Re-cap and vortex the sample

4) If a greater dilution is required or the sample is for more than one analyses, then vortex the sample. Remove the cap. Using a clean syringe, put an aliquot into a new, labeled microvial with solvent already added. Cap and vortex the new microvial.

5) For a sample in a robovial, the same steps are following with the following exception: the direction dilution can occur if the final volume is 2 mL or less.

NOTE: If a sample is suspected of being high level prior to analysis, an instrument blank is run immediately after it or the high level sample can be put at the end of the run.

9.3 Additional Cleanups & Reworks (Procedural Blank Generation)

If a sample requires additional work in the lab, a procedural blank is generated at the instrument, which accompanies the samples and the rework document as it goes back out into the prep lab. The procedure below outlines how to generate a procedural blank.

1) Refer to the sample batch sheet to identify the injection and extraction standard types and amounts used in generating the batch.
2) Retrieve the corresponding injection and extraction standards from the standards fridge.
3) A new vial is obtained, and labelled as PB-xxx (where xxx is a sequential, unique numerical identifier)
4) Using proper syringe techniques, spike the appropriate amount of each standard into the procedural blank vial. If in a robovial, add solvent before spiking the standards.
5) Submit the procedural blank vial, alongside the sample(s) that require additional work, to the prep lab.

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9.4 HRMS Operation and Conditions:

The operation of the HRMS instrument is described in Work Instruction 'BU-WI-3007 GCMS Operation'. Specific MS conditions and parameters may vary slightly for each instrument, but nominal operating parameters can be found in BU-WI-3214 OCP (Parlar) HRMS-Autospec Premier.

The exact masses monitored, and suggested acquisition descriptor parameters are shown in Table 3. Note that depending on the instrument used, alternate or additional PFK masses may be used. Also, the number of scan descriptors and which masses appear in each descriptor may vary depending on the scan and mass calibration capabilities and methodologies of a given instrument. The exact MS acquisition methods used are referenced in BU-WI-3214 OCP (Parlar) HRMS-Autospec Premier.

9.5 GC Conditions:

GC Temperature profiles and conditions must be designed so that the performance meets all method RT, RRT and chromatographic resolution criteria as required by the applicable reference method (see Section 8.0). GC operating conditions are optimized on each HRGC/HRMS system. Once optimized, the same conditions must be used for analysis of all calibration standards, Quality Control Samples, and samples. Nominal GC operating conditions can be found in BU-WI-3214 OCP (Parlar) HRMS-Autospec Premier.
For the analysis of extracts for organochlorine pesticides and related compounds, sample extracts (typically a final volume of 500 µL) and calibration standards/solutions are placed in robovials on the autosampler of the GC/MS instrument.

Instrumental calibration and instrument control is as defined in Section 8.0. The HRMS instrumental run conditions are listed in Table 3 and BU-WI-3214 OCP (Parlar) HRMS-Autospec Premier.

A portion of the sample or standard (between 1 to 2 µL) is injected into the instrument for each instrumental run. The injection volume and run conditions must be the same for all injections within each 12 hour run sequence.

An analytical run sequence may start with either a continuing calibration or a new initial calibration. A typical continuous run sequence is listed below.

1a) Initial calibration
1b) CS4 (pre-run CCV)
2) DDT and Endrin breakdown mix
3) QC samples spiked at the CS3 level or higher
4) One or more method blanks and/or a system blanks
5) Samples
6) CS4 (mid-run VER)
7) DDT and Endrin breakdown mix
8) One of more method blanks and/or system blanks
9) Samples
10) CS4 (post-run VER)
11) DDT and Endrin breakdown mix

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This schematic represents an ongoing run sequence, in which the elapsed time for each segment ending in a VER cannot exceed 12 hours (though these segments are usually somewhat shorter than 12 hours for pesticide analysis, due to rapid calibration drift inherent to this list of analytes). Before and after the analytical sequence, and at least every 12 hours during the sequence, a mass resolution check must demonstrate 1 in 8,000 resolution at 10% valley. Instrument conditions must remain the same during each sequence and no other injections can take place between the last injection of a sequence and the ending mass resolution check.

When a multi-point calibration initiates a run sequence, then the injection of the first standard represents the start of the maximum12-hour time limit before a VER and mass resolution check are required.

9.6 Corrective Actions for Out of Control Situations

1. Initial Calibration

The instrument calibration Standard Concentrations are presented in Table 2. Initial calibrations employ anywhere from five to seven levels of standards (possible available levels CS1 through CS7) depending upon the nominal response curves of individual target compounds. For OCP analysis, exceeding the percent relative standard deviation (%RSD or Coefficient of Variance) criteria on the initial calibration does not prevent the analysis of samples. For those analytes that exceed their respective criteria, the response cannot be assumed to be linear and a quadratic fit must be applied rather than the use of an average response factor.

In addition, each target must have a passing ion abundance ratio for the lowest standard level at which that compound is being calibrated.

2. Continuing Calibration

If the performance criteria are not met, GC maintenance is performed or the system is adjusted and a new CCV is injected, or a new initial calibration is run.

3. Mid and Post Run Verification of Results

While the EPA 1699 does not require a post-run calibration verification standard to be run, this may be in recognition that responses and/or relative responses of some targets may change significantly during HRMS analysis due to sample related contamination of GC or MS components. This problem is compounded by chemical dissimilarities between some targets and their quantification reference standards in the case of internal standard quantification. Enhanced quantification and a measure of confidence in sample results obtained during an analytical shift can be attained by injecting a CS4 calibration verification (VER) standard in the middle of, and at the end of a 12-hour run, and quantifying samples against the average of bracketing calibration standards where improved results would be achieved.

a) Mid-Run VER:

If this analysis meets the performance criteria for a pre-run CCV, then all of the samples preceding the mid-run VER can be quantified vs. the initial calibration, and analysis can proceed.

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If the mid-run VER does not meet pre-run CCV criteria, the preceding samples can be quantified vs. bracketing calibration runs (using the pre-run CCV and mid-run VER as a two-point calibration) and analysis can proceed, provided that the following criteria are met:

- Ion abundance ratios are within their respective theoretical limits (see Table 3) or within 15% of the ratios in the pre-run CCV
- All targets have a s/n ratio of at least 10:1
- The RT of each analyte is within 15 seconds of that in the initial calibration
- Endin and DDT breakdown is less that 20% (see Section 8.2.4, Step 1b)
- The %RPD of the mid-run VER vs. the pre-run CCV meets the CCV %Diff limits (See Table 1)

If the mid-run VER does not meet the above criteria either, analysis cannot continue without corrective action (samples analyzed after the mid-run VER in an automated sequence must be re-analyzed). The samples preceding the failing mid-run VER may be flagged and reported, but must be assessed for impact on data quality:

- If a failing native target is present in any of the preceding samples above the Method Detection Limit (or above the client's lower required Detection Limit, if known), that sample must be re-analyzed for that target.
- If a failing native target's Estimated Detection Limit is above the Method Detection Limit (or above the client's lower required Detection Limit. if known) due to deterioration of system performance, that sample must be re-analyzed for that target.

b) Post-Run VER:

If this analysis meets the performance criteria for a pre-run CCV, then all of the samples preceding the post-run VER can be quantified vs. the initial calibration. If the post-run VER does not meet pre-run CCV criteria, the preceding samples can be quantified vs. bracketing calibration runs (using the post-run VER and mid-run VER as a two-point calibration) provided that the following criteria are met:

- Ion abundance ratios are within their respective theoretical limits (see Table 3) or within 15% of the ratios in the mid-run CCV
- All targets have a s/n ratio of at least 10:1
- The RT of each analyte is within 15 seconds of that in the initial calibration
- Endin and DDT breakdown is less that 20% (see Section 8.2.4, Step 1b)
- The %RPD of the post-run VER vs. the mid-run VER meets the CCV %Diff limits (See Table 1)

If the post-run VER does not meet the above criteria either, the samples preceding the failing post-run VER may be flagged and reported, but must be assessed for impact on data quality:

- If a failing native target is present in any of the preceding samples above the Method Detection Limit (or above the client's lower required Detection Limit, if known), that sample must be re-analyzed for that target.
- If a failing native target's Estimated Detection Limit is above the Method Detection Limit (or above the client's lower required Detection Limit. if known) due to deterioration of system performance, that sample must be re-analyzed for that target.

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9.7 Identification Criteria

The following criteria must be met for a positively identified response to labelled or unlabelled analytes on all field samples, QC samples and calibration runs. Failures on any one of these items for a calibration run means that the calibration run is out of control for that analyte. For calibration runs, Instrument control must be regained by re-injection or instrument maintenance before samples can be analyzed. Sample failures indicate the presence of interferences or responses inadequate for qualitative or quantitative determination.

1. Ion Ratio Criteria

For all compounds, a pair of ions with a specific isotopic ratio are being monitored. To have a confirmed positive response to a native or labelled OCP, that ratio must be within the theoretical limits in Table 1, or within 15% of the observed values on the most recent CS4 analysis. If the ion abundance ratio criteria are not met, the result is reported as Not Detected due to Ratio (NDR) and the result qualified with an "R" flag. In this case the detection limit is elevated to the estimated maximum possible concentration.

2. Signal to Noise Criteria

The signal to noise ratio for each quantification and confirmation ion for labelled and non-labelled analytes must be greater than or equal to 10:1 for the initial calibration CS1 and for each calibration verification CS4. For positive identification of a native target in a sample, both ions must have a s/n ratio exceeding 2.5:1.

3. Matched RT on Peak Maxima

The retention time (RT) of the peak maxima for each pair of quantification ions must be no more than 2 seconds (i.e. 2 scans) difference.

4. Expected Retention Time (RT)

The peak must be at the expected RT

- within -1/+3 seconds of the labelled standard for natives with their own ^{13}C labelled standard
- within +/- 0.010 RRT units of the RRT in the most recent CS4 analysis for targets without their own labelled standard

As stated in EPA Method 1699 Sections 16.5-16.6, it is possible that not all of the positive ID criteria are met. If a pesticide is deemed to be present in this case by the experienced spectroscopist, the result may be flagged as "this result is unconfirmed and must not be used for permitting or regulatory compliance purposes".

9.8 System Performance

These are aspects of instrument control that are mandatory before analysis of samples can proceed. If there is any failure on any one or more of these criteria, then Instrument control

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must be regained by re-injection or instrument maintenance before samples can be analyzed.

1. DDT and Endrin Breakdown

If the breakdown of endrin and/or DDT exceeds 20% in a standard, the targets are decomposing on the inlet or column, and remedial action must be taken (inlet maintenance and trimming of the analytical column) before any valid sample data can be produced. If the breakdown of DDT or endrin in a sample exceeds 20% and there is that native in the sample above the MDL, that sample will have to be reanalyzed for that target (further cleanup or dilution of that sample is recommended before reanalysis).

Breakdown exceedences can be ignored under the following circumstances:

- Where the endrin breakdown fails but DDT breakdown passes and where DDT and/or it's metabolites are the only targets.
- Where the DDT breakdown fails but endrin breakdown passes and where endrin and/or it's metabolites are the only targets.
- For the determination of other pesticide targets (i.e. non-DDT and non-Endrin metabolite targets) which have a corresponding labelled extraction/internal standard of exactly the same isomer.

2. Mass Resolution

At the beginning of and just following the end of each 12 hour run sequence, the instrument must be checked to demonstrate a resolution of 8,000 at 10% valley for each quantification window. If the resolution check fails at the end of the sequence, then the data from the sequence should be reviewed carefully for a) chemical interferences and b) deterioration in instrument sensitivity. If upon review there remains concern for real compromise to data quality, then re-injection of the impacted samples under full instrument control is required. If during the evaluation, the data assessment concludes that there has been no significant compromise to the data quality from the drift in resolution, then the data may be reported while including a comment on the report identifying the control failure and the absence of impact on data quality.

3. Maximum Scan Time

The maximum time between scans within a descriptor is 1 second.

9.9 Individual Run Performance

1. Lock Mass Drift and Response Deviation

Lock mass drift correction must be used within every descriptor to adjust for small changes in mass peak location.

The response of the mass calibrant (typically PFK) is also monitored to determine the presence of interferences. Deviations to the average response must be less than or equal 20%. Deviations outside of this may indicate the presence of chemical interference(s) which may impact data quality.

Where lock mass deviations exceed the 20% value within the target window, an assessment on the impact of data quality is required. Whenever the deviation co-elutes with and compromises

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quantitation of any calibrated target analyte, then one or more of the following options in remedial actions must be implemented: (a) re-injection (b) dilution/re-injection (c) re-clean of the extract followed by re-analysis (c) additional cleanups of the extract followed by re-analysis or (d) re-extraction/re-analysis. If re-extraction is in order, re-extraction of a smaller sample size should be considered. Ultimately, any remedial action needs to provide data within lock mass control and that meets the method and project data quality objectives.

10.0 DATA CALCULATION AND REPORTING

10.1 Data Calculations

1. Calculation of Concentrations:

The relative response factor of each target relative to the standard against which it is to be calculated is determined using the area responses of both quantification ions via equation 1.

In cases where a native target is calculated against an exact labelled analogue, the quantification will be considered to be by isotope dilution. In other cases, the quantification will be considered to be by internal standard.

$$RRF = \frac{(A1_t + A2_t)\ C_s}{(A1_s + A2_s)\ C_t} \qquad \textbf{Equ. 1}$$

Where,

$A1_t + A2_t$ = The areas of the two quantification ions for the target analyte

$A1_s + A2_s$ = The areas of the two quantification ions for the labelled compound against which the target analyte will be calculated.

C_t = The concentration in the calibration standard of the target analyte.

C_s = The concentration in the calibration standard of the labelled compound against which the target will be calculated.

For all analytes to be quantified and from the initial calibration series of standard injections, a table of RRFs is prepared. The relative standard deviation (%RSD, or the coefficient of variance) is checked to confirm that the appropriate method criteria has been met as listed in Table 3. The average of the appropriate RRF from the ICAL (or bracketing calibration standards, as appropriate) for each analyte, RRF_{av} is applied for quantification of samples according to Equations 2 and 3 below.

$$\text{Amount in sample (pg)} = \frac{(A1_n + A2_n)\ Q_l}{(A1_l + A2_l)\ (RRF_{av})} \qquad \textbf{Equ. 2}$$

$$\text{Concentration in sample (pg/g or pg/L)} = \frac{(A1_n + A2_n)\ Q_l}{} \qquad \textbf{Equ. 3}$$

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$$(A1_l + A2_l)\ (RRF_{av})(W_s)$$

Where,

Q_l = The amount (pg) of labelled compound added to the sample

W_s = The weight (g) or volume (l) of sample

2. Extraction, Clean-up, and Sampling Standard Recovery Calculation:

The extraction, clean-up, and sampling standard recoveries are determined by Equation 4 below.

% Recovery = (Amount in sample)/(Amount added to sample) X 100 **Equ. 4**

3. Estimated Detection Limit

$$EDL = \frac{2.5 \times H_x \times Q_{es}}{H_{es} \times W \times RFF_{av}}$$ **Equ. 5**

Where,

EDL = estimated detection limit for homologous OCPs

H_x = sum of the height of the noise level for each quantification ions for the unlabelled OCP.

H_{es} = sum of the heights of responses of both quantification ions for the labelled extraction standard.

W = weight of volume of sample

RRF_{av} = average relative response factor

Q_{es} = amount of extraction standard added

4. Estimated Maximum Possible Concentration (EMPC):

An estimated maximum possible concentration (EMPC) is a worst-case estimate of the concentration of OCP analyte under a chemical interference. It is calculated assuming theoretical isotopic ratio and applying equations 2 and 3.

5. DDT Breakdown

For measurement of DDT breakdown, measure the concentration for $^{13}C_{12}$-44'-DDE, $^{13}C_{12}$-44'-DDD and $^{13}C_{12}$-44'-DDT (the labelled DDT is part of the standard, and the labelled DDE and DDD are breakdown products). Calculate breakdown using the following formula:

$^{13}C_{12}$-44'-DDT % Breakdown =

$$\frac{(\text{concentration of } ^{13}C_{12}\text{-44'-DDD + concentration of } ^{13}C_{12}\text{-44'-DDE}) \times 100\%}{\text{concentration of } ^{13}C_{12}\text{-44'-DDT}}$$

labelled DDT = part of standard; labelled DDE and DDD = breakdown products

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6. Endrin Breakdown

For measurement of endrin breakdown, measure the concentration of endrin, endrin aldehyde, and endrin ketone (these natives are quantified by isotope dilution vs. the $^{13}C_{12}$-endrin). Calculate breakdown using the following formula:

Endrin % Breakdown =

$$\frac{\text{(concentration of endrin aldehyde + concentration of endrin ketone)}}{\text{concentration of endrin}} \times 100\%$$

2.5 Data Transfer to LIMS

Refer to BU-WI-3225 Reporting HRMS Results.

2.6 Data Validation

Peer review and sign off is a requirement, refer to NA-SP-0175 Data Validation for details.

10.3.1 Method Blank Failure

If the blank level exceeds the DQO (see Section 8.0) for any analyte, then the corrective action is to re-analyze any sample within the batch that has a positively determined analyte with up to 10-fold higher response of the blank level and, for regulatory compliance, if the biased analyte in the sample is within a factor of 10 of the regulatory compliance limit.

10.3.2 LCS Failure

When the LCS recoveries are outside the recommended ranges, an evaluation of the impact on the data quality is required. Failure(s) may indicate compromise to one or more of the standards or to the extraction/clean-up process. If the evaluation shows significant compromise to the reported data quality for one or more samples in the batch, then the affected samples should be re-injected and/or repeated for OCP analysis from archived extract and/or from another portion of the original sample. If repeat analysis is desired and additional sample is not available for repeat, then a final evaluation of the pooled results is required to evaluate appropriate data flagging or non-reporting of the results.

10.3.3 MS and MSD Failure

Corrective actions from failure of this criterion include, at the least, re-evaluation of the data set with respect to calculation errors or analytical compromise. Re-injection or re-analysis of the sample for MS/MSD may be considered. Re-extraction and re-analysis of the entire batch may be considered.

10.3.4 DUP Failure

Corrective actions from failure of this criterion include, at the least, examination of the sample for

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evidence of inhomogeneity or evaluation of the data set with respect to calculation errors and analytical compromise. For solids, sample inhomogeneity is a common source of the problem. Documented evidence for inhomogeneity may resolve a criterion failure. Re-injection or re-analysis of the sample and duplicate, or the entire batch may be considered.

10.4 Data Reporting

Results are reported to three significant figures for the OCPs found in all standards, blanks, and samples. Extraction, Clean-up, and Sampling Standards are reported as a percent recovery to the nearest integer.

1. Aqueous samples—Report results in ng/L.

2. Samples containing greater than 1% solids (soils, sediments, filter cake, compost)—Report results in ng/g based on the dry weight of the sample. Report the percent solids so that the result may be converted to aqueous units.

3. Tissues—Report results in ng/g of wet tissue, not on the basis of the lipid content of the tissue. Report the percent lipid content, so that the data user can calculate the concentration on a lipid basis if desired.

4. Reporting level.

a) Results above the minimum level of quantitation (ML) are reported for the analysis of blanks, standards, and samples. The estimated minimum levels (EMLs) in Table 1 are based on common laboratory contamination levels. A laboratory may establish an ML for an OCP lower than the EMLs in Table 1. MLs may be established as low as the lowest calibration point (Table 4) provided that the concentration of the congener in a minimum of 10 blanks for a sample medium (e.g., water, soil, sludge, tissue) is significantly below the EML in Table 1. Significant means that the ML for the congener is no less than the average (mean) plus 2 standard deviations above the level in the minimum of 10 blanks. The blanks must be analyzed during the same period that the sample is analyzed, ideally over an approximately 1-month period.

b) Standards (VER, IPR, OPR) and samples—Report the result for each congener at or above the ML (or EML Table 1) to 3 significant figures. Report results below the ML (or EML) as <ML (where ML is the concentration at the ML) or as required by the regulatory authority or permit.

c) Blanks—Report results above the ML (or EML) to 3 significant figures. Report results below the ML but above the MDL (or EMDL) to 2 significant figures. Report results below the MDL as <MDL (where MDL is the concentration at the MDL) or as required by the regulatory authority or permit.

d) Blank correction—Blank-corrected results may be reported in addition to reporting of separate results for samples and blanks). The recommended procedure for blank correction is that a result is significantly above the blank level, and the level in the blank may be subtracted, if the result is greater than the mean plus 2 standard deviations of results of analyses of 10 or more blanks for a sample medium.

5. Results for an OCP in a sample that has been diluted are reported at the least dilute level at

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which the area at the quantitation m/z is within the calibration range.

6. For an OCP having a labelled analog, report results at the least dilute level at which the area at the quantitation m/z is within the calibration range and the labelled compound recovery is within the normal range for the Method.

11.0 References

11.1 Primary References

US EPA Office of Water, Method 1699 (December, 2007)

11.2 Secondary References

US EPA Office of Hazardous Waste, Method 8290A (February 2007)
40 CFR Part 136 Appendix B

11.3 Instrument Manuals

1. Autospec Premier Operator's Guide 71500089202/Revision B
2. Gerstel Operation Manual Cooled Injection System CIS 4 with C506
3. Gerstel Operation Manual Universal Peltier Cooling UPC Plus

11.4 Software Manuals

1. Waters MassLynx 4.1 Getting Started Guide 71500113203/Revision A

11.5 Supporting ALS Documents and Forms

1. NA-SP-0001 Definitions of Key Terms
2. NA-SP-0100 Initial Method Validation – Chemistry
3. NA-SP-0102 Method Revalidation and MU – Chemistry
4. NA-SP-0175 Data Validation
5. BU-SP-0263 Dilution
6. BU-SP-0021 Waste Handling and Sample Disposal
7. BU-TP-2103 OCP & Toxaphene Prep
8. BU-WI-3000 Organic Glassware/Equipment Cleaning, Proofing and Maintenance
9. BU-WI-3002 Standards Prep, Spiking and Witnessing
10. BU-WI-3007 GCMS Operation
11. BU-WI-3019 Manual Integration
12. BU-WI-3214 OCP (Parlar) HRMS-Autospec Premier
13. BU-WI-3225 Reporting HRMS Results
14. BU-FM-1103a OCPEST-1699-HRMSTL-1.0
15. BU-FM-1103b Parlars-1699-HRMSTL-2.0

12.0 DEVIATIONS FROM THE PRIMARY REFERENCE METHOD

1. Monitoring masses have been optimized and improved relative to the reference method
2. For selected targets, the extraction standards used are different than those prescribed in EPA method 1699. Some extraction standards used are alternate compounds, and additional

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extraction standards have been added, to improve the calibration accuracy and stability of known problematic analytes.

3. For targets in which there are not corresponding 13C- labelled extraction standards (ES), the analyte data are calculated relative to labelled ES of similar structures to those targets, and that have been empirically observed to give similar extraction recoveries. The quantitation references used are not in all cases the same as those selected by EPA 1699.
4. Selected CCV, LCS and Extraction Standard recovery control limits have been changed to reflect the method modifications.
5. The 1699 extraction standard recovery limit for 13C12-44'-DDE in samples is 47-160. This has been changed to 21-125 based off 3s statistical analysis over 2 years and approx. 500 tissue sample data points.
6. Inclusion of three persistent toxaphene components, Parlar-26, -50 & -62.
7. Inclusion of 13C12-PCB-52, -101 as additional injection standards to more closely match retention times and/or masses of extraction standards.
8. Provisions for mid and post-run continuing calibration verification standards and quantification using daily response factors have been added.
9. GC column employed is HP5ms or equivalent rather than the DB17 column recommended by EPA Method 1699. Due to the different column, the method employed also does not follow the 39 min rule for Methoxychlor retention time.
10. Breakdown calculations have been altered relative to the 1699 documented approach to be based upon a concentration rather than area response thus accounting for the differences in area responses applicable to selection ion monitoring (SIM) mode (the 1699 calculation is erroneously based upon ECD responses not SIM responses). Breakdown exceedances can be ignored under the following circumstances:
    - Where the endrin breakdown fails but DDT breakdown passes and where DDT and/or its metabolites are the only targets.
    - Where the DDT breakdown fails but endrin breakdown passes and where endrin and/or its metabolites are the only targets.
    - For the determination of other pesticide targets (i.e. non-DDT and non-Endrin and metabolite targets) which have a corresponding labelled extraction/internal standard of exactly the same isomer.

13.0 REVISION HISTORY

Version #	Date	Summary of Major Changes Made / By
01	2010 June 08	Method creation and definition. Brad Reimer/ Ron McLeod
02	2010 Sep 17	Clarifications on mass resolution and lock./ Ron McLeod
03	2012 Jan 06	Updated reference for routine soxhlet extraction to BU-TM-2100 PAH Prep Method. Added 1245-Tetrachlorobenzene, 1234-Tetrachlorobenzene, Pentachlorobenzene, Octachlorostyrene to the parameter list and updated the tables 1 through 4 appropriately. Ryan Gordon / Brad Reimer / Ron McLeod
04	2012 Nov 15	Updated instrument conditions and added Appendix C. BReimer/PLeung
05	12-Feb-14	Made appendix C into BU-WI-3202. Fixed up to match the national template. Changed targets. Modifications to standards (native and labelled) / SEagle, RMcLeod, SKennedy
06	24-Nov-2014	Referenced WI for Autospec Premier. Incorporated handwritten changes. / BReimer, SEagle
07	2-Feb-2017	Major overhaul in response to technical audit, all sections have been updated.

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Version #	Date	Summary of Major Changes Made / By
		/ R. Saxon, M.McPherson
08	9-Oct-2018	Section 1.2 split Solid and Tissue into different columns, updated LORs. Updated supplies and standards sections. Updated references throughout and in section 11. Corrections made to Table 1 and 3 in Appendix. / RSaxon Updated 13C12-44'-DDE sample limits based on statistical limits. / RSaxon Corrected 13C10-trans-Nonachlor and 13C9-Endosulfan II values in table 1 as per 1699. / RSaxon
09	8-Jul-2020	Various improvements to the wording of some sections. Added native Isodrin and added/replaced ES analytes to the current new list of targets. Corrected standard prep sections to reflect current scheme and analytes. Added updated safety section. / BReimer

14.0 WASTE MANAGEMENT

Refer to 'BU-SP-0021 Waste Handling and Sample Disposal'. Waste information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

15.0 SAFETY

This task may include CHEMICAL, BIOLOGICAL, OPERATIONAL and/or EQUIPMENT hazards. Staff must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

Refer to Safety Data Sheets (SDS) for detailed information about safe handling guidelines and hazards associated with chemicals used in this method.

Refer to BU-WI-3007 GCMS Operation for hazards associated with operation of HRMS equipment.

Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

HAZARD ASSESSMENT			
Job Task #1	**Hazards**		**Preventive Measures**
Sample handling	Exposure to solvents, pesticide residues, and contaminants in samples.		Wear gloves, safety glasses and lab coat. Work in fume hood. Avoid skin contact with solvents, samples, and sample extracts.
Job Task #2: Handling Reagents and Standards	**Hazards (see hazard code)**		**Preventive Measures**
Toluene	Danger: Flammable, irritant, health hazard		Wear gloves, safety glasses and lab coat. Work in fume hood. Avoid skin contact with solvents, samples and sample extracts.

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HAZARD ASSESSMENT			
Pesticide Standards and Residues	Danger: Flammable liquids and vapors, may be fatal if swallowed or inhaled, causes serious skin or eye irritation, may cause drowsiness or dizziness		Wear gloves, safety glasses and lab coat. Work in fume hood. Take all necessary measures to prevent skin contact with solvents and neat or diluted standard materials.
Job Task #3	**Hazards**		**Preventive Measures**
Extended computer use	Eye strain and fatigue		Take regular short breaks to work on different tasks. Focus on objects at varying distances and/or rest your eyes for a few moments throughout the day.

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16.0 APPENDICES

16.1 Appendix A – Tables

Table 1: Acceptance Criteria for CCV, IPR, OPR and Samples

Pesticide		CCV Limits (% Diff)	IPR Rec. Limits (%)	IPR %RSD Max	OPR Rec. Limits (%)	Sample Rec Limits (%)
Native	Hexachlorobutadiene	70-130	5-200	50	5-200	
	1,2,4,5-Tetrachlorobenzene	70-130	5-200	50	5-200	
	1,2,3,4-Tetrachlorobenzene	70-130	5-200	50	5-200	
	Pentachlorobenzene	70-130	5-200	30	5-200	
	Hexachlorobenzene	75-125	10-120	30	10-150	
	3,4,5,6-Tetrachloroveratrole	70-130	20-200	50	20-200	
	Pentachloroanisole	70-130	20-200	50	20-200	
	α-Hexachlorocyclohexane	75-125	55-108	30	50-120	
	β-Hexachlorocyclohexane	75-125	55-108	30	50-120	
	γ-Hexachlorocyclohexane	75-125	55-108	30	50-120	
	□-Hexachlorocyclohexane	75-125	55-108	30	50-120	
	Pentachloronitrobenzene	70-130	20-200	50	20-200	
	Heptachlor	75-125	55-108	30	50-120	
	4,4'-DDNU	75-125	50-150	30	20-160	
	Aldrin	75-125	55-108	30	50-120	
	Isodrin	75-125	55-108	30	50-120	
	Chlorpyrifos	70-130	21-147	46	19-163	
	Dacthal	50-150	20-200	50	20-200	
	Octachlorostyrene	70-130	55-158	30	50-175	
	Heptachlor Epoxide B	70-130	20-200	50	20-200	
	Heptachlor Epoxide A	75-125	55-108	30	50-120	
	Oxychlordane	75-125	55-108	30	50-120	
	trans-Chlordane	75-125	55-108	30	50-120	
	4,4'-DDMU	75-125	50-150	30	20-160	
	2,4'-DDE	75-125	26-111	30	24-123	
	cis-Chlordane	75-125	55-108	30	50-120	
	trans-nonachlor	75-125	55-108	30	50-120	
	Endosulphan I	75-125	55-108	30	50-120	
	Dieldrin	75-125	55-108	30	50-120	
	4,4'-DDE	75-125	55-108	30	50-120	
	2,4'-DDD	75-125	55-108	30	50-120	
	Endrin	75-125	55-108	30	50-120	
	Parlar-26	70-130	20-200	50	20-200	
	Endosulphan II	75-125	5-200	50	5-200	
	cis-Nonachlor	75-125	55-108	30	50-120	
	4,4'-DDD	75-125	47-108	30	42-120	
	2,4'-DDT	75-125	55-108	30	50-120	
	Endosulphan Sulfate	75-125	55-180	50	50-200	
	Endrin Aldehyde	70-130	20-200	50	20-200	
	4,4'-DDT	75-125	55-108	30	50-120	
	Dicofol	50-150	20-200	50	20-200	
	Endrin Ketone	75-125	55-120	30	50-134	
	Methoxychlor	75-125	55-108	30	50-120	
	Mirex	75-125	55-108	30	50-120	
	Parlar 26	70-130	20-200	50	20-200	

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Pesticide		CCV Limits (% Diff)	IPR Rec. Limits (%)	IPR %RSD Max	OPR Rec. Limits (%)	Sample Rec Limits (%)
	Parlar-50	70-130	20-200	50	20-200	
	Parlar-62	70-130	20-200	50	20-200	
Labelled	$^{13}C_6$-Pentachorobenzene	70-130	5-120	70	5-120	5-120
	$^{13}C_6$-Hexachorobenzene	70-130	6-108	70	5-120	5-120
	$^{13}C_6$-alpha-BHC	70-130	14-125	52	13-138	16-129
	$^{13}C_6$-beta-BHC	70-130	6-112	62	5-124	11-120
	$^{13}C_6$-gamma-BHC	70-130	6-112	62	5-124	11-120
	$^{13}C_6$-delta-BHC	70-130	6-112	62	5-124	11-120
	$^{13}C_{10}$-Heptachlor	70-130	6-115	67	5-128	5-120
	$^{13}C_{10}$-Oxychlordane	70-130	6-129	54	5-144	23-135
	$^{13}C_{10}$-trans-Nonachlor	70-130	15-134	49	13-149	14-136
	$^{13}C_{12}$-Dieldrin	70-130	21-145	46	19-161	40-151
	$^{13}C_{12}$-Endrin	70-130	22-141	45	20-157	35-155
	$^{13}C_9$-Endosulfan II	70-130	6-108	59	5-120	5-122
	$^{13}C_{12}$-24'-DDE	70-130	15-180	52	13-200	5-150
	$^{13}C_{12}$-44'-DDE	70-130	29-152	43	26-169	21-125
	$^{13}C_{12}$-44'-DDD	70-130	15-180	52	13-200	5-150
	$^{13}C_{12}$-44'-DDT	70-130	15-180	52	13-200	5-120
	$^{13}C_{12}$-Methoxychlor	70-130	8-180	54	8-200	5-120
	$^{13}C_{10}$-Mirex	70-130	6-125	56	5-138	5-120

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Table 2: Calibration Standards

Pesticide		Concentration in pg/uL						
		CS1 (pg/µL)	CS2 (pg/µL)	CS3 (pg/µL)	CS4 (pg/µL)	CS5 (pg/µL)	CS6 (pg/µL)	CS7 (pg/µL)
Native:	Hexachlorobutadiene	2	7.5	20	50	150	400	1200
	1,2,4,5-Tetrachlorobenzene	2	7.5	20	50	150	400	1200
	1,2,3,4-Tetrachlorobenzene	2	7.5	20	50	150	400	1200
	Pentachlorobenzene	2	7.5	20	50	150	400	1200
	Hexachlorobenzene	2	7.5	20	50	150	400	1200
	3,4,5,6-Tetrachloroveratrole	2	7.5	20	50	150	400	1200
	Pentachloroanisole	2	7.5	20	50	150	400	1200
	α-Hexachlorocyclohexane	2	7.5	20	50	150	400	1200
	β-Hexachlorocyclohexane	2	7.5	20	50	150	400	1200
	γ-Hexachlorocyclohexane	2	7.5	20	50	150	400	1200
	□-Hexachlorocyclohexane	2	7.5	20	50	150	400	1200
	Pentachloronitrobenzene	2	7.5	20	50	150	400	1200
	Heptachlor	2	7.5	20	50	150	400	1200
	4,4'-DDNU	2	7.5	20	50	150	400	1200
	Aldrin	2	7.5	20	50	150	400	1200
	Isodrin	2	7.5	20	50	150	400	1200
	Chlorpyrifos	10	37.5	100	250	750	2000	6000
	Dacthal	2	7.5	20	50	150	400	1200
	Octachlorostyrene	2	7.5	20	50	150	400	1200
	Heptachlor Epoxide B	2	7.5	20	50	150	400	1200
	Heptachlor Epoxide A	2	7.5	20	50	150	400	1200
	Oxychlordane	2	7.5	20	50	150	400	1200
	trans-Chlordane	2	7.5	20	50	150	400	1200
	4,4'-DDMU	2	7.5	20	50	150	400	1200
	2,4'-DDE	2	7.5	20	50	150	400	1200
	cis-Chlordane	2	7.5	20	50	150	400	1200
	trans-nonachlor	2	7.5	20	50	150	400	1200
	Endosulphan I	2	7.5	20	50	150	400	1200
	Dieldrin	2	7.5	20	50	150	400	1200
	4,4'-DDE	2	7.5	20	50	150	400	1200
	2,4'-DDD	2	7.5	20	50	150	400	1200
	Endrin	2	7.5	20	50	150	400	1200
	Parlar-26	2	7.5	20	50	150	400	1200
	Endosulphan II	2	7.5	20	50	150	400	1200
	cis-Nonachlor	2	7.5	20	50	150	400	1200
	4,4'-DDD	2	7.5	20	50	150	400	1200
	2,4'-DDT	2	7.5	20	50	150	400	1200
	Endosulphan Sulfate	2	7.5	20	50	150	400	1200
	Endrin Aldehyde	2	7.5	20	50	150	400	1200
	4,4'-DDT	2	7.5	20	50	150	400	1200
	Endrin Ketone	2	7.5	20	50	150	400	1200
	Methoxychlor	2	7.5	20	50	150	400	1200
	Dicofol	20	75	200	500	1500	4000	12000
	Mirex	2	7.5	20	50	150	400	1200
	Parlar 26	2	7.5	20	50	150	400	1200
	Parlar-50	2	7.5	20	50	150	400	1200
	Parlar-62	2	7.5	20	50	150	400	1200
Labelled:	$^{13}C_6$-Pentachorobenzene	100	100	100	100	100	100	100
	$^{13}C_6$-Hexachorobenzene	100	100	100	100	100	100	100
	$^{13}C_6$-alpha-BHC	200	200	200	200	200	200	200
	$^{13}C_6$-beta-BHC	200	200	200	200	200	200	200

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Pesticide	Concentration in pg/uL						
	CS1 (pg/µL)	CS2 (pg/µL)	CS3 (pg/µL)	CS4 (pg/µL)	CS5 (pg/µL)	CS6 (pg/µL)	CS7 (pg/µL)
$^{13}C_6$-gamma-BHC	200	200	200	200	200	200	200
$^{13}C_6$-delta-BHC	200	200	200	200	200	200	200
$^{13}C_{10}$-Heptachlor	200	200	200	200	200	200	200
$^{13}C_{10}$-Oxychlordane	200	200	200	200	200	200	200
$^{13}C_{10}$-trans-Nonachlor	200	200	200	200	200	200	200
$^{13}C_{12}$-Dieldrin	100	100	100	100	100	100	100
$^{13}C_{12}$-Endrin	200	200	200	200	200	200	200
$^{13}C_9$-Endosulfan II	200	200	200	200	200	200	200
$^{13}C_{12}$-44'-DDE	100	100	100	100	100	100	100
$^{13}C_{12}$-44'-DDE	100	100	100	100	100	100	100
$^{13}C_{12}$-44'-DDD	100	100	100	100	100	100	100
$^{13}C_{12}$-44'-DDT	100	100	100	100	100	100	100
$^{13}C_{12}$-Methoxychlor	100	100	100	100	100	100	100
$^{13}C_{10}$-Mirex	100	100	100	100	100	100	100
PCB-9-$^{13}C_{12}$	100	100	100	100	100	100	100
PCB-52-$^{13}C_{12}$	100	100	100	100	100	100	100
PCB-101-$^{13}C_{12}$	100	100	100	100	100	100	100
PCB-101-$^{13}C_{12}$P	100	100	100	100	100	100	100
1,3-Dibromobenzene	20	20	20	20	20	20	20
1,3,5-Triboromobenzene	20	20	20	20	20	20	20
1,2,4,5-Tetrabromobenzene	20	20	20	20	20	20	20

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Table 3: Suggested HRMS Functions, m/z, Ion Abundance Ratio Tolerance & Quantitative References

Function	Target	Est. RT (min)	Primary m/z	Secondary m/z	Theor. Ion Ratio	Ion Ratio Tolerance (+/-)	Quantitative Reference
1	PFK Lock Mass		242.9851				
	1,3-Dibromobenzene [3]	4.34	233.8680	235.8659	0.51	25%	$^{13}C_{12}$-PCB-9 (IS)
	Hexachlorobutadiene	4.75	259.8102	261.8072	1.25	25%	$^{13}C_6$-Pentachlorobenzene
	1,2,4,5-Tetrachlorobenzene	6.69	215.8881	217.8852	2.08	25%	$^{13}C_6$-Pentachlorobenzene
	1,2,3,4-Tetrachlorobenzene	7.58	215.8881	217.8852	2.08	25%	$^{13}C_6$-Pentachlorobenzene
	1,3,5-Tribromobenzene [3]	8.40	313.7764	315.7744	1.03	25%	$^{13}C_{12}$-PCB-9 (IS)
	Pentachlorobenzene	10.26	249.8491	251.8462	1.56	25%	$^{13}C_6$-Pentachlorobenzene
	$^{13}C_6$-Pentachlorobenzene	10.25	255.8693	257.8663	1.56	25%	$^{13}C_{12}$-PCB-9 (IS)
2	PFK Lock Mass		242.9851				
	Hexachlorobenzene	13.80	283.8102	285.8072	1.23	25%	$^{13}C_6$-Hexachlorobenzene
	$^{13}C_{12}$-PCB-9 (IS)	13.08	234.0406	236.0376	1.56	15%	
	$^{13}C_6$-Hexachlorobenzene	13.79	289.8303	291.8273	1.23	25%	$^{13}C_{12}$-PCB-9 (IS)
	3,4,5,6-Tetrachloroveratrole	13.87	275.9092	277.9063	2.08	25%	$^{13}C_6$-α-Hexachlorocyclohexane
	Pentachloroanisole	14.00	279.8597	281.8568	1.56	35%	$^{13}C_6$-α-Hexachlorocyclohexane
	α-Hexachlorocyclohexane	13.57	218.9116	220.9088	2.10	25%	$^{13}C_6$-α-Hexachlorocyclohexane
	β-Hexachlorocyclohexane	14.45	218.9116	220.9088	2.10	25%	$^{13}C_6$-β-Hexachlorocyclohexane
	γ-Hexachlorocyclohexane	14.70	218.9116	220.9088	2.10	25%	$^{13}C_6$-γ-Hexachlorocyclohexane
	d-Hexachlorocyclohexane	15.55	218.9116	220.9088	2.10	25%	$^{13}C_6$-d-Hexachlorocyclohexane
	d-Hexachlorocyclohexane [3]						$^{13}C_{12}$-PCB-101 (IS)
	$^{13}C_6$-α-Hexachlorocyclohexane	13.57	224.9317	226.9286	2.10	25%	$^{13}C_{12}$-PCB-9 (IS)
	$^{13}C_6$-β-Hexachlorocyclohexane	14.45	224.9317	226.9286	2.10	25%	$^{13}C_{12}$-PCB-9 (IS)
	$^{13}C_6$-γ-Hexachlorocyclohexane	14.65	224.9317	226.9286	2.10	25%	$^{13}C_{12}$-PCB-9 (IS)
	$^{13}C_6$-d-Hexachlorocyclohexane	15.55	224.9317	226.9286	2.10	25%	$^{13}C_{12}$-PCB-9 (IS)
	1,2,4,5-Tetrabromobenzene [3]	14.24	312.7686	314.7666	1.03	25%	$^{13}C_{12}$-PCB-52 (IS)
	Pentachloronitrobenzene	14.88	294.8342	296.8313	1.56	25%	$^{13}C_{10}$-Heptachlor
3	PFK Lock Mass		280.9819				
	Heptachlor	17.09	271.8102	273.8072	1.25	25%	$^{13}C_{10}$-Heptachlor
	$^{13}C_{10}$-Heptachlor	17.07	276.8269	278.8240	1.25	25%	$^{13}C_{12}$-PCB-52 (IS)
	4,4'-DDNU	17.45	248.0160	250.0130	1.56	25%	$^{13}C_{10}$-Oxychlordane
	$^{13}C_{12}$-PCB-52 (IS)	17.75	301.9625	303.9597	0.77	15%	
	Aldrin	18.19	262.8570	264.8540	1.56	25%	$^{13}C_{10}$-Oxychlordane
4	PFK Lock Mass		280.9819				
	Dacthal	18.73	331.8991	333.8961	2.08	25%	$^{13}C_{10}$-Oxychlordane
	Chlorpyrifos	18.50	313.9574	315.9545	1.44	25%	$^{13}C_{10}$-Oxychlordane
	Isodrin	19.05	262.8570	264.8540	1.56	25%	$^{13}C_{10}$-Oxychlordane
5	PFK Lock Mass		380.9755				
	Octachlorostyrene	19.42	342.7790	344.7761	1.04	25%	$^{13}C_{10}$-Oxychlordane
	Heptachlor Epoxide B	19.50	352.8440	354.8410	1.2	25%	$^{13}C_{10}$-Oxychlordane
	Heptachlor Epoxide A	19.64	352.8440	354.8410	1.2	25%	$^{13}C_{10}$-Oxychlordane
	Oxychlordane	19.54	386.8050	388.8020	1.02	25%	$^{13}C_{10}$-Oxychlordane
	$^{13}C_{10}$-Oxychlordane	19.53	396.8385	398.8355	1.02	25%	$^{13}C_{12}$-PCB-52 (IS)
6	PFK Lock Mass		268.9819				
	trans-Chlordane	20.26	262.8570	264.8540	1.56	25%	$^{13}C_{10}$-*trans*-nonachlor
	4,4'-DDMU	20.30	247.0081	249.0052	1.56	25%	$^{13}C_{12}$-4,4'-DDE
	$^{13}C_{12}$-PCB-101 (IS)	20.49	337.9206	339.9177	1.56	25%	
	2,4'-DDE	20.50	246.0003	247.9974	1.56	25%	$^{13}C_{12}$-2,4'-DDE
	cis-Chlordane	20.75	262.8570	264.8540	1.56	25%	$^{13}C_{10}$-*trans*-nonachlor
	trans-nonachlor	20.90	262.8570	264.8540	1.56	25%	$^{13}C_{10}$-*trans*-nonachlor
	$^{13}C_{10}$-*trans*-nonachlor	20.89	269.8804	271.8775	1.56	25%	$^{13}C_{12}$-PCB-101 (IS)
	$^{13}C_{12}$-2,4'-DDE	20.50	258.0405	260.0376	1.56	25%	$^{13}C_{12}$-PCB-101 (IS)
	Endosulphan I	20.63	276.8726	278.8697	1.56	25%	$^{13}C_9$-Endosulfan II
7	PFK Lock Mass		268.9819				

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Function	Target	Est. RT (min)	Primary m/z	Secondary m/z	Theor. Ion Ratio	Ion Ratio Tolerance (+/-)	Quantitative Reference
	Dieldrin	21.44	262.8570	264.8540	1.56	25%	$^{13}C_{12}$-Dieldrin
	$^{13}C_{12}$-Dieldrin	21.43	269.8804	271.8775	1.56	25%	$^{13}C_{12}$-PCB-101 (IS)
	4,4'-DDE	21.51	246.0003	247.9974	1.56	25%	$^{13}C_{12}$-4,4'-DDE
	$^{13}C_{12}$-4,4'-DDE	21.50	258.0405	260.0376	1.56	25%	$^{13}C_{12}$-PCB-101 (IS)
	2,4'-DDD	21.75	235.0081	237.0052	1.56	25%	$^{13}C_{12}$-4,4'-DDD
	Endrin	22.09	262.8570	264.8540	1.56	25%	$^{13}C_{12}$-Endrin
	$^{13}C_{12}$-Endrin	22.08	269.8804	271.8775	1.56	25%	$^{13}C_{12}$-PCB-101 (IS)
	Endosulphan II	22.40	276.8726	278.8697	1.56	25%	$^{13}C_{9}$-Endosulfan II
	$^{13}C_{9}$-Endosulfan II	22.39	284.8995	286.8965	1.56	25%	$^{13}C_{12}$-PCB-101 (IS)
	Parlar-26	22.09	304.9039	306.9010	1.56	35%	$^{13}C_{10}$-Mirex
8	PFK Lock Mass		268.9819				
	cis-Nonachlor	22.83	262.8570	264.8540	1.56	25%	$^{13}C_{10}$-*trans*-nonachlor
	$^{13}C_{12}$-PCB-133 [1.]	22.09	299.9470	301.9440	0.77	25%	$^{13}C_{12}$-PCB-101 (IS)
	4,4'-DDD	22.78	235.0081	237.0052	1.56	25%	$^{13}C_{12}$-4,4'-DDD
	$^{13}C_{12}$-4,4'-DDD	22.78	247.0483	249.0454	1.56	25%	$^{13}C_{12}$-PCB-101 (IS)
	2,4'-DDT	22.98	235.0081	237.0052	1.56	25%	$^{13}C_{12}$-4,4'-DDD
	Endrin Aldehyde	24.00	247.8521	249.8491	0.78	25%	$^{13}C_{12}$-Endrin
	Endrin Aldehyde [2.]	24.00	342.9018	344.8988	0.62	25%	$^{13}C_{12}$-Endrin
9	PFK Lock Mass		268.9819				
	Endosulfan Sulfate	23.71	276.8726	278.8697	1.56	25%	$^{13}C_{9}$-Endosulphan II
	4,4'-DDT	23.90	235.0081	237.0052	1.56	25%	$^{13}C_{12}$-4,4'-DDT
	$^{13}C_{12}$-4,4'-DDT	23.89	247.0483	249.0454	1.56	25%	$^{13}C_{12}$-PCB-101 (IS)
10	PFK Lock Mass		268.9819				
	Endrin Ketone	25.08	316.9040	318.9010	1.56	25%	$^{13}C_{12}$-Endrin
	Endrin Ketone[3.]						$^{13}C_{12}$-PCB-101 (IS)
	Methoxychlor	25.69	227.1072	228.1106	6.20	35%	$^{13}C_{12}$-Methoxychlor
	$^{13}C_{12}$-Methoxychlor	25.51	239.1475	240.1508	24.0	35%	$^{13}C_{12}$-PCB-101 (IS)
	Dicofol	25.51	251.0030	253.0001	1.3	35%	$^{13}C_{12}$-Methoxychlor
	Mirex	26.70	271.8102	273.8072	1.3	25%	$^{13}C_{10}$-Mirex
	$^{13}C_{10}$-Mirex	26.69	276.8269	278.8240	1.25	25%	$^{13}C_{12}$-PCB-101 (IS)
	Parlar-50	24.81	338.8649	340.8620	1.25	35%	$^{13}C_{10}$-Mirex
	Parlar-62	26.32	338.8649	340.8620	1.25	35%	$^{13}C_{10}$-Mirex

[1.] Optional GPC Clean-up Standard
[2.] Alternative m/z
[3.] Client Surrogates
[4.] Alternate quantitative reference of $^{13}C_{9}$-Endosulfan II
IS = Injection Standard

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DETERMINATION OF CONGENER SPECIFIC CHLORINATED BIPHENYLS BY HIGH RESOLUTION GCMS

Document Authorization

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Technical Authorization

Implementation Authorization

Authorized By: _____ Date: _____
Laboratory Manager

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1.0 SCOPE AND APPLICATION

1.1 Scope

This method provides procedures for the instrumental detection and quantitative measurement of chlorinated biphenyls (CBs) from extracts via isotope dilution GC/MS. [For the sample extraction, clean-up and preparation of extracts for this instrumental method see BU-TM-1110 Overall HR Prep.] Variations of this instrumental method herein and the preparative method BU-TM-1110 incorporate the concurrent analysis of selected PCDD/F congeners (see BU-TM-1113 & BU-TP-2113).

The method covers the ultra trace analysis (ppq to ppt) of extracts prepared from most standard environmental or biologically related samples including but not limited to matrices such as:

Food
- Meats
- Fish
- Eggs
- Cereals
- Dairy
- Edible Oils
- Processed Foods
- Fresh Fruits and Vegetables

Feeds
Blood & Blood Products
Other Biota & Tissues
Food Additives & Nutrients
Air
- Ambient
- Stack

Environmental Solids
- Soils
- Sediments etc.

Water
- Potable
- Non-Potable

Waste Materials

This document covers the analysis of CBs via the reference method US EPA Office of Water, Method 1668A and 1668C. Although EPA 1668C is the more recent method, some regulations still require EPA 1668A.

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The CBs that can be determined by this Method are the 12 polychlorinated biphenyls (PCBs) designated as toxic by the World Health Organization (WHO) plus the remaining 197 CBs. The 12 PCBs designated as toxic by WHO (Toxics; also known as dioxin-like PCBs; DLPCBs) and the earliest and latest eluting congener at each level of chlorination (LOC CBs) are determined by the isotope dilution quantitation technique; the remaining congeners are determined by the internal standard quantitation technique. This method allows determination of the PCB toxicity equivalent (TEQPCB) for the Toxics in a sample using toxicity equivalency factors and allows unique determination of 19 of 21 CBs of interest to the National Oceanic and Atmospheric Administration (NOAA). This method also allows estimation of homolog totals by level of chlorination (LOC) and estimation of total CBs in a sample by summation of the concentrations of the CB congeners and congener groups. The list of 209 CBs is given below with the Toxics, the CBs of interest to NOAA, and the LOC CBs identified.

No single GC column can uniquely resolve all 209 congeners. Many GC columns are commonly used for PCB congener analysis each with differing co-elutions. Therefore each column can have its own advantages/disadvantages and applications. Columns commonly applied to PCB analysis include SPB-octyl (the column recommended as the primary column in EPA method 1668A/C), DB-5, DB-5MS, DB-1 or equivalent columns. This document does not exclude the use of any columns but recommends the column that best suits the application or contract.

The SPB-Octyl column is recommended as the primary column within EPA method 1668A/C since it was considered at the time of its publication to be the best column for resolving the 12 toxic PCBs from the non-toxic PCBs.

NOTE: With the SPB-Octyl column, the two toxic PCBs with IUPAC numbers 156 and 157 co-elute. Nevertheless since they have the same 2,3,7,8-TCDD Toxic Equivalence Factor (TEF) by WHO 1998 and WHO 2005 lists, resolving this co-elution is not necessary in order to determine the total toxic equivalency. Where necessary, PCB-156 and PCB-157 can easily be resolved with DB-1, DB-5 or DB-5MS columns.

This method is primarily designed as a high resolution mass spectrometry (HRMS) method at 10,000 resolution or better in accordance to US EPA Method 1668A/C.

The HRMS option of the method herein provides '4 identification point' level of confirmation for each analyte (see Table 6 in EU decision 2002/657/EC at http://old.eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2002:221:0008:0036:EN:PDF)

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1.2 Parameters and Limits of Reporting

1.2.1 Environmental Matrices

Reporting limits for PCB congeners in environmental matrices are presented in the table below (congener numbers as per 1668C).

Instrument calibration ranges are listed in Table 3	Water (Mat# 1) [pg/L]	Soil, Solid, Tissue, Food (Mat# 2, 45, 20, 30) [pg/g]	Air, Stack (Mat# 4, 45) [pg]
PCB 1	40.0	2.0	40.0
PCB 2	40.0	2.0	40.0
PCB 3	100.0	5.0	100.0
PCB 4	100.0	5.0	100.0
PCB 5	40.0	2.0	40.0
PCB 6	40.0	2.0	40.0
PCB 7	40.0	2.0	40.0
PCB 8	100.0	5.0	100.0
PCB 9	40.0	2.0	40.0
PCB 10	100.0	5.0	100.0
PCB 11	200.0	25.0	200.0
PCB 13/12	100.0	5.0	100.0
PCB 14	40.0	2.0	40.0
PCB 15	100.0	5.0	100.0
PCB 16	40.0	2.0	40.0
PCB 17	40.0	2.0	40.0
PCB 19	40.0	2.0	40.0
PCB 21/33	100.0	5.0	100.0
PCB 22	40.0	2.0	40.0
PCB 23	40.0	2.0	40.0
PCB 24	40.0	2.0	40.0
PCB 25	40.0	2.0	40.0
PCB 27	40.0	2.0	40.0
PCB 28/20	100.0	5.0	100.0
PCB 29/26	100.0	5.0	100.0
PCB 30/18	100.0	5.0	100.0
PCB 31	100.0	5.0	100.0
PCB 32	40.0	2.0	40.0
PCB 34	40.0	2.0	40.0
PCB 35	40.0	2.0	40.0
PCB 36	40.0	2.0	40.0
PCB 37	40.0	2.0	40.0
PCB 38	40.0	2.0	40.0
PCB 39	40.0	2.0	40.0
PCB 41/71/40	200.0	10.0	200.0
PCB 42	100.0	5.0	100.0
PCB 43	100.0	5.0	100.0
PCB 44/47/65	200.0	10.0	200.0
PCB 45/51	100.0	5.0	100.0
PCB 46	40.0	2.0	40.0
PCB 48	100.0	5.0	100.0
PCB 50/53	200.0	10.0	200.0
PCB 52	100.0	5.0	100.0
PCB 54	100.0	5.0	100.0
PCB 55	100.0	5.0	100.0
PCB 56	100.0	5.0	100.0
PCB 57	100.0	5.0	100.0
PCB 58	100.0	5.0	100.0
PCB 59/62/75	200.0	10.0	200.0
PCB 60	100.0	5.0	100.0
PCB 61/70/74/76	400.0	20.0	400.0
PCB 63	100.0	5.0	100.0
PCB 64	100.0	5.0	100.0
PCB 66	100.0	5.0	100.0

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Instrument calibration ranges are listed in Table 3	Water (Mat# 1) [pg/L]	Soil, Solid, Tissue, Food (Mat# 2, 45, 20, 30) [pg/g]	Air, Stack (Mat# 4, 45) [pg]
PCB 67	100.0	5.0	100.0
PCB 68	200.0	5.0	100.0
PCB 69/49	100.0	10.0	200.0
PCB 72	100.0	5.0	100.0
PCB 73	100.0	5.0	100.0
PCB 77	100.0	5.0	100.0
PCB 78	100.0	5.0	100.0
PCB 79	100.0	5.0	100.0
PCB 80	100.0	5.0	100.0
PCB 81	100.0	5.0	100.0
PCB 82	100.0	5.0	100.0
PCB 83/99	200.0	20.0	400.0
PCB 84	100.0	5.0	100.0
PCB 88/91	100.0	5.0	100.0
PCB 89	100.0	5.0	100.0
PCB 92	100.0	5.0	100.0
PCB 94	100.0	5.0	100.0
PCB 95	400.0	20.0	400.0
PCB 96	100.0	5.0	100.0
PCB 100/93/102/98	400.0	20.0	400.0
PCB 103	100.0	5.0	100.0
PCB 104	100.0	5.0	100.0
PCB 105	100.0	5.0	100.0
PCB 106	100.0	5.0	100.0
PCB 107	100.0	5.0	100.0
PCB 108/124	200.0	10.0	200.0
PCB 109/119/86/97/125/87	400.0	20.0	400.0
PCB 111	100.0	5.0	100.0
PCB 112	100.0	5.0	100.0
PCB 113/90/101	400.0	20.0	400.0
PCB 114	100.0	5.0	100.0
PCB 117/116/85/110/115	200.0	20.0	200.0
PCB 118	200.0	10.0	200.0
PCB 120	100.0	5.0	100.0
PCB 121	100.0	5.0	100.0
PCB 122	100.0	5.0	100.0
PCB 123	100.0	5.0	100.0
PCB 126	100.0	5.0	100.0
PCB 127	100.0	5.0	100.0
PCB 128/166	200.0	10.0	200.0
PCB 130	100.0	5.0	100.0
PCB 131	100.0	5.0	100.0
PCB 132	100.0	5.0	100.0
PCB 133	100.0	5.0	100.0
PCB 134/143	200.0	10.0	200.0
PCB 136	100.0	5.0	100.0
PCB 137/164	100.0	5.0	100.0
PCB 138/163/129	400.0	10.0	200.0
PCB 139/140	200.0	10.0	200.0
PCB 141	100.0	5.0	100.0
PCB 142	100.0	5.0	100.0
PCB 144	100.0	5.0	100.0
PCB 145	100.0	5.0	100.0
PCB 146	100.0	5.0	100.0
PCB 147/149	200.0	10.0	200.0
PCB 148	100.0	5.0	100.0
PCB 150	100.0	5.0	100.0
PCB 151/135	200.0	10.0	200.0
PCB 152	100.0	5.0	100.0
PCB 154	200.0	10.0	200.0
PCB 155	100.0	5.0	100.0
PCB 156/157	200.0	10.0	100.0

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Instrument calibration ranges are listed in Table 3	Water (Mat# 1) [pg/L]	Soil, Solid, Tissue, Food (Mat# 2, 45, 20, 30) [pg/g]	Air, Stack (Mat# 4, 45) [pg]
PCB 158	100.0	5.0	100.0
PCB 159	100.0	5.0	100.0
PCB 160	400.0	10.0	200.0
PCB 161	100.0	5.0	100.0
PCB 162	100.0	5.0	100.0
PCB 165	100.0	5.0	100.0
PCB 167	100.0	5.0	100.0
PCB 168/153	200.0	10.0	200.0
PCB 169	100.0	5.0	100.0
PCB 170	100.0	5.0	100.0
PCB 171/173	200.0	10.0	200.0
PCB 172	100.0	5.0	100.0
PCB 174	100.0	5.0	100.0
PCB 175	100.0	5.0	100.0
PCB 176	100.0	5.0	100.0
PCB 177	100.0	5.0	100.0
PCB 178	100.0	5.0	100.0
PCB 179	100.0	5.0	100.0
PCB 180/193	200.0	10.0	200.0
PCB 181	100.0	5.0	100.0
PCB 182	100.0	5.0	100.0
PCB 183	200.0	10.0	200.0
PCB 184	100.0	5.0	100.0
PCB 185	200.0	10.0	200.0
PCB 186	100.0	5.0	100.0
PCB 187	100.0	5.0	100.0
PCB 188	100.0	5.0	100.0
PCB 189	100.0	5.0	100.0
PCB 190	100.0	5.0	100.0
PCB 191	100.0	5.0	100.0
PCB 192	100.0	5.0	100.0
PCB 194	100.0	5.0	100.0
PCB 195	100.0	5.0	100.0
PCB 196	100.0	5.0	100.0
PCB 197	200.0	10.0	200.0
PCB 198/199	200.0	10.0	200.0
PCB 200	200.0	10.0	200.0
PCB 201	100.0	5.0	100.0
PCB 202	200.0	10.0	200.0
PCB 203	100.0	5.0	100.0
PCB 204	100.0	5.0	100.0
PCB 205	100.0	5.0	100.0
PCB 206	100.0	5.0	100.0
PCB 207	100.0	5.0	100.0
PCB 208	100.0	5.0	100.0
PCB 209	100.0	10.0	200.0

1.2.2 Food Matrices

Detection limits (DL) and limits of Quantification (LOQ) for PCDD/F in food matrices for validated analyses via GC/HRMS are presented in the table below.

In regards to the CFIA RFSO #39903-150123/B and standing offers derived from this solicitation, all of the DL and LOQ listed below meet the respective individual food group towards the minimum requirements as referenced in Annex "A" of the RFSO.

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	CFIA CRDL	LOQ	Dairy		Eggs		Meat		Processed Foods	
			Soxhlet	Base Digestion	Soxhlet	Base Digestion	Soxhlet	Base Digestion	Soxhlet	Base Digestion
GC Column:			SPB-Octyl	ZB-5	SPB-Octyl	ZB-5	SPB-Octyl	ZB-5	SPB-Octyl	ZB-5
Sample Size (g):		100	20	100	20	100	20	100	20	100
DL Determination Method:		MDL	MDL	MDL	MDL	MDL	MDL	MDL	MDL	MDL
Target Analytes	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g
PCB-1	1	0.4	0.05	0.02	0.05	0.02	0.05	0.02	0.05	0.02
PCB-3	1	0.4	0.09	0.04	0.09	0.04	0.09	0.04	0.09	0.04
PCB-4	1	0.4	0.07	0.12	0.07	0.12	0.07	0.12	0.07	0.12
PCB-8	1	0.4	0.18	0.14	0.18	0.14	0.18	0.14	0.18	0.14
PCB-10	1	0.4	0.09	0.12	0.09	0.12	0.09	0.12	0.09	0.12
PCB-15	1	0.4	0.13	0.06	0.13	0.06	0.13	0.06	0.13	0.06
PCB-18	0.5	0.4	0.22	0.07	0.22	0.07	0.22	0.07	0.22	0.07
PCB-19	0.5	0.4	0.07	0.01	0.07	0.01	0.07	0.01	0.07	0.01
PCB-22	0.5	0.4	0.08	0.03	0.08	0.03	0.08	0.03	0.08	0.03
PCB-28	0.5	0.4	0.10	0.07	0.10	0.07	0.10	0.07	0.10	0.07
PCB-33	0.5	0.4	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06
PCB-37	0.5	0.4	0.07	0.01	0.07	0.01	0.07	0.01	0.07	0.01
PCB-40	0.5	0.4	0.16	0.03	0.16	0.03	0.16	0.03	0.16	0.03
PCB-41	0.5	0.4	0.16	0.20	0.16	0.20	0.16	0.20	0.16	0.20
PCB-44	0.5	0.4	0.20	0.07	0.20	0.07	0.20	0.07	0.20	0.07
PCB-49	0.5	0.4	0.14	0.06	0.14	0.06	0.14	0.06	0.14	0.06
PCB-52	0.5	0.4	0.08	0.07	0.08	0.07	0.08	0.07	0.08	0.07
PCB-54	0.5	0.4	0.13	0.02	0.13	0.02	0.13	0.02	0.13	0.02
PCB-60	0.5	0.4	0.10	0.07	0.10	0.07	0.10	0.07	0.10	0.07
PCB-66	0.5	0.4	0.12	0.04	0.12	0.04	0.12	0.04	0.12	0.04
PCB-70	0.5	0.4	0.16	0.06	0.16	0.06	0.16	0.06	0.16	0.06
PCB-74	0.5	0.4	0.16	0.04	0.16	0.04	0.16	0.04	0.16	0.04
PCB-77	0.5	0.4	0.08	0.01	0.08	0.01	0.08	0.01	0.08	0.01
PCB-81	0.5	0.4	0.14	0.02	0.14	0.02	0.14	0.02	0.14	0.02
PCB-87	0.5	0.4	0.30	0.04	0.30	0.04	0.30	0.04	0.30	0.04
PCB-95	0.5	0.4	0.19	0.08	0.19	0.08	0.19	0.08	0.19	0.08
PCB-99	0.5	0.4	0.15	0.04	0.15	0.04	0.15	0.04	0.15	0.04
PCB-104	0.5	0.4	0.12	0.02	0.12	0.02	0.12	0.02	0.12	0.02
PCB-105	0.5	0.4	0.11	0.03	0.11	0.03	0.11	0.03	0.11	0.03
PCB-110	0.5	0.4	0.28	0.02	0.28	0.02	0.28	0.02	0.28	0.02
PCB-114	0.5	0.4	0.13	0.01	0.13	0.01	0.13	0.01	0.13	0.01
PCB-118	0.5	0.4	0.07	0.05	0.07	0.05	0.07	0.05	0.07	0.05
PCB-119	0.5	0.4	0.30	0.02	0.30	0.02	0.30	0.02	0.30	0.02
PCB-123	0.5	0.4	0.14	0.02	0.14	0.02	0.14	0.02	0.14	0.02
PCB-126	0.5	0.4	0.09	0.01	0.09	0.01	0.09	0.01	0.09	0.01

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	CFIA CRDL	LOQ	Dairy		Eggs		Meat		Processed Foods	
			Soxhlet	Base Digestion	Soxhlet	Base Digestion	Soxhlet	Base Digestion	Soxhlet	Base Digestion
GC Column:			SPB-Octyl	ZB-5	SPB-Octyl	ZB-5	SPB-Octyl	ZB-5	SPB-Octyl	ZB-5
Sample Size (g):		100	20	100	20	100	20	100	20	100
DL Determination Method:		MDL	MDL	MDL	MDL	MDL	MDL	MDL	MDL	MDL
Target Analytes	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g
PCB-128	0.5	0.4	0.17	0.01	0.17	0.01	0.17	0.01	0.17	0.01
PCB-129	0.5	0.4	0.18	0.01	0.18	0.01	0.18	0.01	0.18	0.01
PCB-137	0.5	0.4	0.12	0.02	0.12	0.02	0.12	0.02	0.12	0.02
PCB-138	0.5	0.4	0.18	0.05	0.18	0.05	0.18	0.05	0.18	0.05
PCB-141	0.5	0.4	0.14	0.02	0.14	0.02	0.14	0.02	0.14	0.02
PCB-149	0.5	0.4	0.23	0.03	0.23	0.03	0.23	0.03	0.23	0.03
PCB-151	0.5	0.4	0.12	0.02	0.12	0.02	0.12	0.02	0.12	0.02
PCB-153	0.5	0.4	0.09	0.03	0.09	0.03	0.09	0.03	0.09	0.03
PCB-155	0.5	0.4	0.06	0.01	0.06	0.01	0.06	0.01	0.06	0.01
PCB-156	0.5	0.4	0.14	0.02	0.14	0.02	0.14	0.02	0.14	0.02
PCB-157	0.5	0.4	0.14	0.01	0.14	0.01	0.14	0.01	0.14	0.01
PCB-158	0.5	0.4	0.13	0.02	0.13	0.02	0.13	0.02	0.13	0.02
PCB-167	0.5	0.4	0.06	0.01	0.06	0.01	0.06	0.01	0.06	0.01
PCB-168	0.5	0.4	0.09	0.04	0.09	0.04	0.09	0.04	0.09	0.04
PCB-169	0.5	0.4	0.10	0.01	0.10	0.01	0.10	0.01	0.10	0.01
PCB-170	0.5	0.4	0.17	0.05	0.17	0.05	0.17	0.05	0.17	0.05
PCB-171	0.5	0.4	0.18	0.02	0.18	0.02	0.18	0.02	0.18	0.02
PCB-177	0.5	0.4	0.15	0.02	0.15	0.02	0.15	0.02	0.15	0.02
PCB-178	0.5	0.4	0.14	0.01	0.14	0.01	0.14	0.01	0.14	0.01
PCB-180	0.5	0.4	0.30	0.02	0.30	0.02	0.30	0.02	0.30	0.02
PCB-183	0.5	0.4	0.28	0.02	0.28	0.02	0.28	0.02	0.28	0.02
PCB-187	0.5	0.4	0.21	0.04	0.21	0.04	0.21	0.04	0.21	0.04
PCB-188	0.5	0.4	0.09	0.01	0.09	0.01	0.09	0.01	0.09	0.01
PCB-189	0.5	0.4	0.09	0.02	0.09	0.02	0.09	0.02	0.09	0.02
PCB-191	0.5	0.4	0.15	0.01	0.15	0.01	0.15	0.01	0.15	0.01
PCB-193	0.5	0.4	0.30	0.03	0.30	0.03	0.30	0.03	0.30	0.03
PCB-194	0.5	0.4	0.15	0.05	0.15	0.05	0.15	0.05	0.15	0.05
PCB-199	0.5	0.4	0.50	0.02	0.50	0.02	0.50	0.02	0.50	0.02
PCB-201	0.5	0.4	0.08	0.05	0.08	0.05	0.08	0.05	0.08	0.05
PCB-202	0.5	0.4	0.20	0.02	0.20	0.02	0.20	0.02	0.20	0.02
PCB-203	0.5	0.4	0.26	0.04	0.26	0.04	0.26	0.04	0.26	0.04
PCB-205	0.5	0.4	0.20	0.03	0.20	0.03	0.20	0.03	0.20	0.03
PCB-206	0.5	0.4	0.12	0.02	0.12	0.02	0.12	0.02	0.12	0.02
PCB-208	0.5	0.4	0.10	0.02	0.10	0.02	0.10	0.02	0.10	0.02
PCB-209	0.5	0.4	0.11	0.1	0.11	0.1	0.11	0.1	0.11	0.1
Average PCB DL			0.16	0.026	0.16	0.026	0.16	0.026	0.16	0.026

CRDL = Contract required detection limits as defined in the RFSO 39903-150123/B
MDL = DL as determined by the method defined in the US Code of Registry Volume 40 Appendix B to Part 136.
LOQ = Limit of Quantification as defined by the level equivalent to the low calibration standard

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It should be noted that ALS Burlington does not generally report HRMS PCB data to pre-determined MDLs (as determined by the method defined in Pt 136 to 40CFR Appendix B) but to EDLs (Estimated Detection Limits) as determined in each instrument run and a per target basis. As such EDLs are also available and as relevant an approach to demonstrate detection limit capabilities. This approach provides limits of detection obtained from real samples with real background.

The following table provides average detection limits obtained on 10 real animal fat samples plus one laboratory method blank as determined empirically by 2.5:1 signal to noise (ie. as average EDLs) on the 70 CFIA PCB targets and using two GC columns in series (SPB-Octyl and RTX-1) from a single GC run and following the base digestion procedure. This confirms the ability to provide all CFIA contracted targets in a single GC run while readily achieving the desired detection limits.

	DL#1 pg/g	DL#2 pg/g	DL#3 pg/g	DL#4 pg/g	DL#5 pg/g	DL#6 pg/g	DL#7 pg/g	DL#8 pg/g	DL#9 pg/g	DL#10 pg/g	DL#11 pg/g	Av. DL pg/g	CFIA Max. DL. pg/g
PCB #001	0.06	0.05	0.05	0.05	0.04	0.04	0.04	0.06	0.07	0.07	0.07	0.05	1
PCB #003	0.06	0.06	0.05	0.05	0.04	0.05	0.05	0.06	0.08	0.07	0.08	0.06	1
PCB #004	0.71	0.55	0.45	0.53	0.35	0.43	0.30	0.37	0.74	0.59	0.66	0.5	1
PCB #008	0.33	0.27	0.24	0.26	0.20	0.24	0.17	0.20	0.41	0.30	0.35	0.3	1
PCB #010	0.32	0.26	0.21	0.23	0.18	0.21	0.16	0.18	0.39	0.29	0.34	0.3	1
PCB #015	0.33	0.28	0.24	0.27	0.22	0.26	0.19	0.21	0.44	0.31	0.37	0.3	1
PCB #018	0.08	0.11	0.09	0.10	0.09	0.09	0.11	0.09	0.16	0.11	0.15	0.1	0.5
PCB #019	0.13	0.15	0.14	0.17	0.13	0.13	0.16	0.15	0.25	0.18	0.21	0.2	0.5
PCB #022	0.05	0.07	0.04	0.05	0.06	0.07	0.05	0.04	0.11	0.06	0.11	0.06	0.5
PCB #028	0.04	0.06	0.04	0.05	0.05	0.06	0.05	0.04	0.09	0.05	0.09	0.06	0.5
PCB #033	0.04	0.06	0.04	0.05	0.06	0.07	0.05	0.04	0.10	0.06	0.10	0.06	0.5
PCB #037	0.04	0.08	0.05	0.07	0.08	0.09	0.07	0.06	0.13	0.07	0.13	0.08	0.5
PCB #040	0.46	0.35	0.33	0.36	0.31	0.33	0.27	0.32	0.50	0.45	0.46	0.4	0.5
PCB #041	0.29	0.21	0.20	0.23	0.19	0.21	0.17	0.20	0.30	0.27	0.27	0.2	0.5
PCB #044	0.22	0.34	0.27	0.28	0.24	0.31	0.25	0.23	0.44	0.36	0.45	0.3	0.5
PCB #049	0.21	0.34	0.27	0.28	0.24	0.31	0.25	0.23	0.43	0.35	0.44	0.3	0.5
PCB #052	0.22	0.35	0.27	0.28	0.24	0.30	0.25	0.22	0.44	0.36	0.45	0.3	0.5
PCB #054	0.15	0.27	0.25	0.25	0.19	0.27	0.22	0.22	0.35	0.33	0.31	0.3	0.5

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PCB #060	0.25	0.19	0.17	0.19	0.16	0.17	0.14	0.17	0.27	0.24	0.25	0.2	0.5
PCB #066	0.25	0.20	0.18	0.20	0.17	0.19	0.15	0.18	0.28	0.25	0.26	0.2	0.5
PCB #070	0.25	0.18	0.16	0.18	0.16	0.17	0.14	0.16	0.25	0.23	0.23	0.2	0.5
PCB #074	0.25	0.18	0.17	0.19	0.16	0.17	0.14	0.17	0.26	0.23	0.23	0.2	0.5
PCB #077	0.24	0.17	0.15	0.17	0.15	0.15	0.12	0.14	0.24	0.21	0.23	0.2	0.5
PCB #081	0.24	0.16	0.14	0.15	0.14	0.14	0.12	0.13	0.23	0.19	0.22	0.2	0.5
PCB #087	0.08	0.11	0.09	0.09	0.09	0.09	0.08	0.08	0.17	0.14	0.21	0.1	0.5
PCB #095	0.09	0.11	0.11	0.10	0.10	0.10	0.09	0.09	0.18	0.15	0.22	0.1	0.5
PCB #099	0.08	0.10	0.09	0.08	0.08	0.08	0.08	0.07	0.15	0.13	0.19	0.1	0.5
PCB #104	0.04	0.06	0.05	0.06	0.05	0.07	0.05	0.06	0.10	0.08	0.09	0.06	0.5
PCB #105	0.07	0.12	0.09	0.08	0.13	0.09	0.11	0.08	0.16	0.11	0.18	0.1	0.5
PCB #110	0.06	0.08	0.07	0.06	0.07	0.06	0.06	0.06	0.12	0.10	0.15	0.08	0.5
PCB #114	0.06	0.10	0.07	0.08	0.11	0.08	0.09	0.07	0.14	0.10	0.16	0.1	0.5
PCB #118	0.07	0.08	0.07	0.06	0.07	0.07	0.06	0.06	0.12	0.10	0.17	0.08	0.5
PCB #119	0.11	0.14	0.13	0.12	0.12	0.12	0.11	0.10	0.21	0.18	0.27	0.1	0.5
PCB #123	0.07	0.09	0.08	0.07	0.07	0.07	0.07	0.06	0.13	0.11	0.18	0.09	0.5
PCB #126	0.08	0.17	0.09	0.10	0.15	0.10	0.10	0.08	0.18	0.12	0.21	0.1	0.5
PCB #128	0.08	0.13	0.09	0.10	0.10	0.13	0.08	0.11	0.25	0.15	0.19	0.1	0.5
PCB #129	0.11	0.18	0.13	0.13	0.15	0.18	0.11	0.16	0.35	0.21	0.27	0.2	0.5
PCB #137	0.09	0.18	0.10	0.11	0.12	0.15	0.09	0.13	0.35	0.21	0.27	0.2	0.5
PCB #138	0.07	0.13	0.08	0.09	0.10	0.12	0.08	0.10	0.26	0.16	0.20	0.1	0.5
PCB #141	0.09	0.17	0.10	0.11	0.12	0.14	0.09	0.13	0.33	0.20	0.26	0.2	0.5
PCB #149	0.06	0.11	0.07	0.09	0.08	0.09	0.08	0.07	0.21	0.13	0.15	0.1	0.5
PCB #151	0.07	0.13	0.08	0.10	0.09	0.11	0.09	0.08	0.23	0.14	0.17	0.1	0.5
PCB #153	0.07	0.14	0.09	0.09	0.10	0.12	0.08	0.11	0.27	0.16	0.21	0.1	0.5
PCB #155	0.04	0.06	0.05	0.06	0.07	0.16	0.15	0.14	0.31	0.22	0.08	0.1	0.5
PCB #156	0.07	0.16	0.11	0.13	0.12	0.12	0.09	0.09	0.22	0.15	0.26	0.1	0.5

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PCB #157	0.08	0.18	0.12	0.14	0.13	0.13	0.09	0.09	0.24	0.16	0.28	0.1	0.5
PCB #158	0.07	0.11	0.08	0.08	0.09	0.11	0.07	0.10	0.22	0.13	0.17	0.1	0.5
PCB #167	0.07	0.12	0.07	0.08	0.09	0.09	0.06	0.08	0.18	0.10	0.16	0.1	0.5
PCB #168	0.06	0.11	0.08	0.08	0.09	0.11	0.07	0.10	0.23	0.14	0.17	0.1	0.5
PCB #169	0.09	0.20	0.13	0.15	0.15	0.15	0.11	0.11	0.29	0.19	0.32	0.2	0.5
PCB #170	0.13	0.17	0.21	0.17	0.17	0.16	0.14	0.15	0.36	0.24	0.30	0.2	0.5
PCB #171	0.13	0.16	0.20	0.16	0.16	0.15	0.13	0.14	0.33	0.22	0.27	0.2	0.5
PCB #177	0.13	0.30	0.10	0.14	0.14	0.16	0.12	0.13	0.27	0.25	0.29	0.2	0.5
PCB #178	0.13	0.30	0.10	0.13	0.14	0.16	0.12	0.13	0.27	0.24	0.28	0.2	0.5
PCB #180	0.11	0.16	0.19	0.16	0.15	0.14	0.13	0.14	0.33	0.22	0.27	0.2	0.5
PCB #183	0.12	0.27	0.09	0.12	0.13	0.14	0.11	0.12	0.24	0.22	0.25	0.2	0.5
PCB #187	0.11	0.25	0.08	0.11	0.12	0.14	0.10	0.11	0.22	0.20	0.23	0.2	0.5
PCB #188	0.07	0.16	0.06	0.08	0.08	0.09	0.07	0.08	0.14	0.13	0.16	0.1	0.5
PCB #189	0.12	0.16	0.20	0.16	0.16	0.15	0.12	0.14	0.35	0.23	0.26	0.2	0.5
PCB #191	0.09	0.12	0.14	0.11	0.11	0.11	0.09	0.10	0.25	0.17	0.21	0.1	0.5
PCB #193	0.09	0.12	0.14	0.12	0.12	0.11	0.10	0.11	0.25	0.16	0.20	0.1	0.5
PCB #194	0.12	0.18	0.15	0.18	0.21	0.19	0.15	0.19	0.26	0.20	0.29	0.2	0.5
PCB #199	0.2	0.3	0.2	0.2	0.2	0.3	0.3	0.2	0.5	0.3	0.4	0.3	0.5
PCB #201	0.2	0.2	0.1	0.1	0.2	0.2	0.2	0.2	0.4	0.2	0.3	0.2	0.5
PCB #202	0.2	0.2	0.1	0.1	0.2	0.2	0.2	0.1	0.3	0.2	0.4	0.2	0.5
PCB #203	0.2	0.3	0.2	0.2	0.2	0.3	0.3	0.2	0.5	0.3	0.5	0.3	0.5
PCB #205	0.1	0.2	0.1	0.2	0.2	0.2	0.1	0.2	0.3	0.2	0.3	0.2	0.5
PCB #206	0.4	0.4	0.5	0.5	0.4	0.5	0.4	0.5	1.0	0.5	0.6	0.5	0.5
PCB #208	0.2	0.2	0.3	0.2	0.2	0.3	0.2	0.3	0.5	0.3	0.3	0.3	0.5
PCB #209	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.4	0.2	0.4	0.2	0.5

Reporting Limit(s)

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The reporting limits in this method can be dependent on the level of interferences and laboratory background levels rather than instrumental limitations. The estimated minimum levels of quantitation (EMLs) in Table 1 (Appendix A) are the levels at which the CBs can be determined with laboratory contamination present.

Reporting limits format can be project defined. Therefore the MDLs as determined by 40CFR Appendix B Pt 136 may not always be used to define the reporting limits. A common reporting approach, especially for HRMS data, is to report each congener or co-eluting group to 2.5:1 signal to noise (an estimated detection limit; EDL) as described in OSW SW846 Method 8290A.

1.3 Method Performance Data and Measurement Uncertainty

Refer to NA-SP-0100 Initial Method Validation – Chemistry and NA-SP-0102 Method Revalidation and MU – Chemistry for policies on determination and storage of method performance data.

2.0 PRINCIPLE OF METHOD

Samples are extracted and cleaned. Extracts are analyzed via HRMS at a resolution of 8,000-10,000 and quantified via isotope dilution and internal standard technique.

3.0 DEFINITIONS

Refer to NA-SP-0001 Definitions of Key Terms for definitions of common terms.

1. **Bias** – Characterises the systematic error in a given analytical procedure and is the (positive or negative) deviation of the mean analytical result from the (known or assumed) true value. (Eurachem Guide 1998 - IUPAC Compendium of Chemical Technology, 1985)

2. **Limit of Quantitation (LOQ)** – The lower limit of concentration or amount of substance that must be present before a method is considered to provide quantitative results. By convention, LOQ = $10s_o$, where s_o is the estimate of the standard deviation at the lowest level of measurement. (Talyor 1987). Alternatively, LOQ can be defined as the concentration equivalent to the low concentration calibration standard.

3. **Laboratory Duplicates (LD1 and LD2)** - Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of LD1 and LD2 indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures. (EPA 1994)

For isotope dilution analytical methods, there is inconsistent use of terms within the literature and reference methods. This inconsistency is avoided herein by applying terms pertaining to working standards that are defined by the timing of their addition rather than by their use in the calculation of the data.

Extraction Standards: are the labelled analytes added just prior to extraction. For the data calculations, these are true internal standards - used for determining analyte concentration.

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Bernice Kidd, Jacobs
15-April-2021

Clean-up Standards: are the labelled analytes added just prior to clean-up. Recoveries of the clean-up standard(s) provide an indication on the losses that occur during the extract clean-up and preparation for instrumental analysis.

Injection Standards: are the labelled analytes added just prior to instrumental analysis. These are used as an internal standard reference to determine the recoveries of the extraction and clean-up standards.

Sampling Standard: (also called field spikes) are the labelled analytes added, where applicable, prior to sampling onto sorbant media used for analyte capture during the sampling event.

4.0 INTERFERENCES AND MATRIX EFFECTS

Solvents, reagents, glassware, and other sample processing hardware may yield artifacts, elevated baselines, and/or lock-mass suppression causing misinterpretation of chromatograms. Where possible, reagents are cleaned by extraction or solvent rinse. Environmentally abundant CBs, as well as toxic congeners 105, 114, 118, 123, 156, 157, and 167 have been shown to be very difficult to completely eliminate from the laboratory. Proper cleaning of glassware is extremely important, because glassware may not only contaminate the samples but may also remove the analytes of interest by adsorption on the glass surface. Disposable glassware should be used where possible.

Immediately prior to use, the Soxhlet apparatus should be pre-extracted with acetone for approximately 3 hours. Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the CBs. The most frequently encountered interferences are chlorinated dioxins and dibenzofurans, methoxy biphenyls, hydroxydiphenyl ethers, benzylphenyl ethers, brominated diphenyl ethers, polynuclear aromatics, polychlorinated naphthalenes, and pesticides. High concentration hydrocarbons and phthalates can cause suppression. The interferences may apply to any or all PCB congeners. Analysis of an extract that has received additional clean up steps or dilution may be required.

5.0 SAMPLE HANDLING AND STORAGE

Parameter(s) and Matrix: PCBs - Water	
Container	Pre-cleaned amber 1L glass bottles with caps or lids that are lined with polytetrafluoroethene (PTFE) Reference: 1668C Section 6.1.1
Preservative	80mg/L of sodium thiosulphate to remove residual chlorine by ALS upon receipt. See BU-WI-0325 Water Preservation. Reference: 1668C Section 8.2.2
Lab Storage Conditions	>0 - 6°C Reference: 1668C Section 8.2.3
Maximum Sample Holding Time (Provinces / Reference)	365 days Reference: 1668C Section 8.5.1
Maximum Extract / Digest Holding Time (Provinces / References)	365 days @ ≤ -10°C Reference: 1668C Section 8.5.2

Parameter(s) and Matrix: PCBs – Soil/Sediment/Solid/Waste (including Non-perishable Feed/Foods/Nutraceuticals)

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Parameter(s) and Matrix: PCBs – Soil/Sediment/Solid/Waste (including Non-perishable Feed/Foods/Nutraceuticals)	
Container	Pre-cleaned amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE) 250mL preferred if sufficient sample available Reference: 1668C Section 6.1.1
Lab Storage Conditions	>0-6°C in the dark Reference: 1668C Section 8.3.2
Maximum Sample Holding Time (Provinces / Reference)	365 days Reference: 1668C Section 8.5.1
Maximum Extract / Digest Holding Time (Provinces / References)	365 days @ ≤ -10°C Reference: 1668C Section 8.5.2

Parameter(s) and Matrix: PCBs – Tissue (including perishable Feed/Foods/Nutraceuticals)	
Container	Pre-cleaned amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE). Solid tissues such as whole fish can be wrapped in solvent cleaned aluminum foil. Reference: 1668C Section 6.1.1
Lab Storage Conditions	<-10°C in the dark Reference: 1668C Section 8.4.3
Maximum Sample Holding Time (Provinces / Reference)	365 days Reference: 1668C Section 8.5.1
Maximum Extract / Digest Holding Time (Provinces / References)	365 days @ ≤ -10°C Reference: 1668C Section 8.5.2

Parameter(s) and Matrix: PCBs – Blood and Blood products	
Container	Plastic test tubes Reference: CDC Laboratory Procedure Manual
Lab Storage Conditions	<-80°C in the dark Reference: CDC Laboratory Procedure Manual
Maximum Sample Holding Time (Provinces / Reference)	365 days Reference: 1668C Section 8.5.1
Maximum Extract / Digest Holding Time (Provinces / References)	365 days @ ≤ -10°C Reference: 1668C Section 8.5.2

ALS requirements are to store all samples and extracts in the dark and to comply with the method recommended hold times and temperature storage requirements. Short term storage of extracts (i.e. prior to the completion of analysis) is in a refrigerator at 4°C. Long term storage of extracts is below -10°C.

5.1 Sampling Instructions

Sample collection is not conducted by the laboratory.

5.2 Sampling Handling and Transportation

Glass Petri dishes are not recommended for transportation of filters to and from the field since these are susceptible to breakage during transportation. Instead amber glass wide mouthed bottles are recommended.

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Refer to ALS Sample Handling and Preservation Guidelines and BU-SP-0261 Sample Control, Login and Receipt for further details on the sample handling procedures recommended by ALS.

If holding time violations occur, Account Manager(s) must be notified.

6.0 EQUIPMENT AND SUPPLIES

Alternative equivalent equipment and supplies may be substituted for those listed below if suitability is demonstrated.

6.1 Primary Equipment

1. **Autosamplers**

Capable of dependable automated injection of liquid extracts down to 1µl, including pre- and post-injection automated syringe rinsing. Example:

- CTC GC-PAL GC-xt Autosampler or functionally equivalent or superior, as provided with, or upgraded on, Waters Autospec HRMS system.

2. **Gas Chromatographs**

Capable of temperature programming with isothermal hold. Electronic Pressure Control recommended but not essential. Example:

- Agilent 7890 GC

3. **High Resolution Mass Spectrometers**

Capable of repetitive and selective monitoring of at least 12 exact m/z's at high resolution (10,000) during a period of approximately one second. Example:

- Waters Autospec Premier HRMS

Specific and detailed instrument operational settings and conditions are listed in the HRMS application notes BU-WI-3201 PCB Octyl HRMS-Autospec Premier, BU-WI-3105a PCB by HRMS - Premier CFIA Methods, and BU-WI-3105b PCB HT8 Saudi Arabia HRMS – Premier..

6.2 Support Equipment

1. Rotovap
2. Centrifuge
3. N-Evap

6.3 Software

1. TargetLynx/MassLynx v4.1

6.4 Supplies

6.4.1 Sample Prep Suppliers

See BU-TM-1110 for supplies

6.4.2 Instrument Supplies

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1. Focusing column (4m x Intermediate Polarity Fused Silica or equivalent; Canadian Life Sciences OV6250350-105, MEOL# 3080182) coupled to SPB Octyl 30±5m x 0.25±0.02mm id x 0.25 um film thickness 30-m long × 0.25-mm ID; 0.25-um film (Supelco 24218-U, MEOL# 4320155, or equivalent). This column system is capable of meeting the requirements as outlined in the 'NOTE' below.

From 1668C:
NOTE: Any GC column or column system (2 or more columns) may be used that provides unique resolution and identification of the Toxics for determination of a TEQPCB using TEFs. Isomers may be unresolved so long as they have the same TEF and response factor and so long as these unresolved isomers are uniquely resolved from all other congeners. For example, the SPB-octyl column achieves unique GC resolution of all Toxics except congeners with IUPAC numbers 156 and 157. This isomeric pair is uniquely resolved from all other congeners and these congeners have the same TEF and response factor.

The column must uniquely resolve congeners 34 from 23 and 187 from 182, and congeners 156 and 157 must coelute within 2 seconds at the peak maximum. Unique resolution means a valley height less than 40 percent of the shorter of the two peaks that result when the diluted combined 209 congener solution is analyzed. The column must be serviced or replaced when any of the above criteria in are not met. If a column or column system alternate to the SPB-octyl column is used, specifications similar to those for the SPB-octyl column must be developed and be functionally equivalent to those specifications.

6.4.3 Standard Preparation Supplies

1. 5 µL-5000 µL syringes, Hamilton, MEOL # for various syringes can be found in G:\Enviro\Purchasing\Organic Prep\Approved Supplies.xls (Syringes Tab)
2. 2 mL Robovials, Chromagraphic Specialities MEOL #4001399
3. Robovial Lids, Chromagraphic Specialities MEOL #4001399
4. 15 mL Centrifuge tubes, VWR MEOL# 4000187
5. 5 0 mL Centrifuge tubes, VWR MEOL# 4000202
6. 7 mL Graduated screw cap vials (MEOL# 4000687) with solid PTFE lined caps (MEOL# 4010593)
7. 22 mL Graduated screw cap vials (MEOL# 4000738) with solid PTFE lined caps (MEOL# 4010609)

7.0 REAGENTS AND STANDARDS

Refer to BU-SP-0250 Control of Reagents and Reference Materials.

7.1 Reagents

1. Nonane, Glass Distilled, Stored at ambient temperature. Stored in standard fumehood. Expiry date: 3 months following distillation. Supplier: Caledon; Supplier# CAL 1368-05, MEOL# 6010079,

2. Methanol, Stored at ambient temperature. Stored in flammable materials storage cabinet. Expiry date: 1year following date of receipt, 3 months after opening. Supplier: Caledon, Supplier # 6701-2-40MEOL # 6010026.

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3. Perfluorokerosene, Either high-boiling or low-boiling may be used. Stored at ambient temperature. Expiry date: 5 years from received date. Supplier: Cambridge Isotope Laboratories; Supplier# PFK-HIGH-1, MEOL# 5005748.

4. Helium, Ultra High Purity, Expiry date: 6 months. Supplier: Praxair; HE 5.0UH-T, ordered through Praxair website.

5. Perfluorotributylamine (PFTBA). Store at ambient temperature. Expiry: 5 years following the year of receipt. Supplier: Agilent, Supplier # 8500-0656, MEOL# 6040032

7.2 Standards

All standards are stored in the fridge. Unless otherwise indicated, the default expiry date is 1 year, or longer if revalidation is done. Expiry dates are stated on the supplier certificate of analysis.

7.2.1 Extraction Standards

7.2.1.1 Extraction Standard Supplier Information

Compound	PCB	Supplier	Catalogue #	MEOL #
13C12-2-Chlorobiphenyl	1L	Wellington	MBP-1	5005079
13C12-4-Chlorobiphenyl	3L	Wellington	MBP-3	5004617
13C12-2,2'-Dichlorobiphenyl	4L	Wellington	MBP-4	5005907
13C12-4,4'-Dichlorobiphenyl	15L	Wellington	MBP-15	5005908
13C12-2,2',6-Trichlorobiphenyl	19L	Wellington	MBP-19	5005909
13C12-3,4,4'-Trichlorobiphenyl	37L	Wellington	MBP-37	5005910
13C12-2,2',6,6'-Tetrachlorobiphenyl	54L	Wellington	MBP-54	5005911
13C12-3,3',4,4'-Tetrachlorobiphenyl	77L	Wellington	MBP-77	5004422
13C12-3,4,4',5-Tetrachlorobiphenyl	81L	Wellington	MBP-81	5005090
13C12-2,2',4,6,6'-Pentachlorobiphenyl	104L	Wellington	MBP-104	5005912
13C12-2,3,3',4,4'-Pentachlorobiphenyl	105L	Wellington	MBP-105	5005080
13C12-2,3,4,4',5-Pentachlorobiphenyl	114L	Wellington	MBP-114	5005081
13C12-2,3',4,4',5-Pentachlorobiphenyl	118L	Wellington	MBP-118	5005082
13C12-2',3,4,4',5-Pentachlorobiphenyl	123L	Wellington	MBP-123	5005083
13C12-3,3',4,4',5-Pentachlorobiphenyl	126L	Wellington	MBP-126	5005084
13C12-2,2',4,4',6,6'-Hexachlorobiphenyl	155L	Wellington	MBP-155	5005913
13C12-2,3,3',4,4',5-Hexachlorobiphenyl	156L	Wellington	MBP-156	5005085
13C12-2,3,3',4,4',5'-Hexachlorobiphenyl	157L	Wellington	MBP-157	5005086
13C12-2,3',4,4',5,5'-Hexachlorobiphenyl	167L	Wellington	MBP-167	5005087
13C12-3,3',4,4',5,5'-Hexachlorobiphenyl	169L	Wellington	MBP-169	5005088
13C12-2,2',3,4',5,6,6'-Heptachlorobiphenyl	188L	Wellington	MBP-188	5005914
13C12-2,3,3',4,4',5,5'-Heptachlorobiphenyl	189L	Wellington	MBP-189	5005089
13C12-2,2',3,3',5,5',6,6'-Octachlorobiphenyl	202L	Wellington	MBP-202	5004618
13C12-2,3,3',4,4',5,5',6-Octachlorobiphenyl	205L	Wellington	MBP-205	5005915
13C12-2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	206L	Wellington	MBP-206	5005916
13C12-2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl	208L	Wellington	MBP-208	5005917
13C12-Decachlorobiphenyl	209L	Wellington	MBP-209	5004619

7.2.1.2 Extraction Stock Standard Solution Preparation (1668-ES$1-xxx)

- Using a syringe, add 4.6 mL of nonane to a c-tube.
- Using a syringe, add 200 µL of each compound listed in 7.2.1.1 to the nonane
- The final volume should be 10 mL. The final concentration should be 1000 ng/mL
- Aliquot into robovials
- Name the standard 1668A-ES$1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

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7.2.1.3 Extraction Working Standard Solution Preparation (1668-ES#2-xxx)

- Using a syringe, add 18 mL of nonane to a c-tube
- Using a syringe, add 2000 µL of 1668-ES$1-xxx to nonane
- The final volume should be 20 mL. The final concentration should be 100 ng/mL
- Aliquot into robovials
- Name the standard 1668A-ES#2-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.2 Cleanup Standards

7.2.2.1 Cleanup Standard Supplier Information

Compound	PCB	Supplier	Catalogue #	MEOL #
13C12-2,4,4'-Trichlorobiphenyl	28L	Wellington	MBP-28	5005091
13C12-2,3,3',5,5'-Pentachlorobiphenyl	111L	Wellington	MBP-111	5006076
13C12-2,2',3,3',5,5',6-Heptachlorobiphenyl	178L	Wellington	MBP-178	5007222

7.2.2.2 Cleanup Stock Standard Solution Preparation (1668-CL$1-xxx)

- Using a syringe, add 940 µL of nonane to a robovial
- Using a syringe, add 20 µL of each compound listed in 7.2.2.1 to the nonane
- The final volume should be 1 mL. The final concentration should be 1000 ng/mL
- Name the standard 1668A-CL$1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.2.3 Cleanup Working Standard Solution Preparation (1668-CL#2-xxx)

- Using a syringe, add 19.88 mL of nonane to a c-tube
- Using a syringe, add 40 µL of each compound listed in Section 7.2.2.1, to the nonane
- The final volume should be 20 mL. The final concentration should be 100 ng/mL
- Aliquot into robovials
- Name the standard 1668A-CL#2-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.3 Injection Standards

7.2.3.1 Injection Standard Supplier Information

Compound	PCB	Supplier	Catalogue #	MEOL #
13C12-2,5-Dichlorobiphenyl	9L	Wellington	MBP-9	5005092
13C12-2,2',5,5'-Tetrachlorobiphenyl	52L	Wellington	MBP-52	5004572
13C12-2,2',4,5,5'-Pentachlorobiphenyl	101L	Wellington	MBP-101	5004571
13C12-2,2',4,4',5'-Hexachlorobiphenyl	138L	Wellington	MBP-138	5004709
13C12-2,2',3,3',4,4',5,5'-Octachlorobiphenyl	194L	Wellington	MBP-194	5005093

7.2.3.2 Injection Standard Supplier Information (Additional Targets)

Compound	DX	Supplier	Catalogue #	MEOL #
13C12-1,2,3,7,8,9-HxCDD	1,2,3,7,8,9-HxCCD	Wellington	MDD-123789	5005097
13C12-1,2,3,4-TCDD	1,2,3,4-TCDD	Wellington	MDD-1234	5005094

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7.2.3.3 Injection Stock Standard Solution Preparation (1668A-IS$1-xxx)

- Using a syringe, add 250 µL of nonane to a robovial
- Using a syringe, add 50 µL of each compound listed in 7.2.3.1 to the nonane
- The final volume should be 500 µL. The final concentration should be 5000 ng/mL.
- Name the standard 1668A-IS$1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.3.4 Injection Working Standard Solution Preparation (1668A-IS#2-xxx)

- Using a syringe, add 6200 µL of nonane to a c-tube
- Using a syringe, add 50 µL of each compound listed in 7.2.3.1 to the nonane
- The final volume should be 6250 µL. The final concentration should be 400 ng/mL.
- Aliquot into robovials
- Name the standard 1668A-IS#2-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.3.5 Injection Working Standard Solution Preparation (DXPCB-IS#1-xxx)

- Using a syringe, add 9440 uL of nonane to a c-tube
- Using a syringe, add 80 uL of each compound listed in 7.2.3.1 to the nonane
- Using a syringe, add 80 uL of each compound listed in 7.2.3.2 to the nonane
- The final volume should be 10 mL. The final concentration should be 400 ng/mL.
- Aliquot into robobials
- Name the standard DXPCB-IS#1-xxx where xxx represents a sequential lot number, and refers to a specific preparation

7.2.3.6 Diluted Injection Working Standard Solution Preparation (1668A-IS#2-xxx 4x diln)

- For some PCB analyses a diluted injection standard is required.
- Using a syringe, add 2250 µL of nonane to a c-tube
- Using a syringe add 750 µL of 1668A-IS#2-xxx to the nonane
- The final volume should be 3000 µL. The final concentration should be 100 ng/mL.
- Aliquot into robovials
- Name the standard 1668A-IS#2-xxx 4x diln, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.4 Field Spike Standards

7.2.4.1 Field Spike Standard Supplier Information

Compound	PCB	Supplier	Catalogue #	MEOL #
2,4',5-Trichloro[13C12]biphenyl	31L	Wellington	MBP-31	5004718
2,2',3,5',6-Pentachloro[13C12]biphenyl	95L	Wellington	MBP-95	5004719
2,2',4,4',5,5-Hexachloro[13C12]biphenyl	153L	Wellington	MBP-153	5004606

7.2.4.2 Field Spike Stock Standard Solution Preparation (1668A-FS$1-xxx)

- Using a syringe add 940 µL of nonane to a robovial
- Using a syringe add 20 µL of each compound listed to the nonane
- The final volume should be 1000 µL. The final concentration should be 1000 ng/mL.

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- Name the standard 1668A-FS$1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.4.3 Field Spike Working Standard Solution Preparation (1668A-FS#2-xxx)

- Using a syringe, add 9940 µL of nonane to a c-tube
- Using a syringe, add 20 µL of each compound listed to the nonane
- The final volume should be 10000 µL. The final concentration should be 100 ng/mL.
- Aliquot into robovials
- Name the standard 1668A-FS#2-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.5 Native Standards

7.2.5.1.1. AccuStandard Native Mix Catalogue#1668A-C-NT-LOC-WD, MEOL#5002675

Compound	PCB
2-Chlorobiphenyl	1
4-Chlorobiphenyl	3
2,2'-Dichlorobiphenyl	4
4,4'-Dichlorobiphenyl	15
2,2',6-Trichlorobiphenyl	19
2,3,5-Trichlorobiphenyl	23
2',3,5-Trichlorobiphenyl	34
3,4,4'-Trichlorobiphenyl	37
2,2',6,6'-Tetrachlorobiphenyl	54
3,3',4,4'-Tetrachlorobiphenyl	77
3,4,4',5-Tetrachlorobiphenyl	81
2,2',4,6,6'-Pentachlorobiphenyl	104
2,3,3',4,4'-Pentachlorobiphenyl	105
2,3,4,4',5-Pentachlorobiphenyl	114
2,3',4,4',5-Pentachlorobiphenyl	118
2',3,4,4',5-Pentachlorobiphenyl	123
3,3',4,4',5-Pentachlorobiphenyl	126
2,2',4,4',6,6'-Hexachlorobiphenyl	155
2,3,3',4,4',5-Hexachlorobiphenyl	156
2,3,3',4,4',5'-Hexachlorobiphenyl	157
2,3',4,4',5,5'-Hexachlorobiphenyl	167
3,3',4,4',5,5'-Hexachlorobiphenyl	169
2,2',3,3',4,4',5-Heptachlorobiphenyl	170
2,2',3,4,4',5,5'-Heptachlorobiphenyl	180
2,2',3,4,4',5,6'-Heptachlorobiphenyl	182
2,2',3,4',5,5',6-Heptachlorobiphenyl	187
2,2',3,4',5,6,6'-Heptachlorobiphenyl	188
2,3,3',4,4',5,5'-Heptachlorobiphenyl	189
2,2',3,3',5,5',6,6'-Octachlorobiphenyl	202
2,3,3',4,4',5,5',6-Octachlorobiphenyl	205
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	206
2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl	208
Decachlorobiphenyl	209

7.2.5.1.2. Wellington Native Mix Catalogue#68C-PAR, MEOL#5006139

Compound	PCB
2-Chlorobiphenyl	1
4-Chlorobiphenyl	3
2,2'-Dichlorobiphenyl	4
4,4'-Dichlorobiphenyl	15
2,2',6-Trichlorobiphenyl	19
2,3,5-Trichlorobiphenyl	23

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2',3,5-Trichlorobiphenyl	34
3,4,4'-Trichlorobiphenyl	37
2,2',6,6'-Tetrachlorobiphenyl	54
3,3',4,4'-Tetrachlorobiphenyl	77
3,4,4',5-Tetrachlorobiphenyl	81
2,2',4,6,6'-Pentachlorobiphenyl	104
2,3,3',4,4'-Pentachlorobiphenyl	105
2,3,4,4',5-Pentachlorobiphenyl	114
2,3',4,4',5-Pentachlorobiphenyl	118
2',3,4,4',5-Pentachlorobiphenyl	123
3,3',4,4',5-Pentachlorobiphenyl	126
2,2',4,4',6,6'-Hexachlorobiphenyl	155
2,3,3',4,4',5-Hexachlorobiphenyl	156
2,3,3',4,4',5'-Hexachlorobiphenyl	157
2,3',4,4',5,5'-Hexachlorobiphenyl	167
3,3',4,4',5,5'-Hexachlorobiphenyl	169
2,2',3,3',4,4',5-Heptachlorobiphenyl	170
2,2',3,4,4',5,5'-Heptachlorobiphenyl	180
2,2',3,4,4',5,6'-Heptachlorobiphenyl	182
2,2',3,4',5,5',6-Heptachlorobiphenyl	187
2,2',3,4',5,6,6'-Heptachlorobiphenyl	188
2,3,3',4,4',5,5'-Heptachlorobiphenyl	189
2,2',3,3',5,5',6,6'-Octachlorobiphenyl	202
2,3,3',4,4',5,5',6-Octachlorobiphenyl	205
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	206
2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl	208
Decachlorobiphenyl	209

7.2.5.2 Native Stock Standard Solution Preparation (1668A-NS$1-xxx)

- Using a syringe, add 9.99 mL of nonane to a c-tube
- Using a syringe, add 10 µL of AccuStandard Native Mix, Section 7.2.5.1.1 to the nonane
- The final volume should be 10 mL. The final concentration should be 20 ng/mL.
- Aliquot into robovials
- Name the standard 1668A-NS$1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.5.3 Native Working Standard Solution Preparation (1668A-NS#1-xxx)

- Using a syringe, add 9750 µl of nonane to a c-tube
- Using a syringe, add 250 µL of Wellington Native Mix, Section 7.2.5.1.2 to the nonane
- The final volume should be 10 mL. The final concentration should be 50 ng/mL.
- Aliquot into robovials
- Name the standard 1668A-NS#1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.6 Calibration Standards

7.2.6.1 Calibration Standard Supplier Information

All stock solutions for the calibration standards are highlighted in sections 7.2.1 to 7.2.5.

7.2.6.2 Calibration Standard CS0 Preparation (1668A-CS0-xxx)

Type of Standard	Source	Amount Added (µL)	Final Concentration (ng/mL)

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Native	1668A-NS$1	10	0.2
Extraction	1668A-ES$1	100	100
Clean up	1668A-CL$1	100	100
Injection	1668A-IS$1	20	100
Field Spike	1668A-FS$1	100	100

- Using a syringe, add 670 µL of nonane to a robovial
- Using a syringe, add standards listed to the nonane
- The final volume should be 1 mL. The final concentrations should be as shown.
- Name the standard 1668A-CS#0-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.6.3 Calibration Standard CS1 Preparation (1668A-CS1-xxx)

Type of Standard	Source	Amount Added (µL)	Final Concentration (ng/mL)
Native	1668A-NS$1	50	1
Extraction	1668A-ES$1	100	100
Clean up	1668A-CL$1	100	100
Injection	1668A-IS$1	20	100
Field Spike	1668A-FS$1	100	100

- Using a syringe, add 630 µL of nonane to a robovial
- Using a syringe, add standards listed to the nonane
- The final volume should be 1 mL. The final concentrations should be as shown.
- Name the standard 1668A-CS#1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.6.4 Calibration Standard CS2 Preparation (1668A-CS2-xxx)

Type of Standard	Source	Amount Added (µL)	Final Concentration (ng/mL)
Native	1668A-NS$1	250	5
Extraction	1668A-ES$1	100	100
Clean up	1668A-CL$1	100	100
Injection	1668A-IS$1	20	100
Field Spike	1668A-FS$1	100	100

- Using a syringe, add 430 µL of nonane to a robovial
- Using a syringe, add standards listed to the nonane
- The final volume should be 1 mL. The final concentrations should be as shown.
- Name the standard 1668A-CS#2-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.6.5 Calibration Standard CS3 Preparation (1668A-CS3-xxx)

Type of Standard	Source	Amount Added (µL)	Final Concentration (ng/mL)
Native	M-1668A-C-NT-LOC-WD	5	50
Extraction	1668A-ES$1	200	100
Clean up	1668A-CL$1	200	100
Injection	1668A-IS$1	40	100

- Using a syringe, add 1355.0 µL of nonane to a c-tube
- Using a syringe, add standards listed to the nonane
- The final volume should be 2 mL. The final concentrations should be as shown.
- Aliquot into robovials
- Name the standard 1668A-CS#3-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

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7.2.6.6 Calibration Standard CS4 Preparation (1668A-CS4-xxx)

Type of Standard	Source	Amount Added (µL)	Final Concentration (ng/mL)
Native	M-1668A-C-NT-LOC-WD	20	400
Extraction	1668A-ES$1	100	100
Clean up	1668A-CL$1	100	100
Injection	1668A-IS$1	20	100
Field Spike	1668A-FS$1	100	100

- Using a syringe, add 660 µL of nonane to a robovial
- Using a syringe, add standards listed to the nonane
- The final volume should be 1 mL. The final concentrations should be as shown.
- Name the standard 1668A-CS#4-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.6.7 Calibration Standard CS5 Preparation (1668A-CS5-xxx)

Type of Standard	Source	Amount Added (µL)	Final Concentration (ng/mL)
Native	M-1668A-C-NT-LOC-WD	100	2000
Extraction	1668A-ES$1	100	100
Clean up	1668A-CL$1	100	100
Injection	1668A-IS$1	20	100
Field Spike	1668A-FS$1	100	100

- Using a syringe, add 580 µL of nonane to a robovial
- Using a syringe, add standards listed to the nonane
- The final volume should be 1 mL. The final concentrations should be as shown.
- Name the standard 1668A-CS#5-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.7 Window/GC Resolution Standard

7.2.7.1 Calibration Standard Supplier Information

Expiry dates are stated on the supplier label.

Standard	PCB	Supplier	Catalogue #	MEOL #
M-1668A-1-0.01X	See 7.2.7.2	AccuStandard	M-1668A-0.01X-SET	5000760
M-1668A-2-0.01X	See 7.2.7.2			
M-1668A-3-0.01X	See 7.2.7.2			
M-1668A-4-0.01X	See 7.2.7.2			
M-1668A-5-0.01X	See 7.2.7.2			

7.2.7.2 PCB Congeners in M-1668A-0.01X Set (as per 1668C)

M-1668A-1-0.01X	2, 10, 9, 6, 8, 14, 11, 30, 27, 32, 34, 26, 31, 33, 36, 38, 35, 50, 45, 52, 49, 75, 41, 72, 57, 63, 66, 79, 78, 81, 96, 103, 95, 88, 89, 92, 113, 83, 119, 87, 85, 82, 120, 124, 106, 122, 105, 127, 152, 136, 148, 151, 144, 143, 142, 133, 161, 153, 130, 129, 166, 159, 167, 156, 179, 176, 178, 175, 183, 177, 171, 172, 191, 170, 190, 201, 204, 200, 198, 196, 195, 194, 207
M-1668A-2-0.01X	7, 5, 12, 18, 24, 23, 28, 22, 39, 53, 51, 73, 48, 62, 71, 68, 58, 61, 55, 60, 94, 100, 91, 121, 90, 99, 109, 117, 111, 108, 118, 114, 150, 145, 135, 149, 139, 132, 165, 168, 137, 160, 128, 162, 157, 184, 186, 187, 185, 181, 192, 197, 199, 203
M-1668A-3-0.01X	13, 17, 29, 20, 46, 65, 59, 40, 67, 76, 80, 93, 84, 101, 112, 86, 116, 107, 154, 147, 140, 146, 141, 164, 158, 182, 174, 173, 193
M-1668A-4-0.01X	25, 21, 69, 47, 42, 64, 70, 102, 97, 115, 123, 134, 131, 163, 180
M-1668A-5-0.01X	1, 3, 4, 15, 19, 16, 37, 54, 43, 44, 74, 56, 77, 104, 98, 125, 110, 126, 155, 138, 169, 188, 189, 202, 205, 208, 206, 209

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7.2.7.3 Window/GC Resolution Calibration Standard CS7 Preparation (1668A-CS7-xxx)

Type of Standard	Source	Amount Added (µL)	Final Concentration (ng/mL)
Native	M-1668A-1-0.01X M-1668A-2-0.01X M-1668A-3-0.01X M-1668A-41-0.01X M-1668A-5-0.01X	10 10 10 10 10	25* 25* 25* 25* 25*
Extraction	1668A-ES$1	100	100
Clean up	1668A-CL$1	100	100
Injection	1668A-IS$1	20	100
Field Spike	1668A-FS$1	100	100

*This is the concentration for the mon- through tri- congeners. For tetra- through hepta- congeners, final concentrations are 100ng/ml, and for octa- through deca-, final concentrations are 150 ng/ml.

- Using a syringe, add 630 µL of nonane to a robovial
- Using a syringe, add standards listed to the nonane
- The final volume should be 1 mL. The final concentrations should be as shown.
- Name the standard 1668A-CS#7-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.8 Reference Standards

7.2.8.1 Reference Standard for Initial Calibration Preparation (1668A-RS#1-xxx)

Type of Standard	Source	Amount Added (µL)	Final Concentration (ng/mL)
Native	68C-PAR	25	50
Extraction	1668A-ES$1	100	100
Clean up	1668A-CL$1	100	100
Injection	1668A-IS$1	20	100
Field Spike	1668A-FS$1	100	100

- Using a syringe, add 655 µL of nonane to a robovial
- Using a syringe, add standards listed to the nonane
- The final volume should be 1 mL. The final concentrations should be as shown.
- Name the standard 1668A-RS#1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

7.2.8.2 Agilent Native Mix Catalogue# RPCM-245-1 MEOL# 5007846

Compound	PCB	Concentration (ng/ml)
2,2',4-trichlorobiphenyl	17	500
2,2',5-trichlorobiphenyl	18	2000
2,4,4'-trichlorobiphenyl	28	2000
2,4',5-trichlorobiphenyl	31	1500
2',3,4-trichlorobiphenyl	33	2000
2,2',3,5'-tetrachlorobiphenyl	44	2000
2,2',4,5'-tetrachlorobiphenyl	49	2000
2,2',5,5'-tetrachlorobiphenyl	52	2000
2,3',4',5-tetrachlorobiphenyl	70	2000
2,4,4',5-tetrachlorobiphenyl	74	2000
2,2',3,3',4-tetrachlorobiphenyl	82	500
2,2',3,4,5'-pentachlorobiphenyl	87	2000
2,2',3,5',6-pentachlorobiphenyl	95	1000
2,2',4,4',5-pentachlorobiphenyl	99	2000
2,2',4,5,5'-pentachlorobiphenyl	101	2000
2,3,3',4,4'-pentachlorobiphenyl	105	500
2,3,3',4',6-pentachlorobiphenyl	110	2000
2,3',4,4',5-pentachlorobiphenyl	118	2000

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2,2',3,3',4,4'-hexachlorobiphenyl	128	2000
2,2',3,3',4,6'-hexachlorobiphenyl	132	1000
2,2',3,4,4',5'-hexachlorobiphenyl	138	2000
2,2',3,4',5',6-hexachlorobiphenyl	149	2000
2,2',3,4,4',6-hexachlorobiphenyl	151	2000
2,2',3,3',5,5'-hexachlorobiphenyl	153	2000
2,3,3',4,4',5-hexachlorobiphenyl	156	2000
2,3,3',4,4',6-hexachlorobiphenyl	158	500
3,3',4,4',5,5'-hexachlorobiphenyl	169	2000
2,2',3,3',4,4',5-heptachlorobiphenyl	170	2000
2,2',3,3',4,4',6-heptachlorobiphenyl	171	2000
2,2',3,3',4',5,6-heptachlorobiphenyl	177	2000
2,2',3,4,4',5,5'-heptachlorobiphenyl	180	2000
2,2',3,4,4',5',6-heptachlorobiphenyl	183	2000
2,2',3,4'5,5',6-heptachlorobiphenyl	187	2000
2,3,3',4,4',5',6-heptachlorobiphenyl	191	2000
2,2',3,3',4,4',5,5'-octachlorobiphenyl	194	2000
2,2',3,3',4,4',5,6-octachlorobiphenyl	195	2000
2,2',3,3',4,5,5',6'-octachlorobiphenyl	199	1500
2,3,3',4,4',5,5',6-octachlorobiphenyl	205	2000
2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206	2000
2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208	2000
Decachlorobiphenyl	209	2000

7.2.8.3 Reference Standard for 209 Mix Preparation (1668A-RS#2-xxx)

Type of Standard	Source	Amount Added (µL)	Final Concentration (ng/mL)
Native	RPCM-241-1	50	25-100
Extraction	1668A-ES$1	100	100
Clean up	1668A-CL$1	100	100
Injection	1668A-IS$1	20	100
Field Spike	1668A-FS$1	100	100

- Using a syringe, add 630 µL of nonane to a robovial
- Using a syringe, add standards listed in the table to the nonane
- The final volume should be 1 mL. The final concentrations should be as shown.
- Name the standard 1668A-RS#2-xxx, where xxx represents a sequential lot number, and refers to a specific preparation

8.0 QUALITY CONTROL

8.1 Method QC

Maximum (Sample Preparation) Batch Size =	20 client samples

Method QC	Min. Frequency	Data Quality Objective (units)
Method Blank (MB)[1]	1 per batch	< 2 times the Estimated Minimum Level (EML) from Table 1, or < ½ regulatory compliance limit, or < 1/10th of target in any client sample in that batch
Laboratory Control Sample (LCS)[2] [aka Clean Matrix Spike or On-going Precision and Recovery (OPR)]	1 per batch	See Table 2
Reference Material (RM)	Upon Client Request	As per supplier

[1] For DoD sample analysis, MB criteria is <1/2 x LOQ or 1/10th of what is in the samples or 1/10th of the regulatory limit whichever is largest.
[2] PCB LCS control limits for DoD analysis are listed in Table 7.

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15-April-2021

Matrix Spike (MS) and Matrix Spike Duplicate (MSD)[3]	Upon Client Request	%RPD < 50% for targets spiked at >10x the incurred sample concentration
Extraction Standards (SURR)	All samples and QC samples	See Table 2
Clean-up Standards	All samples and QC samples	See Table 2
Sampling Standards	Air: all samples spiked prior to submission of sampling apparatus to client	These standards are not included in the reference methods. DQO are determined based on contractual requirements
Sample Duplicate (DUP)	All matrices expect water: 1 per batch, or as specified in contract or client request	%RPD < 50% for targets detected above the LOQ
Procedural Blank (PB)	1 per batch*	Visual Assessment of Contamination

*If all samples from a batch require further cleanup, the QC samples must be included. If only selected samples require further cleanup, a procedural blank must be created which will receive the prescribed cleanup to ensure no contamination has been acquired through the additional steps.

8.2 Instrument QC

Maximum Batch Size =	Every 12 hours (every shift)

Instrument QC	Minimum Frequency	Control Limit
Initial Calibration Curve (IC)[4]	As required to maintain a valid initial calibration as demonstrated by required CCV injections	The %RSD of each target must be less than or equal to 20% for utilization of the average RRF for calibration.
Calibration Verification Standard (CVS) (Section 7.2.8)	Run together with every initial calibration	Assessed vs. Quality control criteria for a CCV standard, or other applicable corporate DQO criteria
Continuing Calibration Verification (CCV)[5] (CS3)	Every 12 hours	Table 3 m/z abundance within limits of Table 5
Instrument Sensitivity Check[6] (CS0)	Run together with every initial calibration, or more frequently due to contractual requirements	Assessed vs, quality control criteria for a CS1 standard and CCV standard
Instrument Blank	Every analytical batch. Run before Method Blank.	Visually assessed to be free of carry-over contamination from previous injection of standard or Laboratory Control Sample
Single-Point 209 Calibration Mix	Every 12 hours	This injection is used for RRT calibration for

[3] MS/MSD is to be evaluated relative to the acceptance criteria listed in Table 7, for DoD analysis.

[4] If any calibration criteria are not met, system must be evaluated and corrective action must be taken; the entire initial calibration procedure must be repeated before sample analysis begins.

[5] If all acceptance criteria are met for the Continuing Calibration Verification Standard, then the initial calibration can be used for the analysis of additional samples. In the event of a CCV failure during DoD sample analysis, proceed with the following corrective action:
- Immediately analyze 2 additional consecutive CCVs, both must meet acceptance criteria in order for samples to be reported without reanalysis.
- If these do not pass, recalibration must occur and all samples since the last acceptable CCV must be reanalyzed.

[6] If the Instrument Sensitivity Check Standard does not meet CCV acceptance criteria, it must be assessed whether the instrument is sensitive enough to meet the contract required detection limits for any samples to be analyzed on that instrument.

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Instrument QC	Minimum Frequency	Control Limit
		that analytical shift, and for single point Relative Response calibration for all congeners not included in the Initial Calibration
Chromatographic Performance	Every 12 hours	The 209 Calibration mix is also assessed for minimum chromatographic separation criteria (See section 6.4.1)
MS resolution (HRMS option only)	Start and end of each shift	10,000 for each quantification window; ≥8,000 throughout the mass range and must be ≥10,000 in the center of the mass range for each function

Corrective Actions for Recovery Limit Failures:

a) Extraction Standards:

Evaluate source of the recovery failure. Actions may include re-injection, clean-up/analysis of an archived portion of the raw extract, re-extraction or flagging of the data. Unless there is clear evidence for a matrix bias, every reasonable effort should be taken to resolve a failed extraction recovery issue. When simpler corrective actions such as re-injection do not resolve the issue, re-extraction or, where there is insufficient sample available, analysis of an archived portion of the raw extract should be the appropriate course. If the recovery bias is repeatable, then this constitutes clear evidence for a matrix bias, no further action is required and the data can be flagged appropriately and reported.

b) Clean-up Standards:

Clean-up recoveries represent a tool to help assess whether a difficulty originates from the extraction of the sample, or the clean-up of the extract. Other than evaluation of the data set, failures in clean-up recoveries do not force a corrective action.

c) Injection Standards:

A low injection standard response indicates a missed injection. A repeat injection is the most common course of action. A high response indicates extract concentration. A review of the extract final volume is required. Otherwise, a low or high recovery indicates a potential compromise to the handling of the sample extract or interference. Unless there is a clear explanation for the failure, then re-extraction/ re-analysis or analysis of an archived portion of the extract is necessary.

d) Sampling Standards:

For stack and ambient sampling, the entire sample is consumed in the extraction. Therefore re-extraction/re-analysis is not normally an option. Re-injection and/or analysis of an archived portion of the extract are the appropriate corrective actions.

9.0 TEST PROCEDURE

9.1 Sample Preparation

See BU-TM-1110 Overall HR Prep.

9.2 Dilution Procedure

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9.2.1 The target analyte concentrations in the sample extract must be within the calibrated range of the instrument.

9.2.1.1 See BU-SP-0263 for Dilution policy and general procedures.

9.2.2 If a sample is diluted 100x or greater, then spike with ES and IS at the regular amount. The results will no longer be recovery corrected; therefore, a comment will need to be made on the report.

9.2.3 If the sample is in a microvial and the final volume will be 500 µL or less, then a measured amount of solvent (toluene or distilled nonane) is added directly to the uncapped microvial. Re-cap and vortex the sample

9.2.4 If a greater dilution is required or the sample is for more than one analyses, then vortex the sample. Remove the cap. Using a clean syringe, put an aliquot into a new, labeled microvial. Cap and vortex the new microvial.

NOTE: If a sample is suspected of being high level prior to analysis, an instrument blank is run immediately after it or the high level sample can be put at the end of the run.

9.3 Additional Cleanups & Reworks (Procedural Blank Generation)

If a sample requires additional work in the lab, a procedural blank is generated at the instrument, which accompanies the samples and the rework document as it goes back out into the prep lab. The procedure below outlines how to generate a procedural blank.

1) Refer to the sample batch sheet to identify the injection and extraction standard types and amounts used in generating the batch.
2) Retrieve the corresponding injection and extraction standards from the standards fridge.
3) A new vial is obtained, and labelled as PB-xxx (where xxx is a sequential, unique numerical identifier)
4) Using proper syringe techniques, spike the appropriate amount of each standard into the procedural blank vial.
5) Submit the procedural blank vial, alongside the sample(s) that require additional work, to the prep lab.

9.4 Instrumentation

9.4.1 System Initialization & Maintenance

Refer to BU-WI-3007 GCMS Operation and Instrument Application Notes BU-WI-3201 PCB Octyl HRMS-Autospec Premier.

9.4.2 Instrument Operating Parameters

The operation of the HRMS instrument is described in Work Instruction BU-WI-3007 GCMS Operation. Specific MS conditions and parameters and GC operating conditions can be found in Application Notes (BU-WI-3201 PCB Octyl HRMS-Autospec, BU-WI-3105a PCB by HRMS - Premier CFIA Methods, and BU-WI-3105b PCB HT8 Saudi Arabia HRMS – Premier).

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The exact masses monitored, and suggested acquisition descriptor parameters are shown in Table 6. Note that depending on the instrument used, alternate or additional PFK masses may be used (for example, on the Thermo DFS instrument, each analytical segment requires three PFK masses – one each for lock and calibration masses, and another to be monitored by the data processing software for lock mass and response drift). The exact MS acquisition methods used by each instrument are referenced in Application Notes (BU-WI-3201 PCB Octyl HRMS-Autospec Premier, BU-WI-3105a PCB by HRMS - Premier CFIA Methods, and BU-WI-3105b PCB HT8 Saudi Arabia HRMS – Premier).

The maximum time between scans within a descriptor is 1 second.

9.3.3 Mass spectrometer (MS) resolution

Using PFK (or other reference substance) and a molecular leak, tune the instrument to meet the minimum required resolving power of 10,000 (10% valley) at m/z 293 or any other significant PFK fragment in the range of 280 to 350. For each descriptor (Table 6), monitor and record the resolution and exact m/z's of at least three reference peaks covering the mass range of the descriptor. The level of PFK (or other reference substance) metered into the HRMS during analyses should be adjusted so that the amplitude of the most intense selected lock-mass m/z signal (regardless of the descriptor number) does not exceed 10% of the full-scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

Note: Different lots and types of PFK can contain varying levels of contamination, and excessive PFK (or other reference substance) may cause noise problems and contamination of the ion source necessitating increased frequency of source cleaning. It is recommended that an adequate level of PFK be employed for instrument calibration, and then reduced to the minimum amount required to maintain mass lock, in order to reduce noise, particularly for the dichlorobiphenyls M+2.

The analysis time for CBs may exceed the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, mass-drift correction is mandatory and a lock-mass m/z from perfluorokerosene (PFK) or other reference substance is used for drift correction. The lock-mass m/z is dependent on the exact m/z's monitored within each descriptor, as shown in Table 6. The deviation between the exact m/z and the theoretical m/z (Table 6) for each exact m/z monitored must be less than 5 ppm.

9.4.4 Instrument Calibration

Establish the operating conditions necessary to achieve, at a minimum, the chromatographic separations as outlined in Table 1. Note that these conditions are applicable only to the focusing (4m x Restek Intermediate Polarity Fused Silica or equivalent) coupled to SPB-Octyl GC column chemistry. Different chromatographic criteria should be established when an alternate GC column phase is used.

9.4.4.1 GC operating conditions

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See Application Notes (BU-WI-3201 PCB Octyl HRMS-Autospec Premier, BU-WI-3105a PCB by HRMS - Premier CFIA Methods, and BU-WI-3105b PCB HT8 Saudi Arabia HRMS – Premier) for the operating conditions for each instrument. The conditions may be optimized for compound separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, blanks, IPR and LCS standards, and samples.

9.4.4.2 Retention time calibration for the CB congeners

1. Separately inject each of the diluted individual congener solutions.
2. Establish the beginning and ending retention times for the scan descriptors in Table 6. Scan descriptors other than those listed in Table 6 may be used provided the MLs in Table 1 are met.
3. The absolute retention time of CB 209 must exceed 55 minutes on the SPB-octyl column if required by the contract; otherwise, the GC temperature program must be adjusted and this test repeated until the minimum retention time criterion is met.
4. If required by the contract, adjust the chromatographic conditions and scan descriptors until the RT and RRT for all congeners are within the windows in 1668 Table 2 and the column performance specifications in Sections 6.4.1 are met.
5. If an alternate column system is used, adjust the conditions for that system. If system performance is unacceptable, optimize the analysis conditions or replace the column system and repeat the performance tests. See Section 12.0 subsection 2. A different column system has been developed that meets all of the separation criteria of the Octyl column, therefore Appendix A Table 1 lists the current conditions.
6. Confirm that the scan descriptor changes at times when CBs do not elute.

9.4.5 Run Processing

9.4.5.1 Samples in autosampler

1. Place samples extracts and standards in microvials onto the autosampler tray on the GC/MS instrument.
2. The autosampler injects 1 to 2 µL into the instrument for each instrumental run. The injection volume and run conditions must be the same for all injections within each 12 hour run sequence.

9.4.5.2 Run sequence set up

A typical continuous run sequence is listed below:

1A) CS7 diluted combined 209 congener solution[1]
2A) CS3
3A) QC samples spiked at the CS3 level or higher
4A) One or more method blanks and/or a system blanks[3]
5A) Samples
6A) CS3
1B) CS7 diluted combined 209 congener solution*
2B) QC samples spiked at the CS3 level or higher
3B) One or more method blanks and/or a system blanks[3]
4B) Samples
5B) CS3
1C).....

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1. 1A and 2A can be switched.
2. Optionally, or if the continuing calibration does not meet method control limits, an initial calibration can be injected as item (2).
3. EPA 1668C allows the injection of an instrument blank before analysis of the Method Blank, in order to minimize the impact of instrument carryover on assessment of the Method Blank.

Additional Considerations

- The 1A to 6A portion of the continuous sequence represents the first 12-hour run sequence. The timing of the last injection of each 12-hour sequence must not be greater than 12 hours following the first injection, either the calibration verification CS3 or 209 congener solution. Before and after each 12 hour sequence a mass resolution check must demonstrate resolution of 10,000 (refer to section 9.3.6.1). Instrument conditions must remain the same during each 12-hour sequence and no other injections can take place between the last injection of the 12 hour sequence and the ending mass resolution check.

- When a multi-point calibration initiates a 12-hour run sequence, then the injection of the first standard represents the start of the 12-hour sequence.

9.4.6 System Performance

At the beginning of each 12-hour shift during which analyses are performed, GC/MS system performance and calibration are verified for all native CBs and labelled compounds. For these tests, analysis of the CS-3 continuing calibration verification (CCV) standard (Table 3) and/or the diluted combined 209 congener solution (Table 4) must be used to verify all performance criteria. Sample and QC extract results may be reported if the performance criteria have been met. If there are DQO failures, the potential impact to data quality must be assessed and affected data appropriately qualified, otherwise adjustment and/or recalibration (Section 9.3.4) must be performed until all performance criteria are met

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9.4.6.1 MS resolution: Static resolving power checks must be performed at the beginning and at the end of each shift to demonstrate a resolution of 10,000 for each quantification window. Because of the extensive mass range covered in each function, it may not be possible to maintain 10,000 resolution throughout the mass range during the function. Therefore, resolution must be ≥8,000 throughout the mass range and must be ≥10,000 in the center of the mass range for each function. If analyses are performed on successive shifts, only the beginning of shift static resolving power check is required. If the requirement in Section 9.3.3 cannot be met before the run, the problem must be corrected before analyses can proceed. If any of the samples in the previous shift may be affected by poor resolution, as demonstrated by the end-of-shift resolution check, those samples must be assessed to determine the need for re-analysis. The expectation is for the 10,000 resolution to be maintained over the run sequence to help ensure an absence of chemical interferences while failures in the criterion normally initiate re-injection as a corrective action. Mass resolution of 10,000 is not always required in order to obtain quality data, therefore if the mass resolution check fails at the end of the sequence, the data may be reviewed carefully for a) chemical interferences and b) deterioration in instrument sensitivity. If upon review there is an absence of concern for real compromise to analyte data quality, then re-injection of the impacted samples is not required and the data may be reported.

9.4.6.2 Continuing Calibration Verification (CCV): Inject the CS-3 standard or diluted combined 209 congener solution using the procedure in Section 9.3.5. The m/z abundance ratios for all CBs must be within the limits in Table 5; otherwise, the mass spectrometer must be adjusted until the m/z abundance ratios fall within the limits specified when the verification test is to be repeated. If the adjustment alters the resolution of the mass spectrometer, resolution must be verified (9.3.3) prior to repeat of the verification test. The GC peak representing each native CB and labelled compound in the standard must be present with a S/N of at least 10; otherwise, the mass spectrometer must be adjusted and the verification test repeated. For each compound, compare the concentration with the calibration verification limit in Table 1. If all compounds meet the acceptance criteria, calibration has been verified and analysis of standards and sample extracts may proceed. If, however, any compound fails its respective limit, the measurement system is not performing properly. In this event, prepare a fresh calibration standard or correct the problem and repeat the resolution and verification tests, or recalibrate. If recalibration is required, recalibration for the 209 congeners must also be performed.

It is possible that in some cases the daily calibration verification may pass the acceptance criteria, but the values for some native and/or labelled congeners are still far enough from the initial calibration mean RRFs, that sample and/or QC data are negatively impacted. For determination of the most accurate analyte data, the analyst may choose as an acceptable option to calculate target Native Toxic/LOC/WD analyte data from the average RRF_{ID} of the daily pre- and post-calibration verification runs, and labelled analyte data from the average RRF_{LIS} (see Section 10.1) from the daily pre- and post- calibration runs, and the daily diluted 209 congener solution run.

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9.4.6.3 Relative Retention Times (RRT): The relative retention times of native CBs and labelled compounds in the verification test must be within their respective RRT limits in Table 1 or, if an alternate column, column system, or set of GC conditions is employed, within their respective RRT limits for the alternate column, column system or set of GC conditions.

9.4.6.4 Absolute Retention Time (RT): The absolute retention times of the Labelled Toxics/LOC/window defining standard congeners in the CCV must be within ±15 seconds of the respective retention times in the initial calibration.

9.4.6.5 Laboratory Control Sample (LCS): Analyze the extract of the LCS prior to analysis of samples from the same batch. Compute the percent recovery of the Toxics/LOC CBs by isotope dilution. Compute the percent recovery of each labelled compound by the internal standard method.

For the Toxics/LOC CBs and labelled compounds, compare the recovery to the LCS limits given in Table 2. If all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may proceed. If, however, any individual concentration falls outside of the range given, the extraction/concentration processes are not being performed properly for that compound. In this event, correct the problem, re-prepare, extract, and clean up the sample batch and repeat the ongoing precision and recovery test.

10.0 DATA CALCULATION AND REPORTING

10.1 Qualitative Analysis

The following criteria must be met for a positively identified response to labelled or unlabelled analytes on all field samples, QC samples and calibration runs. Failures on any one of these items for a calibration run means that the calibration run is out of control for that analyte. For calibration runs, Instrument control must be regained by re-injection or instrument maintenance before samples can be analysed. Sample failures indicate the presence of interferences or responses inadequate for qualitative or quantitative determination.

10.1.1 Ion Ratio Criteria

The ratio of the integrated areas of the two exact m/z's specified in Table 5 must be within the limit in Table 5, or within ±15 percent of the ratio in the midpoint (CS-3) CCV or CVS, whichever is most recent.

10.1.2 Signal to Noise Criteria

The signal-to-noise ratio (S/N) for the GC peak at each exact m/z must be greater than or equal to 2.5 for each CB detected in a sample extract, and greater than or equal to 10 for all CBs in the calibration and verification standards.

10.1.3 Matched RT on Peak Maxima

The signals for the two exact m/z's in Table 6 must be present and must maximize within two seconds.

10.1.4 Retention Time (RT) Match between Native to Labelled Analyte

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The relative retention time of the peak for a CB must be within the RRT QC limits specified in Table 1 or, if an alternate column or column system is employed, within its respective RRT QC limits for the alternate column or column system.

10.1.5 Ambiguous Identification

Because of congener overlap and the potential for interfering substances, it is possible that all of the identification criteria (Sections 10.1.1 to 10.1.4) may not be met. It is also possible that loss of one or more chlorines from a highly chlorinated congener may inflate or produce a false concentration for a less-chlorinated congener that elutes at the same retention time. If identification is ambiguous, an experienced spectrometrist must determine the presence or absence of the congener (1668C 16.5)

If the criteria for identification in Sections 10.1.1 to 10.1.4 are not met, the CB has not been identified and the result for that congener may not be reported or used for permitting or regulatory compliance purposes. If interferences preclude identification, a new aliquot of sample must be extracted, further cleaned up, and analyzed.

10.2 Isotope Dilution and Internal Standard Calibration

The calibration scheme of this method involves a 3-part approach:

- Isotope dilution calculation is performed to establish Relative Response Factors for the subset of native CB congeners which are included in the multipoint initial calibration. This includes the native toxic and the Level-Of-Chlorination Window-Defining congeners, to be henceforth referred to as the native "Toxic/LOC/WD CBs". This calculation technique uses the multipoint, multilevel initial calibration to establish the Isotope Dilution Relative Response Factor (RRF_{ID}) of each Native Toxic/LOC/WD CB vs. its exact labelled analogue.

- Multipoint Internal Standard calculation is performed to establish Relative Response Factors for the labelled analogues of the Toxic/LOC/WD CBs, the labelled cleanup congeners (and sampling standards where used), and for determination of the labelled injection internal standards except for $^{13}C_{12}$-PCB-138. This calculation is also performed on the initial calibration data, and is based on a multipoint calibration to establish the Labelled Internal Standard Relative Response Factor (RRF_{LIS}) of each labelled congener vs. its designated quantitation reference standard.

- Single-point Internal Standard calculation is used for determination of native congeners that are not included in the Toxic/LOC/WD CB subset. This calculation technique is based on a daily single-point calibration using the diluted 209 congener mix, to determine the Native Internal Standard Relative Response Factor (RRF_{NIS}) of each applicable native congener (or coeluting native congener cluster) vs. the average internal standard response (the mean response of the chosen quantitation reference standards at the same level of chlorination).

The RRFs established above can then be averaged for each target to compute calibration tables, which are used to calculate the concentrations of native and labeled congeners in the extracts of samples and QC samples.

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10.2.1 Isotope Dilution Calibration

For the isotope dilution calculation of a native Toxic/LOC/WD CB, the response factor of the native congener relative to its labelled analogue, and factored for concentration (Isotope Dilution Relative Response Factor, RRF_{ID}) is computed. The responses are determined using the area responses of both the primary and secondary exact m/z's specified in Table 6, as follows:

$$RRF_{ID} = \frac{(A1_n + A2_n)C_L}{(A1_L + A2_L)C_n}$$

Where:

$A1_n$ and $A2_n$ - The areas of the primary and secondary m/z's for the Native Toxic/LOC/WD CB in the initial calibration standard

$A1_L$ and $A2_L$ - The areas of the primary and secondary m/z's for the Labelled Toxic/LOC/WD CB

C_L - The concentration of the labelled Toxic/LOC/WD CB in the calibration standard (Table 3).

C_n - The concentration of the Native Toxic/LOC/WD CB in the calibration standard (Table 3).

To run an Initial Calibration of the analytical system by isotope dilution, a 5- or 6-point calibration encompassing the concentration range is prepared for each Toxic/LOC/WD CB congener (the instrument calibration standard concentrations are presented in Table 3). Inject calibration standards CS1 (or CS-0.2) through CS-5. Compute and store the Isotope Dilution Relative Response Factor, RRF_{ID} for each Native Toxic/LOC/WD CB at each concentration. Compute the average Isotope Dilution Relative Response Factor $^{AVG}RRF_{ID}$ and the RSD of the 5 (or 6) RRF_{ID}s. If the RRF_{ID} for any Native Toxic/LOC/WD CB is constant across the concentration range (less than 20% RSD), the $^{AVG}RRF_{ID}$ may be used to quantify that congener in extracts; otherwise, the complete calibration curve for that congener must be used over the calibration range.

10.2.2 Multipoint Labelled Internal Standard Calibration

For the multipoint internal standard calculation of a Labelled Toxic/LOC/WD CB, the response factor of the labelled congener relative to its designated internal standard, and factored for concentration (Labelled Internal Standard Relative Response Factor RRF_{LIS}) is computed. The reference, or internal standard for each compound is listed in Table 1. The responses are determined using the area responses of both the primary and secondary exact m/z's specified in Table 6, as follows:

$$RRF_{LIS} = \frac{(A1_L + A2_L)C_{is}}{(A1_{is} + A2_{is})C_L}$$

Where:

$A1_L$ and $A2_L$ - The areas of the primary and secondary m/z's for the Labelled Toxic/LOC/WD CB

$A1_{is}$ and $A2_{is}$ - The areas of the primary and secondary m/z's for the internal standard (see Table 1 for the reference, or internal standard for each compound).

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C_{is} - The concentration of the internal standard (Table 3).

C_L - The concentration of the labelled Toxic/LOC/WD CB in the calibration standard (Table 3).

To calibrate the analytical system by multipoint internal standard calibration, a 5- or 6-point calibration is run as a combined analysis during the Initial Calibration of natives by isotope dilution. The Labelled Toxic/LOC/WD CB congeners are also in each calibration standard at a single level of concentration (the instrument calibration standard concentrations are presented in Table 3). Inject calibration standards CS1 (or CS-0) through CS-5. Compute and store the Labelled Internal Standard Relative Response Factor (RRF_{LIS}) for each Labelled Toxic/LOC/WD CB in each calibration standard except for $^{13}C_{12}$-PCB-138. For the Labelled Toxics/LOC/WD compounds and the Labelled cleanup standards, use the nearest eluted Labelled injection internal standard as the quantitation reference, as given in Table 1. The Labelled injection internal standards are referenced to CB 138, as shown in Table 1. Compute the average Labelled Internal Standard Relative Response Factor $^{AVG}RRF_{LIS}$ for all 5 (or 6) calibration points.

10.2.3 Single-point Labelled Internal Standard Calibration

Native CBs that are not included in the Toxic/LOC/WD subset do not have their own labelled standard, and are not part of the multi-level calibration set. The diluted 209 Congener Standard is run daily, and these CBs are calculated by single-point internal standard calculation. The response factor of the native congener (or coeluting congener cluster) is calculated relative to the average internal standard response at that level of chlorination, and factored for concentration (Native Internal Standard Relative Response Factor RRF_{NIS}). The responses are determined using the area responses of both the primary and secondary exact m/z's specified in Table 6, as follows:

$$RRF_{NIS} = \frac{(A1_{209} + A2_{209})C_L}{^{AVG}(A1_L + A2_L)C_{209}}$$

Where:

$A1_{209}$ *and* $A2_{209}$ - The areas of the primary and secondary m/z's for the CB or CB congener cluster in the diluted 209 Congener Standard

$^{AVG}(A1_L + A2_L)$- The average of the areas of the primary and secondary m/z's for the chosen labelled internal standards at that level of chlorination (See Table 1 for the quantitation references for each target)

C_L - The concentration of the internal standard (Table 4).

C_{209} - The concentration of the native CB(s) in the diluted 209 calibration standard (Table 4).

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To perform the single-point calibration of the analytical system for native CBs other than the Native Toxic/LOC/WD CBs by internal standard, inject the diluted combined 209 congener solution (Table 4). Compute and store the Native Internal Standard Relative Response Factor (RRF_{NIS}) for all native CBs that are not part of the Native Toxic/LOC/WD subset. As described in the above formula, the average of the chosen internal standard responses at each level of chlorination (LOC) is used as the quantitation reference for targets at that LOC, as shown in Table 1. For the combinations of isomeric congeners that co-elute, compute a combined RRF_{NIS} for the co-eluted group. For example for congener 122, the areas at the two exact m/z's for 104L, 114L, 123L, 118L are summed and the total area is divided by 4 (because there are 4 chosen labeled congeners used as the quantitation reference). Note: Multiple labelled congeners at each LOC are used as reference to reduce the effect of an interference possible if a single congener is used as reference. Other quantitation references and procedures may be used provided that the results produced are as accurate as results produced by the quantitation references and procedures described in this Section.

Initial Calibration vs. daily calibration runs - In certain cases, it may be desirable to compute the RRFs of the daily pre- and post- calibration verification, and diluted 209 congener runs, and use the average of those values of RRF_{ID} and RRF_{LIS} for the Native Toxic/LOC/WD CBs and labelled CBs in that day's run (see Section 9.3.6.2). Note that the natives that are not part of the Toxic/LOC/WD subset are already routinely calibrated using the RRF_{NIS} from the daily calibration.

10.3 Calculation of Extract and Sample Concentration

10.3.1 Calculation of natives from calibration Response Factors

By adding a known amount of the Labelled Toxics/LOC/WD compounds to every sample prior to extraction, correction for extraction recovery of CBs can be made because the native compounds and their exact labelled analogs exhibit similar effects upon extraction, concentration, and gas chromatography (the internal standard calibration of natives that do not have their own labelled analogue assumes overall similar behavior of those natives compared to their chosen quantitation references). Average Relative Response Factors ($^{AVG}RRF_{ID}$ or $^{AVG}RRF_{NIS}$ calculated in Section 10.2.1 and 10.2.3 are used in conjunction with chromatographic peak responses and known labelled standard added amounts, to determine concentrations in the final extract. Compute the concentrations in the extract of the native targets using the following equations as appropriate:

For Native Toxic/LOC/WD targets:

$$C_{ex}\ (ng/mL) = \frac{(A1_{NS} + A2_{NS})C_L}{(A1_L + A2_L)^{AVG}RRF_{ID}}$$

For other natives calibrated using the daily diluted 209 Congener Standard:

$$C_{ex}\ (ng/mL) = \frac{(A1_{NS} + A2_{NS})\ C_L}{^{AVG}(A1_L + A2_L)RRF_{NIS}}$$

where:

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$A1_{NS}$ and $A2_{NS}$ - The areas of the primary and secondary m/z's for the Native Toxic/LOC/WD CB in the extract

C_{ex} = The concentration of the PCB in the extract

The other terms are defined in Section 10.2.1 and 10.2.3.

10.3.2 Calculation of Concentration in Samples

10.3.2.1 The concentration of a native CB in the solid phase of the sample is computed using the concentration of the compound in the extract and the weight of the solids as follows:

$$\text{Concentration in solid (ng/kg)} = \frac{(C_{ex} \times V_{ex})}{Ws}$$

where:

C_{ex} =The concentration of the compound in the extract from 10.3.1.
V_{ex} = The extract volume in mL.
Ws = The sample weight (dry weight) in kg.

10.3.2.2 The concentration of a native CB in the aqueous phase of the sample is computed using the concentration of the compound in the extract and the volume of water extracted as follows:

$$\text{Concentration in aqueous phase (pg/L)} = \frac{(C_{ex} \times V_{ex})}{Vs}$$

where:

C_{ex} = The concentration of the compound in the extract from 10.3.1.
V_{ex} = The extract volume in mL.
Vs = The sample volume in liters.

10.3.3 Labelled Internal Standard Quantitation and Labelled Compound Recovery

Compute the concentrations in the extract of the Labelled Toxic/LOC/WD standard, the Labelled Cleanup Standard (and labeled Sampling Standard where used), and the Labelled Injection Internal Standard (except for $^{13}C_{12}$-PCB-138) using the Average Labelled Internal Standard Relative Response Factors ($^{AVG}RRF_{LIS}$) determined from the calibration data (Section 10.2.2) and the following equation:

$$C_{ex}\ (ng/mL) = \frac{(A1_{LS} + A2_{LS})C_{is}}{(A1_{is} + A2_{is})^{AVG}RRF_{LIS}}$$

where:

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$A1_{LS}$ and $A2_{LS}$ - The areas of the primary and secondary m/z's for the labeled target in the extract

C_{ex} = The concentration of the PCB (non-Toxic/LOC native congener, or Toxic/LOC labelled congener)

The other terms are defined in Section 10.2.2.

Using the concentration in the extract determined above, compute the percent recovery of the Labelled Toxics/LOC/window-defining CBs and the Labelled cleanup standard CBs (and Sampling Standards where used) using the following equation:

$$\text{Recovery (\%)} = \frac{\text{Concentration found (ng/mL) x 100}}{\text{Concentration spiked (ng/mL)}}$$

10.3.4 Reporting samples that have been filtered

For water samples containing solids, they are filtered and the filtrate and particulate are separately extracted. However, the extraction standard used for quantification is added to the sample prior to filtering and the two separate extracts are combined prior to analysis. Therefore, data are reported for the whole sample, using the sample volume prior to filtering and the results represent the concentration for the whole sample.

10.4 Data Transfer to LIMS

Refer to BU-WI-3225 Reporting HRMS Results.

10.5 Data Validation

Peer review and sign off is a requirement, refer to NA-SP-0175 Data Validation for details.

10.5.1 Method Blank Failure

If the blank level exceeds the DQO (see Section 8.0) for any analyte, then the corrective action is to re-analyze any sample within the batch that has a positively determined analyte with up to 10-fold higher response of the blank level and, for regulatory compliance, if the biased analyte in the sample is within a factor of 10 of the regulatory compliance limit.

10.5.2 LCS Failure

When the LCS recoveries are outside the recommended ranges, an evaluation of the impact on the data quality is required. Failure(s) may indicate compromise to one or more of the standards or to the extraction/clean-up process. If the evaluation shows significant compromise to the reported data quality for one or more samples in the batch, then the affected samples should be re-injected and/or repeated for OCP analysis from archived extract and/or from another portion of the original sample. If repeat analysis is desired and additional sample is not available for repeat, then a final evaluation of the pooled results is required to evaluate appropriate data flagging or non-reporting of the results.

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10.5.3 MS and MSD Failure

Corrective actions from failure of this criterion include, at the least, re-evaluation of the data set with respect to calculation errors or analytical compromise. Re-injection or re-analysis of the sample for MS/MSD may be considered. Re-extraction and re-analysis of the entire batch may be considered.

10.5.4 DUP Failure

Corrective actions from failure of this criterion include, at the least, examination of the sample for evidence of inhomogeneity or evaluation of the data set with respect to calculation errors and analytical compromise. For solids, sample inhomogeneity is a common source of the problem. Documented evidence for inhomogeneity may resolve a criterion failure. Re-injection or re-analysis of the sample and duplicate, or the entire batch may be considered.

10.6 Data Reporting

Results are reported to three significant figures for the CBs found in all standards, blanks, and samples. Extraction, Clean-up, and Sampling Standards are reported as a percent recovery to the nearest integer.

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1. Aqueous samples—Report results in pg/L (parts-per-quadrillion).

2. Samples containing greater than 1% solids (soils, sediments, filter cake, compost)—Report results in ng/kg based on the dry weight of the sample. Report the percent solids so that the result may be converted to dry weight solid units (typically pg/g = ng/kg).

3. Tissues—Report results in ng/kg of wet tissue, not on the basis of the lipid content of the tissue. Report the percent lipid content, so that the data user can calculate the concentration on a lipid basis if desired.

10.6.1 Reporting Level

a) Results in samples and Method Blanks for each congener or co-eluting group are reported to an Estimated Detection Limit (EDL) based on 2.5:1 signal to noise as described in OSW SW846 Method 8290A. Minimum Levels (ML) and Estimated Minimum Levels (EML) are also published in Appendix A Table 1. These published MLs were developed by the EPA from pooled interlaboratory data and are intended to serve as a guideline for achievable Minimum Levels in the laboratory. The EMLs are based on common laboratory contamination levels and are used as the acceptance criteria for Method Blank contamination as per the reference method. A laboratory may establish a ML for a CB lower than the MLs in Table 1. MLs may be established as low as the lowest calibration point (Table 3) provided that the concentration of the congener in a minimum of 10 blanks for a sample medium (e.g., water, soil, sludge, tissue) is significantly below the EML in Table 1. Significant means that the ML for the congener is no less than the average (mean) plus 2 standard deviations above the level in the minimum of 10 blanks. The blanks must be analyzed during the same period that the sample is analyzed, ideally over an approximately 1-month period.

b) Results for a CB in a sample that has been diluted are reported at the least dilute level at which the area at the quantitation m/z is within the calibration range.

c) For a CB having a labelled analog, report results at the least dilute level at which the area at the quantitation m/z is within the calibration range and the labelled compound recovery is within the normal range for the Method.

d) If requested, the total concentration of all congeners at a given level of chlorination (i.e., total TrCB, total PeCB, total HxCB) may be reported by summing the concentrations of all congeners identified at that LOC, including both the Toxics and other congeners.

e) Limit of Quantitation (LOQ): The LOQ is defined as the concentration of target in a sample which is equivalent to the lowest calibration standard (CS1, or for higher sensitivity instruments CS0). In some cases the LOQ is used as a Lower Reporting Limit (LOR) by contractual requirement, but be default estimated results between the EDL and the LOQ that meet positive ID requirements are reported as estimated values.

f) Data Qualifiers: The following data qualifiers are normally reported with the analytical results:

- M: Indicates that a peak has been manually integrated

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- U: Indicates that the analyte was not detected at or above the Estimated Detection Limit
- E: Indicates that the compound was detected at levels above the calibrated range
- J: Indicates that the analyte was positively identified at levels below the LOQ; the associated numerical result is an estimate
- R: Indicates that the ion abundance ratio for this analyte did not meet the control limit; the reported elevated detection limit is an Estimated Maximum Possible Concentration.
- B: Indicates that this target was detected in the Method Blank at greater than 10% of the sample concentration.
- 1./EMPC: User-defined flags commonly used to indicate that the Detection Limit is elevated due to a coeluting interference, and is an Estimated Maximum Possible Concentration.

g) In some situations, LORs may need to be raised. Refer to NA-SP-0104 Quality Control for more information.

Instrument and method blank subtraction are not done for this method.

10.7 Estimated Maximum Possible Concentration (EMPC)

An estimated maximum possible concentration (EMPC) is a worst-case estimate of the concentration of a CB analyte under a chemical interference. It may be described as the maximum amount of the target which could theoretically be present but cannot be separated, detected, or confirmed due to the interference. It is reported as an elevated detection limit for that compound.

10.7.1 The most common example is the case where a peak is detected which meets all of the criteria for positive identification of a target compound except for the ion abundance ratio. In this case, the results are flagged on a report with the data qualifier "R" (or other client-specific equivalent qualifier). The EMPC value in this case is a concentration calculated by supposing the ion which is too high to be affected by a coeluting interference, and adjusting its peak area to a lower extrapolated value based on the theoretical ion abundance ratio and the area of the supposed "correct" ion, and then calculating the EMPC from the extrapolated area by applying equations in section 10.

10.7.2 Another commonly occurring scenario in the analysis of PCB congeners is a false positive signal that is caused by a coeluting homologue or homologues at a higher level of chlorination. In this case the EMPC is the calculated concentration of the coeluting interference, and is flagged on a report with a user defined data qualifier, usually "1." or "EMPC", footnoted with a descriptive definition such as "This compound is reported as an EMPC due to a coeluting interference from higher level of chlorination".

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10.7.3 A third notable category of EMPC in this analysis occurs when matrix or sample related chemical interference causes either an elevated baseline or a peak at the retention time of a target compound. In this case the detection limit is elevated by increasing the noise height used for Estimated Detection Limit calculation, or (if the interference is a discrete peak on both masses at the correct retention time for that target) by integrating the peak and reporting the EMPC as the calculated concentration. EMPC results of this nature are also user flagged in the same manner as the previous example.

10.7.4 Also problematic in analysis of PCB congeners is the situation where there are near-eluting (as opposed to co-eluting) peak or peaks which complicate the separation and quantification of a particular congener. This problem can be divided into two different categories:

10.7.4.1 There is a shoulder to the interference with a clear point of inflection so that the shoulder can be integrated separately and targeted as the given congener at that retention time. In this case, the coded/reintegrated shoulder may be reported as the congener at that retention time (as in Example 1 below), or it may still be necessary to render that congener as an EMPC due to another interference at higher level of chlorination (as in Example 2 below).

10.7.4.2 There are near-eluting peak or peaks (from the same level of chlorination as in Example 3 below, or from a higher level of chlorination as in Example 4 below) that by their relative size preclude the separation and quantification of a given congener. This concentration of this congener is relatively small compared to the interfering congener such that it would, if present be obscured by the front or tail of the interfering congener. In this case, the correct procedure is to estimate the peak height that the obscured congener would have to have attained in order to be successfully separated and detected, and then assign an EDL peak height of this estimated height divided by 2.5 for each channel. This will assign an EDL (to be converted in the report to an EMPC) at the estimated magnitude that this peak would have to attain before it could be separated from the interference.

10.7.5 Need for rework: When analyzing PCB congeners using the Supelco Octyl column, there are several potential interferences from co-eluting or near-eluting congeners that may cause unacceptable elevated detection limits (in the form of EMPCs as described above) for toxic PCB congeners with significant Toxic Equivalence Factors. If interferences of this type result in EMPC concentrations for PCB-126 and/or PCB-169 that exceed the Estimated Minimum Levels of the reference method, the sample will require re-work/reanalysis. A carbon column cleanup and/or analysis by DB5ms column is required to separate and confirm the amount of these congeners present.

Example 1: PCB-81

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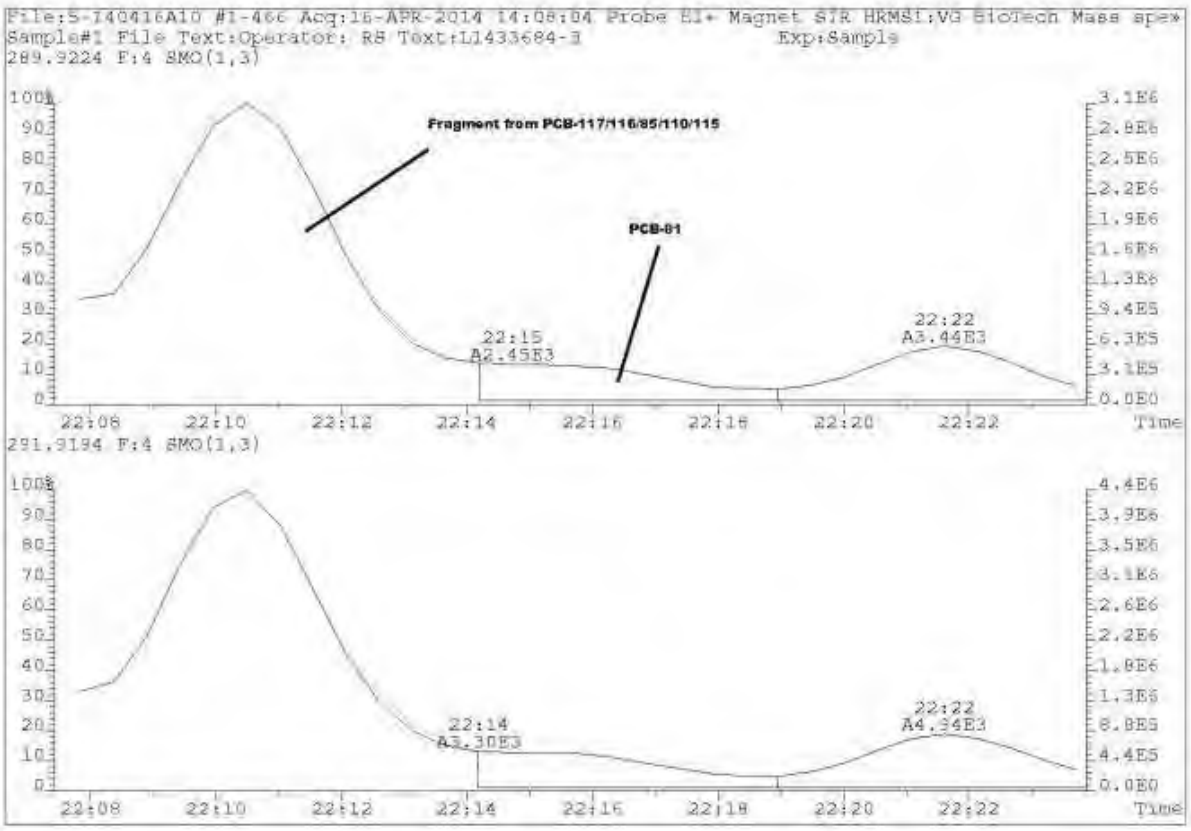

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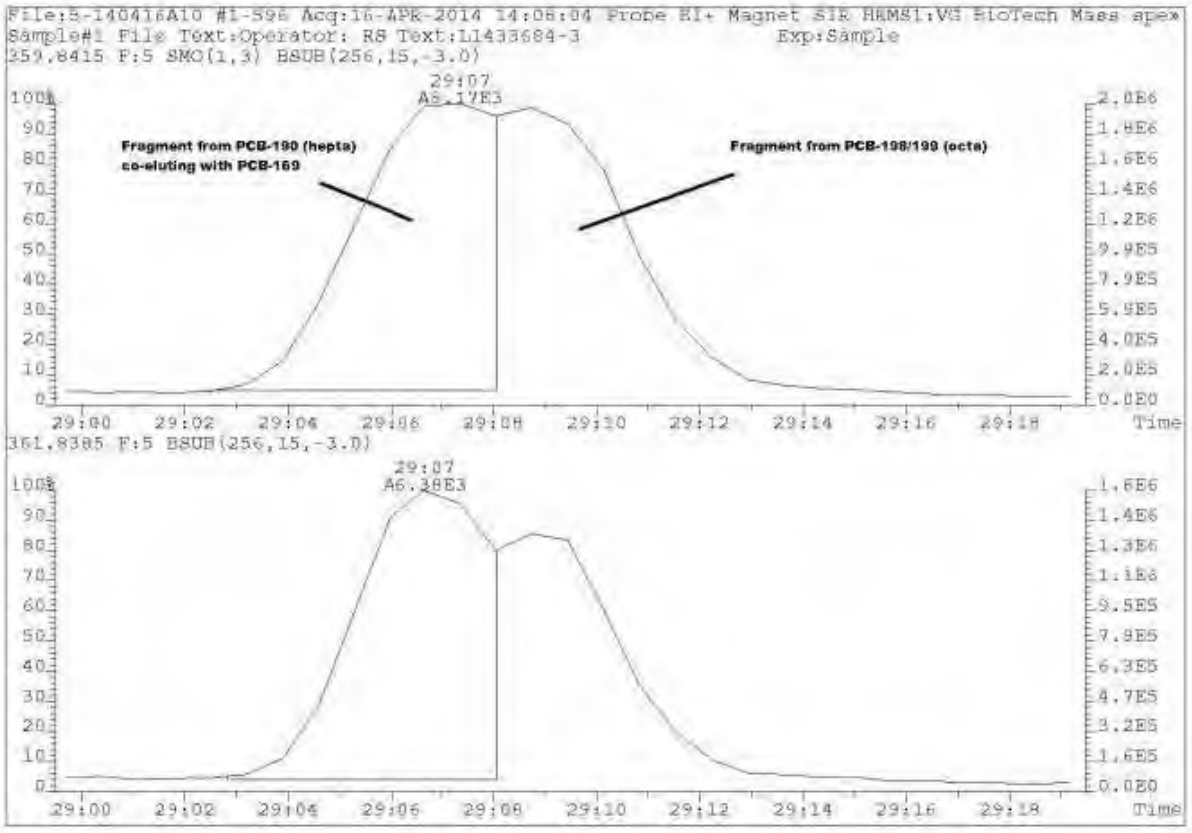
Example 2: PCB-169



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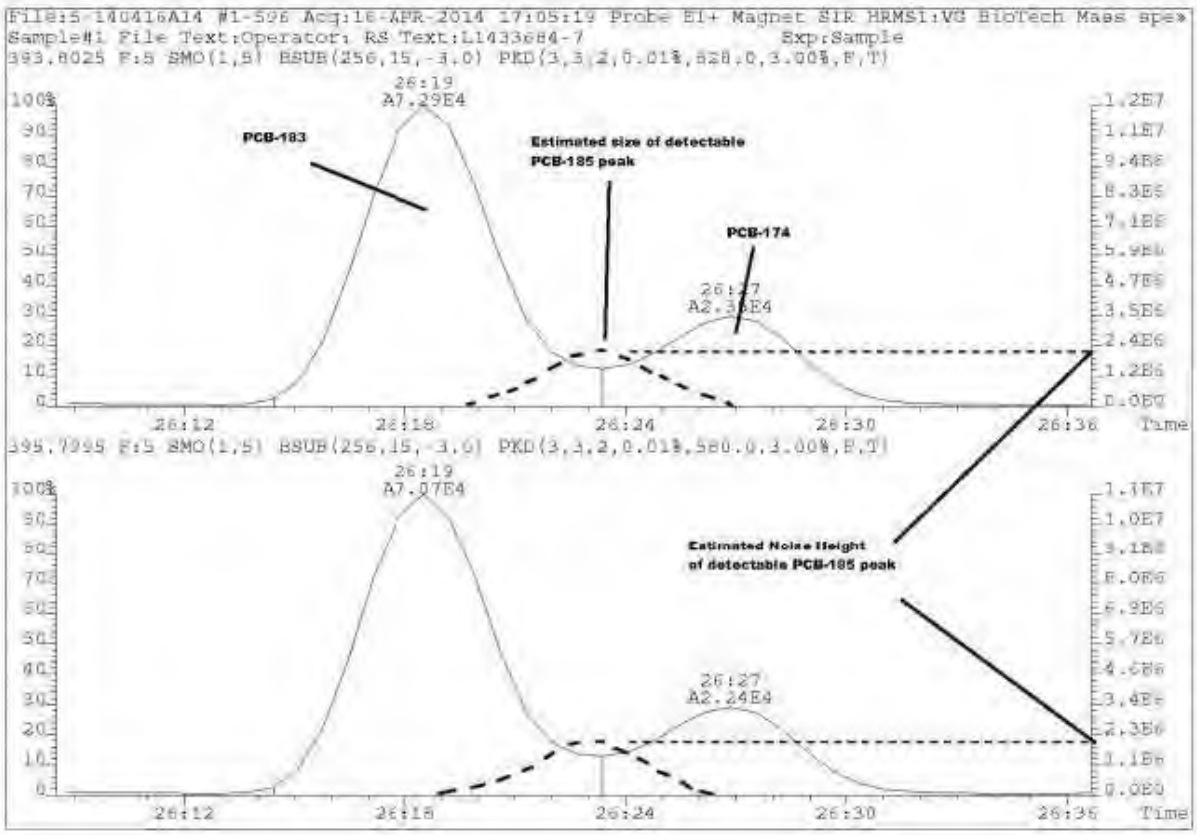
Example 3: PCB-185 obscured by larger congeners in the same level of Chlorination



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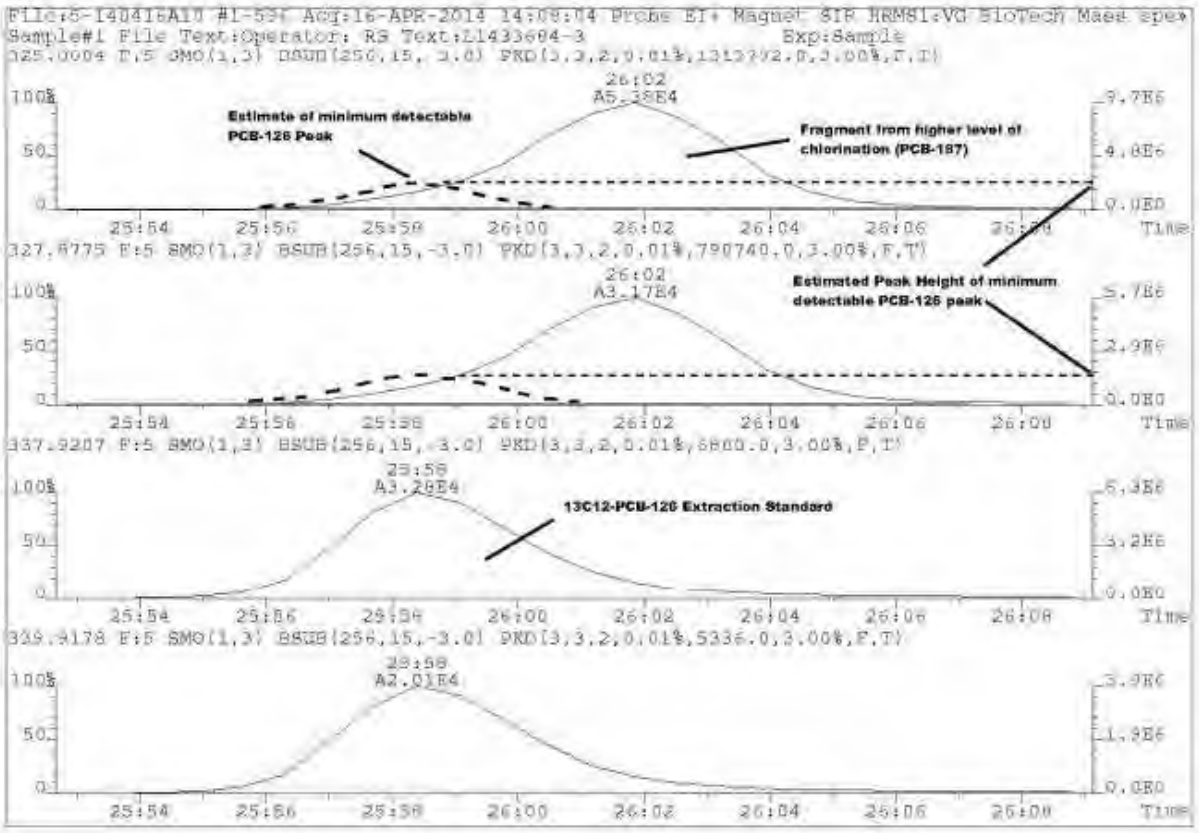
Example 4: PCB-126 obscured by interference at a higher level of chlorination



Interferences may pose a problem in the determination of congeners 81, 123, 126, and 169 in some environmental samples. Loss of one or more chlorines from a highly chlorinated congener may inflate or produce a false concentration for a less-chlorinated congener that elutes at the same retention time. If, upon inspection of the chromatogram, the possibility of interferences is evident (e.g., high concentrations of fragments from loss of one or two chlorines from higher chlorinated closely eluting congeners), carbon column fractionation (BU-WI-3006) and re-analysis is recommended.

11.0 REFERENCES

11.7 Primary Reference

1. EPA 1668, Rev. A, August 2003
2. EPA 1668, Rev. C, April 2010

11.8 Secondary Reference

1. Code of the Federal Registry, Volume 40, Part 60 App. A; Method 23.(May, 1995) (used for calculation of MDLs)

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2. Talyor 1987: Quality Assurance of Chemical Measurements, John KeenanTaylor, Lewis Publishers Inc., 1987. (LOQ definition)
3. EPA 1994: METHOD 218.6 DETERMINATION OF DISSOLVED HEXAVALENT CHROMIUM IN DRINKING WATER, GROUNDWATER, AND INDUSTRIAL WASTEWATER EFFLUENTS BY ION CHROMATOGRAPHY, U.S. Environmental Protection Agency (EPA), Revision 3.3 1994. (lab dup definition)
4. Eurachem Guide 1998 - IUPAC Compendium of Chemical Technology, 1985 (bias definition)
5. CDC Laboratory Procedure Manual, Method No. 28, October 2006 (serum sample handling conditions)
6. OSW SW846 Method 8290A

11.9 Instrument Manuals

1. Autospec Premier Operator's Guide 71500089202/Revision B

11.10 Software Manuals

1. TargetLynx/MassLynx v4.1 Getting Started Guide

11.11 Supporting ALS Documents and Forms

1. NA-SP-0100 Initial Method Validation – Chemistry
2. NA-SP-0102 Method Revalidation and MU – Chemistry
3. NA-SP-0104 Quality Control
4. BU-TM-1110 Overall HR Prep
5. BU-TM-1113 DTOX - Food
6. BU-TP-2113 DTOX Prep - Food
7. BU-SP-0021 Waste Handling and Sample Disoposal
8. BU-SP-0250 Control of Reagents and Reference Materials
9. BU-SP-0261 Sample Control, Login and Receipt
10. BU-SP-0263 Dilution
11. BU-WI-3006 Chromacarb
12. BU-WI-3007 GCMS Operation
13. BU-WI-3019 Manual Integration
14. BU-WI-3105a PCB by HRMS - Premier CFIA Methods
15. BU-WI-3105b PCB HT8 Saudi Arabia HRMS - Premier
16. BU-WI-3201 PCB Octyl HRMS-Autospec Premier
17. BU-WI-3225 Reporting HRMS Results
18. BU-FM-1105e PCB-1668-HRMSTL-1.0
19. BU-FM-1105f PCB-1668-CFIA-HRMSTL-2.0
20. BU-FM-1105h PCB-1668T-HRMSTL-2.0
21. BU-FM-1110j CFIA_DXPCB-HRMSTL-BU-2.0

12.0 DEVIATIONS AND CLARIFICATIONS FROM THE PRIMARY REFERENCE

The reference methods applicable to this document are:
US EPA Office of Water, Method 1668A
US EPA Office of Water, Method 1668C

These methods are referred-to herein as Method 1668

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The following changes and clarifications apply:

1. The absolute retention time criterion for decachlorobiphenyl of 55 minutes and the requirement that the RT and RRT of all congeners are within the limits of 1668 table 2 are not generally followed. These criteria pose an unnecessary restriction since method 1668 was developed without the use of electronic pressure control on the GC injection system, and the GC performance criteria and congener elution patterns can be met without these restrictions. A column system is used that differs from the suggested column but maintains all of the GC performance criteria and elution pattern of the suggested column, but in a shorter run. Alternate RT, RRT, RRT limits, and RT Windows for this column system are listed in Appendix A Table 1. It should be noted that the version of this table in EPA 1668C clarifies some ambiguity in the interpretation of the analogous information from EPA 1668A, and the following interpretations are used in the analysis of PCBs by both EPA 1668A and EPA 1668C:
    - It is recognized in 1668C that the RTs and RRTs may differ slightly from instrument to instrument and run to run.
    - The values presented in Appendix A Table one, like the values in 1668C Table 2, are given as examples and intended to illustrate the mathematical model that is to be used for daily RRT calibration so that RRT limits and RT windows can be established for that run to allow targeting of PCB congeners in sample injections.
    - Appendix A Table 1 includes the MLs and EMLs from Table 2 of both EPA 1668A and EPA 1668C to allow assessment of Method Blanks for analysis by both methods, and utilizes other elements from both methods to allow for clarity and consistency of approach between the two methods.
2. Also stated in method 1668C (13.1), we maintain that each and every individual clean-up procedure is, by definition, performance-based and optional. There is not an expectation within the industry to follow exactly the descriptions of clean-ups in reference methods. Adaptations which meet or exceed the required performance criteria are therefore acceptable within the scope of each reference method. The reference method descriptions are intended as guidelines or templates available to help the laboratory to define effective in-house clean-up methods. The objective within the laboratory is to provide quality clean extracts to the instrument for analysis. Each individual clean-up is part of the laboratory's 'arsenal' in order to achieve this objective.
3. There are differences within the individual reference methods as to the precise spiking protocols for adding extraction standards and native spikes (for LCS, MS and MSD). To ensure consistency within the laboratory between HRMS methods, the associated PCB preparative 'Specific Method' requires solid samples (including stack and ambient sorbants/filters) to be spiked in the soxhlet extractor from a nonane solution and waters are spiked before filtering from an acetone solution.
4. Sub-sampling of solids and pre-extraction processing is done in a manner that minimizes potential for cross-contamination. These processes are designed around SW846 protocols rather than 1668 protocols. Solids are sub-sampled directly from the bottle as submitted to the laboratory wherever practical. If the sample is submitted such that homogenization in the bottle is impractical (eg. the bottle is too full or lumps cannot be broken down), then transferring the sample to a tray or another bottle maybe in order.
5. The concentrations of labelled and native spiking solutions are not consistent with those listed in all of the reference methods. These concentrations are prepared at levels convenient and expedient for accurate laboratory processing. The amount (total pg) of each component added is consistent with the method.
6. Extraction and injection standard concentrations differ from 1668, in order to aid precise measurement and standardise volumes with other reference methods such as PCDD/F by 1613B.
7. A final volume of 45uL is routinely used, in order to limit the number of dilutions required. A final volume of 25uL is used where appropriate and on client request.

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8. Method 1668C recognizes the option to use the 209 congener mix as the daily calibration verification solution rather than the CS3. This document acknowledges and allows either calibration option for both 1668A and 1668C analytical approaches.
9. For method 1668C analysis via this document, the OPR labelled recovery limits are the same as for the sample recovery limits in method 1668C. This represents a broader acceptance range for the OPR than is currently listed in method 1668C. However, the control of the native (i.e. non-labelled) recovery limits is the key item to demonstrate/monitor in the OPR. Furthermore, in the OPR performance, it is important to demonstrate these native controls are maintained within the same range of labelled recoveries as is observed in the sample data. The OPR is called an LCS.
10. Solid, mixed phase, semi-solid, or oily samples are stored at 2-6˚C instead of <-10˚C.
11. Acetone is used as the pre-extraction solvent rather than toluene (1668C).

13.0 REVISION HISTORY

Version #	Date	Summary of Major Changes Made / By
01	20 Dec 2006	Method creation and definition. Stephen Kennedy/Ron McLeod
02	22 Jan 2007	1. Clarification on the application of the 10K resolution criterion. 2. Defining the 2.5:1 signal to noise reporting option 3. Change in title 4. Addition of glassware tracking. Requirement of segregated low level glassware 5. Addition of extract dilution protocol reference Ron McLeod
03	16 Oct 2009	1. Corrected grammatical errors and typos 2. Changes in extract storage conditions, and instrument RT and RRT criteria to reflect our current practices. 3. Addition of sections on calibration of results vs. daily calibration verification runs. Clarification of calibration scenarios as compared to EPA 1668A wording. Brad Reimer
04	NA	NA
05	29 Oct 2012	Added in Appendix C. Updated document names to current versions. Brad Reimer/Priscilla Leung
06	10 Jan 2013	Updated for Method 1668, revision C. Steve Kennedy/Ron McLeod. Corrected reference 15SVSP-DILUTION in 9.2.1 to current document version, BU-SP-0263 Dilution. Priscilla Leung
07	15 Feb 2013	Reorganized sections to fit National TM formatting. Added in computer hardware/software and troubleshooting in Section 6.3. Added in Table 7: LOD/LOQ Levels for DoD PJLA. PLeung
08	31-Jul-2014	Revisions following internal audit. / SEagle. SKennedy, RSaxon, BReimer
09	21-Nov-2014	Incorporating changes from confirmation of compliance, clarifying how to handle near eluting peaks (section 10.5.4), and updating calibration standard terminology to national format. / BReimer, SKennedy, SEagle
10	25-Feb-2015	Addition of Food DL and LOQ table / MKulig, RMcLeod
11	19-Mar-2015	Addition of procedural blanks to method QC. / MKulig
12	17-Sep-2015	Addition of Fat EDL table/RMcLeod
13	10-Nov-2015	Addition of DoD QSM 5.0 requirements to section 8.0, and Table 9a/9b. /MKulig
14	9-Nov-2016	LORs updated based on current revalidation, TargetLynx software added, updates to standards section, procedural blank prep instructions added, work instructions added, minor updates throughout in response to technical audit. /

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Version #	Date	Summary of Major Changes Made / By
		MKulig
15	2-Feb-2017	MB DQO in 8.1 updated to 2xEML as per reference method, MEOLs added, sample handling & storage tables updated, reporting templates added, dilution procedure added. / M.McPherson
16	7-Jan-2019	Updated some LORs in response to revalidation, added matrix 30 to LOR table. Expanded references in section 5. Updated section 7. Updated sections 6 & 10 to national format. Removed references for retired work instructions throughout and updated section 11. Minor updates and corrections throughout. / R. Saxon

14.0 WASTE MANAGEMENT

Refer to 'BU-SP-0021 Waste Handling and Sample Disposal'. Waste information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

15.0 SAFETY

This task may include CHEMICAL, BIOLOGICAL, OPERATIONAL and/or EQUIPMENT hazards. Staff must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

Refer to Material Safety Data Sheets (MSDS) for detailed information about safe handling guidelines and hazards associated with chemicals used in this method.

HAZARD ASSESSMENT		
Job Task #1:	**Hazards**	**Preventative Measures**
Sample Preparation	Exposure to solvents, exposure to contaminants in samples.	Wear gloves, safety glasses and lab coat. Work in fume hood. Avoid skin contact with solvents, samples, and sample extracts.
Job Task #2:	**Hazards**	**Preventative Measures**
Standard preparation	Exposure to solvents, exposure to toxic standard components. The toxicity or carcinogenicity of each chemical used in this Method has not been precisely determined; however, each compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. PCBs have been tentatively classified as known or suspected human or mammalian carcinogens.	Wear gloves, safety glasses and lab coat. Work in fume hood. Avoid skin contact with solvents, samples and sample extracts. On the basis of the available toxicological and physical properties of the CBs, all CBs, particularly pure standards, should be handled only by highly trained personnel thoroughly familiar with handling and cautionary procedures and the associated risks.
Job Task #3:	**Hazards**	**Preventative Measures**
Extended computer use	Eye strain and fatigue	Take regular short breaks to work on different tasks. Focus on objects at varying distances and/or rest your eyes for a few moments throughout the day.

Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

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16.0 APPENDICES

16.1 Appendix A – Tables

Table 1: Retention times (RT), RT references, relative retention times (RRTs), Method Detection Limits (MDLs), and Minimum Levels of Quantitation (MLs) for the 209 CB congeners

Table 2a/b: QC acceptance criteria for chlorinated biphenyls in VER, IPR, LCS, and samples

Table 3: Concentration of CB congeners in calibration and calibration verification standards

Table 4: Concentrations of native and labelled chlorinated biphenyls in stock solutions, spiking solutions, and final extracts

Table 5: Theoretical ion abundance ratios and QC limits

Table 6: Scan descriptors, levels of chlorination, m/z information, and substances monitored by HRGC/HRMS

Table 7: LOD/LOQ Levels (DoD PJLA)

Table 8: Congener, IUPAC numbers, CAS registry number, and labelled analogs

Table 9a: PCB LCS Limits for Solid/Tissue/Air Matrices (DoD Analysis Only)

Table 9b: PCB LCS Limits for Water Matrix (DoD Analysis Only)

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Table 1. Retention times (RT), RT references, relative retention times (RRTs), Method Detection Limits (MDLs), and minimum levels of quantitation (MLs) for the 209 CB congeners using EPA Method 1668

Note: This table was developed for the alternate column system (Section 9.4.2 subsection 5) which incorporates but improves upon the chromatographic performance of the SPB-Octyl column

Cl No [7]	Congener No. [2,3]	RT Ref [4]	RTs [5]	RRT [6]	RRT limits [7]	Window (sec)[8]	Quantitation reference [9]	Det. limits and minimum levels - EPA 1668C Matrix and concentration [10]				
								Water (pg/uL)		Other (ng/kg)		Extract (pg/uL)
								EMDL	EML	EMDL	EML	EML
	Monochlorobiphenyls											
1	1	1L	8:31	1.0020	0.9970-1.0070	-1 +3	1L	10	20	1	2	1
1	2	3L	10:03	0.9885	0.9836-0.9934	6	1L/3L	7	20	0.7	2	1
1	3	3L	10:11	1.0016	0.9966-1.0066	-1 +3	3L	11	50	1.1	5	2.5
	Dichlorobiphenyls											
2	4	4L	10:22	1.0016	0.9966-1.0066	-1 +3	4L	13	50	1.3	5	2.5
2	10	4L	10:30	1.0145	1.0094-1.0196	6	4L/15L	13	50	1.3	5	2.5
2	9	4L	11:49	1.1417	1.1360-1.1474	6	4L/15L	7	20	0.7	2	1
2	7	4L	11:57	1.1546	1.1488-1.1604	6	4L/15L	8	20	0.8	2	1
2	6	4L	12:07	1.1707	1.1648-1.1766	6	4L/15L	7	20	0.7	2	1
2	5	4L	12:20	1.1916	1.1856-1.1976	6	4L/15L	8	20	0.8	2	1
2	8	4L	12:26	1.2013	1.1953-1.2073	6	4L/15L	15	50	1.5	5	2.5
2	14	15L	13:36	0.9294	0.9248-0.9340	6	4L/15L	8	20	0.8	2	1
2	11	15L	14:12	0.9704	0.9655-0.9753	6	4L/15L	34	100	3.4	10	5
2 2 2	13 12 13/12	15L	14:25	0.9852	0.9803-0.9901	6	4L/15L	19	50	1.9	5	2.5
2	15	15L	14:38	1.0000	0.9950-1.0050	-1 +3	15L	16	50	1.6	5	2.5
	Trichlorobiphenyls											
3	19	19L	12:38	1.0013	0.9963-1.0063	-1 +3	19L	8	20	0.8	2	1
3 3 3	30 18 30/18	19L	13:58	1.1070	1.1015-1.1125	6	19L/37L	16	50	1.6	5	2.5
3	17	19L	14:16	1.1308	1.1251-1.1365	6	19L/37L	9	20	0.9	2	1
3	27	19L	14:24	1.1413	1.1356-1.1470	6	19L/37L	8	20	0.8	2	1
3	24	19L	14:30	1.1493	1.1436-1.1550	6	19L/37L	10	20	1	2	1
3	16	19L	14:36	1.1572	1.1514-1.1630	6	19L/37L	9	20	0.9	2	1
3	32	19L	14:58	1.1863	1.1804-1.1922	6	19L/37L	8	20	0.8	2	1
3	34	19L	15:49	1.2536	1.2473-1.2599	6	19L/37L	7	20	0.7	2	1
3	23	19L	15:57	1.2642	1.2579-1.2705	6	19L/37L	7	20	0.7	2	1
3 3 3	29 26 26/29	19L	16:10	1.2814	1.2750-1.2878	10	19L/37L	12	50	1.2	5	2.5
3	25	37L	16:20	0.8376	0.8334-0.8418	6	19L/37L	8	20	0.8	2	1
3	31	37L	16:32	0.8479	0.8437-0.8521	6	19L/37L	18	50	1.8	5	2.5
3 3 3	28 20 28/20	37L	16:45	0.8590	0.8547-0.8633	10	19L/37L	22	50	2.2	5	2.5
3 3 3	21 33 21/33	37L	16:54	0.8667	0.8624-0.8710	10	19L/37L	21	50	2.1	5	2.5
3	22	37L	17:12	0.8821	0.8777-0.8865	6	19L/37L	9	20	0.9	2	1
3	36	37L	18:15	0.9359	0.9312-0.9406	6	19L/37L	8	20	0.8	2	1
3	39	37L	18:30	0.9487	0.9440-0.9534	6	19L/37L	8	20	0.8	2	1
3	38	37L	18:54	0.9692	0.9644-0.9740	6	19L/37L	7	20	0.7	2	1
3	35	37L	19:14	0.9863	0.9814-0.9912	6	19L/37L	9	20	0.9	2	1
3	37	37L	19:31	1.0009	0.9959-1.0059	-1 +3	37L	10	20	1	2	1
	Tetrachlorobiphenyls											
4	54	54L	14:50	1.0000	0.9950-1.0050	-1 +3	54L	14	50	1.4	5	2.5
4 4 4	50 53 50/53	54L	16:21	1.1022	1.0967-1.1077	10	54L/81L/77L	25	100	2.5	10	5
4 4 4	45 51 45/51	54L	16:50	1.1348	1.1291-1.1405	10	54L/81L/77L	22	50	2.2	5	2.5
4	46	54L	17:01	1.1472	1.1415-1.1529	6	54L/81L/77L	10	20	1	2	1
4	52	54L	17:59	1.2124	1.2063-1.2185	6	54L/81L/77L	15	50	1.5	5	2.5
4	73	54L	18:05	1.2191	1.2130-1.2252	6	54L/81L/77L	14	50	1.4	5	2.5

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Cl No [7]	Congener No. [2,3]	RT Ref [4]	RTs [5]	RRT [6]	RRT limits [7]	Window (sec)[8]	Quantitation reference [9]	Det. limits and minimum levels - EPA 1668C Matrix and concentration [10]				
								Water (pg/uL)		Other (ng/kg)		Extract (pg/uL)
								EMDL	EML	EMDL	EML	EML
4	43	54L	18:10	1.2247	1.2186-1.2308	6	54L/81L/77L	14	50	1.4	5	2.5
4	69											
4	49											
4	69/49	54L	18:18	1.2337	1.2275-1.2399	10	54L/81L/77L	26	100	2.6	10	5
4	48	54L	18:31	1.2483	1.2421-1.2545	6	54L/81L/77L	14	50	1.4	5	2.5
4	65											
4	47											
4	44											
4	44/47/65	54L	18:42	1.2607	1.2544-1.2670	10	54L/81L/77L	40	100	4	10	5
4	62											
4	75											
4	59											
4	59/62/75	54L	18:54	1.2742	1.2678-1.2806	10	54L/81L/77L	37	100	3.7	10	5
4	42	54L	19:02	1.2831	1.2767-1.2895	6	54L/81L/77L	16	50	1.6	5	2.5
4	41											
4	71											
4	40											
4	41/40/71	54L	19:21	1.3045	1.2980-1.3110	10	54L/81L/77L	42	100	4.2	10	5
4	64	54L	19:31	1.3157	1.3091-1.3223	6	54L/81L/77L	13	50	1.3	5	2.5
4	72	81L	20:05	0.8351	0.8309-0.8393	6	54L/81L/77L	13	50	1.3	5	2.5
4	68	81L	20:17	0.8434	0.8392-0.8476	6	54L/81L/77L	14	50	1.4	5	2.5
4	57	81L	20:34	0.8552	0.8509-0.8595	6	54L/81L/77L	11	50	1.1	5	2.5
4	58	81L	20:43	0.8614	0.8571-0.8657	6	54L/81L/77L	14	50	1.4	5	2.5
4	67	81L	20:51	0.8669	0.8626-0.8712	6	54L/81L/77L	12	50	1.2	5	2.5
4	63	81L	21:01	0.8739	1.8689-0.8783	6	54L/81L/77L	12	50	1.2	5	2.5
4	61											
4	70											
4	76											
4	74											
4	61/70/74/76	81L	21:15	0.8836	1.8786-0.8880	12	54L/81L/77L	59	200	5.9	20	10
4	66	81L	21:28	0.8926	0.8881-0.8971	6	54L/81L/77L	17	50	1.7	5	2.5
4	55	81L	21:36	0.8981	0.8936-0.9026	6	54L/81L/77L	12	50	1.2	5	2.5
4	56	81L	21:55	0.9113	0.9067-0.9159	6	54L/81L/77L	15	50	1.5	5	2.5
4	60	81L	22:05	0.9182	0.9136-0.9228	6	54L/81L/77L	14	50	1.4	5	2.5
4	80	81L	22:20	0.9286	0.9240-0.9332	6	54L/81L/77L	11	50	1.1	5	2.5
4	79	81L	23:23	0.9723	0.9674-0.9772	6	54L/81L/77L	13	50	1.3	5	2.5
4	78	81L	23:45	0.9875	0.9826-0.9924	6	54L/81L/77L	16	50	1.6	5	2.5
4	81	81L	24:03	1.0000	0.9950-1.0050	-1 +3	81L	18	50	1.8	5	2.5
4	77	77L	24:26	1.0000	0.9950-1.0050	-1 +3	77L	14	50	1.4	5	2.5
	Pentachlorobiphenyls											
5	104	104L	18:39	1.0009	0.9959-1.0059	-1 +3	104L	14	50	1.4	5	2.5
5	96	104L	18:54	1.0143	1.0092-1.0194	10	104L/123L/114L/118L	15	50	1.5	5	2.5
5	103	104L	20:12	1.0841	1.0787-1.0895	6	104L/123L/114L/118L	11	50	1.1	5	2.5
5	94	104L	20:21	1.0921	1.0866-1.0976	6	104L/123L/114L/118L	13	50	1.3	5	2.5
5	95											
5	100											
5	93											
5	102											
5	98											
5	95/100/93/102/98	104L	20:52	1.1199	1.1143-1.1255	15	104L/123L/114L/118L	77	200	7.7	20	10
5	88											
5	91											
5	88/91	104L	21:13	1.1386	1.1329-1.1443	12	104L/123L/114L/118L	22	50	2.2	5	2.5
5	84	104L	21:24	1.1485	1.1428-1.1542	6	104L/123L/114L/118L	11	20	1.1	2	1
5	89	104L	21:43	1.1655	1.1597-1.1713	6	104L/123L/114L/118L	13	50	1.3	5	2.5
5	121	104L	21:59	1.1798	1.1739-1.1857	6	104L/123L/114L/118L	12	50	1.2	5	2.5
5	92	104L	22:14	1.1932	1.1872-1.1992	6	104L/123L/114L/118L	13	50	1.3	5	2.5
5	113											
5	90											
5	101											
5	113/90/101	104L	22:37	1.2138	1.2077-1.2199	10	104L/123L/114L/118L	47	200	4.7	20	10
5	83											
5	99											
5	83/99	104L	23:00	1.2343	1.2281-1.2405	12	104L/123L/114L/118L	29	100	2.9	10	5
5	112	104L	23:06	1.2397	1.2335-1.2459	6	104L/123L/114L/118L	14	50	1.4	5	2.5
5	119											
5	109 [11]											
5	86											
5	97											

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								Water (pg/uL)		Other (ng/kg)		Extract (pg/uL)
								EMDL	EML	EMDL	EML	EML
5	125											
5	87											
5	109/119/86/97/125/87	104L	23:23	1.2549	1.2486-1.2612	16	104L/123L/114L/118L	74	200	7.4	20	10
5	117											
5	116											
5	85											
5	110											
5	115											
5	117/116/85/110/115	104L	23:55	1.2835	1.2771-1.2899	12	104L/123L/114L/118L	38	100	3.8	10	5
5	82	104L	24:11	1.2979	1.2914-1.3044	6	104L/123L/114L/118L	15	50	1.5	5	2.5
5	111	104L	24:25	1.3104	1.3038-1.3170	6	104L/123L/114L/118L	14	50	1.4	5	2.5
5	120	104L	24:44	1.3274	1.3208-1.3340	6	104L/123L/114L/118L	13	50	1.3	5	2.5
5	108 [12]											
5	124											
5	108/124	104L	25:31	1.3694	1.3626-1.3762	10	104L/123L/114L/118L	29	100	2.9	10	5
5	107 [13]	104L	25:40	1.3775	1.3706-1.3844	6	104L/123L/114L/118L	17	50	1.7	5	2.5
5	123	123L	25:44	1.0000	0.9950-1.0050	-1 +3	123L	17	50	1.7	5	2.5
5	106	123L	25:50	1.0039	0.9989-1.0089	6	104L/123L/114L/118L	17	50	1.7	5	2.5
5	118	118L	25:59	1.0006	0.9956-1.0056	-1 +3	118L	30	100	3	10	5
5	122	118L	26:12	1.0090	1.0040-1.0140	6	104L/123L/114L/118L	12	50	1.2	5	2.5
5	114	114L	26:21	1.0006	0.9956-1.0056	-1 +3	114L	15	50	1.5	5	2.5
5	105	105L	26:47	1.0006	0.9956-1.0056	-1 +3	105L	17	50	1.7	5	2.5
5	127	105L	27:45	1.0367	1.0315-1.0419	6	104L/123L/114L/118L	14	50	1.4	5	2.5
5	126	126L	28:50	1.0006	0.9956-1.0056	-1 +3	126L	16	50	1.6	5	2.5
	Hexachlorobiphenyls											
6	155	155L	22:30	1.0015	0.9965-1.0065	-1 +3	155L	14	50	1.4	5	2.5
6	152	155L	22:38	1.0074	1.0024-1.0124	6	155L/156L/157L/167L	14	50	1.4	5	2.5
6	150	155L	22:44	1.0119	1.0068-1.0170	6	155L/156L/157L/167L	15	50	1.5	5	2.5
6	136	155L	22:59	1.0230	1.0179-1.0281	6	155L/156L/157L/167L	16	50	1.6	5	2.5
6	145	155L	23:11	1.0319	1.0267-1.0371	6	155L/156L/157L/167L	16	50	1.6	5	2.5
6	148	155L	24:11	1.0764	1.0710-1.0818	6	155L/156L/157L/167L	14	50	1.4	5	2.5
6	151											
6	135											
6	151/135	155L	24:35	1.0942	1.0887-1.0997	10	155L/156L/157L/167L	46	100	4.6	10	5
6	154	135L	24:45	1.1016	1.0961-1.1071	6	155L/156L/157L/167L	46	100	4.6	10	5
6	144	155L	24:58	1.1113	1.1057-1.1169	6	155L/156L/157L/167L	15	50	1.5	5	2.5
6	147											
6	149											
6	147/149	155L	25:13	1.1224	1.1168-1.1280	10	155L/156L/157L/167L	35	100	3.5	10	5
6	134											
6	143											
6	134/143	155L	25:23	1.1298	1.1242-1.1354	10	155L/156L/157L/167L	33	100	3.3	10	5
6	139											
6	140											
6	139/140	155L	25:37	1.1402	1.1345-1.1459	10	155L/156L/157L/167L	29	100	2.9	10	5
6	131	155L	25:45	1.1461	1.1404-1.1518	6	155L/156L/157L/167L	17	50	1.7	5	2.5
6	142	155L	25:51	1.1506	1.1448-1.1564	6	155L/156L/157L/167L	17	50	1.7	5	2.5
6	132	155L	26:03	1.1595	1.1537-1.1653	10	155L/156L/157L/167L	16	50	1.6	5	2.5
6	133	155L	26:22	1.1736	1.1677-1.1795	6	155L/156L/157L/167L	12	50	1.2	5	2.5
6	165	167L	26:37	0.8867	0.8823-0.8911	6	155L/156L/157L/167L	13	50	1.3	5	2.5
6	146	167L	26:48	0.8928	0.8883-0.8973	6	155L/156L/157L/167L	14	50	1.4	5	2.5
6	161	167L	26:54	0.8962	0.8917-0.9007	6	155L/156L/157L/167L	13	50	1.3	5	2.5
6	153											
6	168											
6	153/168	167L	27:13	0.9067	0.9022-0.9112	10	155L/156L/157L/167L	30	100	3	10	5
6	141	167L	27:21	0.9112	0.9066-0.9158	6	155L/156L/157L/167L	17	50	1.7	5	2.5
6	130	167L	27:37	0.9200	0.9154-0.9246	6	155L/156L/157L/167L	13	50	1.3	5	2.5
6	137											
6	164											
6	137/164	167L	27:49	0.9267	0.9221-0.9313	6	155L/156L/157L/167L	15	50	1.5	5	2.5
6	138											
6	163											
6	129											
6	138/163/129	167L	28:04	0.9350	0.9303-0.9397	10	155L/156L/157L/167L	63	200	6.3	20	10
6	160	167L	28:10	0.9384	0.9337-0.9431	10	155L/156L/157L/167L	63	200	6.3	20	10
6	158	167L	28:19	0.9434	0.9387-0.9481	6	155L/156L/157L/167L	16	50	1.6	5	2.5
6	166											
6	128											
6	128/166	167L	28:54	0.9628	0.9580-0.9676	10	155L/156L/157L/167L	29	100	2.9	10	5

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								Water (pg/uL)		Other (ng/kg)		Extract (pg/uL)
								EMDL	EML	EMDL	EML	EML
6	159	167L	29:32	0.9839	0.9790-0.9888	6	155L/156L/157L/167L	14	50	1.4	5	2.5
6	162	167L	29:44	0.9906	0.9856-0.9956	6	155L/156L/157L/167L	13	50	1.3	5	2.5
6	167	167L	30:02	1.0006	0.9956-1.0056	-1 +3	155L/156L/157L/167L	13	50	1.3	5	2.5
6	156											
6	157											
6	156/157	156L	30:49	1.0005	0.9955-1.0055	6	156L/157L	23	100	2.3	10	5
6	169	169L	32:58	1.0005	0.9955-1.0055	-1 +3	169L	15	50	1.5	5	2.5
	Heptachlorobiphenyls											
7	188	188L	26:19	1.0006	0.9956-1.0056	-1 +3	188L	15	50	1.5	5	2.5
7	179	188L	26:32	1.0089	1.0039-1.0139	6	188L/189L	14	50	1.4	5	2.5
7	184	188L	26:54	1.0228	1.0177-1.0279	6	188L/189L	14	50	1.4	5	2.5
7	176	188L	27:08	1.0317	1.0265-1.0369	6	188L/189L	12	50	1.2	5	2.5
7	186	188L	27:26	1.0431	1.0379-1.0483	6	188L/189L	15	50	1.5	5	2.5
7	178	188L	28:19	1.0767	1.0713-1.0821	6	188L/189L	14	50	1.4	5	2.5
7	175	188L	28:45	1.0932	1.0877-1.0987	6	188L/189L	14	50	1.4	5	2.5
7	187	188L	28:56	1.1001	1.0946-1.1056	6	188L/189L	17	50	1.7	5	2.5
7	182	188L	29:04	1.1052	1.0997-1.1107	6	188L/189L	13	50	1.3	5	2.5
7	183	188L	29:20	1.1153	1.1097-1.1209	6	188L/189L	28	100	2.8	10	5
7	185	188L	29:25	1.1185	1.1129-1.1241	6	188L/189L	28	100	2.8	10	5
7	174	188L	29:30	1.1217	1.1161-1.1273	6	188L/189L	15	50	1.5	5	2.5
7	177	188L	29:48	1.1331	1.1274-1.1388	6	188L/189L	11	50	1.1	5	2.5
7	181	188L	30:04	1.1432	1.1375-1.1489	6	188L/189L	13	50	1.3	5	2.5
7	171											
7	173											
7	171/173	188L	30:12	1.1483	1.1426-1.1540	6	188L/189L	30	100	3	10	5
7	172	189L	31:17	0.9041	0.8996-0.9086	6	188L/189L	13	50	1.3	5	2.5
7	192	189L	31:28	0.9094	0.9049-0.9139	6	188L/189L	13	50	1.3	5	2.5
7	193											
7	180											
7	180/193	189L	31:41	0.9157	0.9111-0.9203	6	188L/189L	30	100	3	10	5
7	191	189L	31:56	0.9229	0.9183-0.9275	6	188L/189L	13	50	1.3	5	2.5
7	170	189L	32:33	0.9408	0.9361-0.9455	6	188L/189L	12	50	1.2	5	2.5
7	190	189L	32:55	0.9513	0.9465-0.9561	6	188L/189L	14	50	1.4	5	2.5
7	189	189L	34:37	1.0005	0.9955-1.0055	-1 +3	189L	13	50	1.3	5	2.5
	Octachlorobiphenyls											
8	202	202L	29:52	1.0000	0.9950-1.0050	-1 +3	202L	24	100	2.4	10	5
8	201	202L	30:29	1.0206	1.0155-1.0257	10	202L/205L	20	50	2	5	2.5
8	204	202L	30:56	1.0357	1.0305-1.0409	6	202L/205L	21	50	2.1	5	2.5
8	197	202L	31:04	1.0402	1.0350-1.0454	6	202L/205L	43	100	4.3	10	5
8	200	202L	31:10	1.0435	1.0383-1.0487	6	202L/205L	43	100	4.3	10	5
8	198											
8	199											
8	198/199	202L	33:00	1.1049	1.0994-1.1104	10	202L/205L	37	100	3.7	10	5
8	196	205L	33:26	0.9215	0.9169-0.9261	6	202L/205L	20	50	2	5	2.5
8	203	205L	33:34	0.9251	0.9205-0.9297	6	202L/205L	18	50	1.8	5	2.5
8	195	205L	34:28	0.9499	0.9452-0.9546	6	202L/205L	22	50	2.2	5	2.5
8	194	205L	36:00	0.9922	0.9872-0.9972	6	202L/205L	18	50	1.8	5	2.5
8	205	205L	36:18	1.0005	0.9955-1.0055	-1 +3	205L	15	50	1.5	5	2.5
	NonaCBs											
9	208	208L	34:18	1.0005	0.9955-1.0055	-1 +3	208L	16	50	1.6	5	2.5
9	207	208L	34:55	1.0185	1.0134-1.0236	6	208L/206L	19	50	1.9	5	2.5
9	206	206L	37:29	1.0933	1.0878-1.0988	-1 +3	206L	16	50	1.6	5	2.5
	DecacCB											
10	209	209L	38:43	1.0004	0.9954-1.0054	-1 +3	209L	16	50	1.6	5	2.5
Labelled Extraction Standards												
1	1L	9L	8:30	0.7203	0.7167-0.7239	30	9L					
1	3L	9L	10:10	0.8616	0.8573-0.8659	30	9L					
2	4L	9L	10:21	0.8771	0.8727-0.8815	30	9L					
2	15L	9L	14:38	1.2401	1.2339-1.2463	20	9L					
3	19L	9L	12:37	1.0692	1.0639-1.0745	30	9L					
3	37L	9L	19:30	1.0853	1.0799-1.0907	30	52L					
4	54L	52L	14:50	0.8256	0.8215-0.8297	20	52L					
4	81L	52L	24:03	1.0634	1.0581-1.0687	20	52L					
4	77L	52L	24:26	1.0803	1.0749-1.0857	20	52L					
5	104L	101L	18:38	0.8239	0.8198-0.8280	20	101L					
5	123L	101L	25:44	1.1378	1.1321-1.1435	20	101L					

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Cl No [7]	Congener No. [2,3]	RT Ref [4]	RTs [5]	RRT [6]	RRT limits [7]	Window (sec)[8]	Quantitation reference [9]	Det. limits and minimum levels - EPA 1668C Matrix and concentration [10]				
								Water (pg/uL)		Other (ng/kg)		Extract (pg/uL)
								EMDL	EML	EMDL	EML	EML
5	118L	101L	25:58	1.1481	1.1424-1.1538	20	101L					
5	114L	101L	26:20	1.1643	1.1585-1.1701	20	101L					
5	105L	101L	26:46	1.1835	1.1776-1.1894	20	101L					
5	126L	101L	28:49	1.2741	1.2677-1.2805	20	101L					
6	155L	138L	22:28	0.9934	0.9884-0.9984	20	138L					
6	167L	138L	30:01	1.0707	1.0653-1.0761	20	138L					
6	156L	138L	30:48	1.0987	1.0932-1.1042	20	138L					
6	157L	138L	30:48	1.0987	1.0932-1.1042	20	138L					
6	156L/157L	138L	30:48	1.0987	1.0932-1.1042	20	138L					
6	169L	138L	32:57	1.1754	1.1695-1.1813	20	138L					
7	188L	194L	26:18	0.9382	0.9335-0.9429	20	194L					
7	189L	194L	34:36	1.2342	1.2280-1.2404	20	194L					
8	202L	194L	29:52	1.0654	1.0601-1.0707	20	194L					
8	205L	194L	36:17	1.0083	1.0033-1.0133	30	194L					
9	208L	194L	34:17	0.9528	0.9480-0.9576	20	194L					
9	206L	194L	37:28	1.0412	1.0360-1.0464	30	194L					
10	209L	194L	38:42	1.0755	1.0701-1.0809	30	194L					
Labelled clean-up standards												
3	28L	52L	16:44	0.9314	0.9267-0.9361	20	52L					
5	111L	101L	24:24	1.0789	1.0735-1.0843	20	101L					
7	178L	194L	28:19	1.0101	1.0050-1.0152	20	138L					
Labelled injection internal standards												
2	9L	138L	11:48	0.4209	0.4188-0.4230	25	138L					
4	52L	138L	17:58	0.6409	0.6377-0.6441	25	138L					
5	101L	138L	22:37	0.8068	0.8028-0.8108	25	138L					
6	138L		28:02			100						
8	194L	138L	35:59	1.2836	1.2772-1.2900	25	138L					

1. Number of chlorines on congener.
2. Suffix "L" indicates labelled compound.
3. Multiple congeners in a box indicates a group of congeners that co-elute or may not be adequately resolved on a 30-m SPBOctyl column. Congeners included in the group are listed as the last entry in the box.
4. Retention time reference that is used to locate target congener.
5. Retention time of target congener.
6. RRT between the RT for the congener and RT for the reference.
7. RRT limits based on RT window. RTs, RRTs, and RRT limits may differ slightly from those in this table. Nominal limits during analysis based on an ± 0.5% of the RRT from daily calibration data, and in a sample where matrix retention time shifting may occur, may also be adjusted for the nearest eluted isomer, RT of labeled targets within that window, and environmental pattern recognition by the experienced spectrometrist..
8. RT window width for congener or group of two or more congeners.
9. Labelled congeners that form the quantitation reference. Areas from the exact m/z's of the congeners listed in the quantitation reference are summed, and divided by the number of congeners in the quantitation reference. For example, for congener 10, the areas at the exact m/z's for 4L and 15L are summed and the sum is divided by 2 (because there are 2 congeners in the quantitation reference).
10. EMDLs and EMLs with common laboratory interferences present. Without interferences, EMDLs and EMLs will be, respectively, 5 and 10 pg/L for aqueous samples, 0.5 and 1.0 ng/kg for soil, tissue, and mixed-phase samples, and EMLs for extracts will be 0.5 pg/uL. These are the limits published in EPA 1668C. For EPA 1668A limits, refer to EPA 1668A Table 2.
11. EPA 1668A lists this congener as PCB-108
12. EPA 1668A lists this congener as PCB-107
13. EPA 1668A lists this congener as PCB-109

Table 2A. QC acceptance criteria for chlorinated biphenyls in VER, IPR, LCS, and samples for Method 1668A [1]

Congener	IUPAC Number [2]	Test Conc (ng/mL)	VER (%)	IPR		LCS (%)	Labelled compound recovery in samples (%)
				RSD (%)	X (%)		
2-MoCB	1	50	70-130	40	60-140	50-150	
4-MoCB	3	50	70-130	40	60-140	50-150	
2,2'-DiCB	4	50	70-130	40	60-140	50-150	
4,4'-DiCB	15	50	70-130	40	60-140	50-150	
2,2'6-TrCB	19	50	70-130	40	60-140	50-150	
3,4,4'-TrCB	37	50	70-130	40	60-140	50-150	
2,2'6,6'TeCB	54	50	70-130	40	60-140	50-150	
3,3',4,4'-TeCB	77	50	70-130	40	60-140	50-150	
3,4,4',5-TeCB	81	50	70-130	40	60-140	50-150	
2,2',4,6,6'-PeCB	104	50	70-130	40	60-140	50-150	
2,3,3',4,4'-PeCB	105	50	70-130	40	60-140	50-150	
2,3,4,4',5-PeCB	114	50	70-130	40	60-140	50-150	
2,3',4,4',5-PeCB	118	50	70-130	40	60-140	50-150	
2',3,4,4',5-PeCB	123	50	70-130	40	60-140	50-150	
3,3',4,4',5-PeCB	126	50	70-130	40	60-140	50-150	

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2,2',4,4',6,6'-HxCB	155	50	70-130	40	60-140	50-150	
2,3,3',4,4',5-HxCB5	156	50	70-130	40	60-140	50-150	
2,3,3',4,4',5'-HxCB5	157	50	70-130	40	60-140	50-150	
2,3',4,4',5,5'-HxCB	167	50	70-130	40	60-140	50-150	
3,3',4,4',5,5'-HxCB	169	50	70-130	40	60-140	50-150	
2,2',3,4',5,6,6'-HpCB	188	50	70-130	40	60-140	50-150	
2,3,3',4,4',5,5'-HpCB	189	50	70-130	40	60-140	50-150	
2,2',3,3',5,5',6,6'-OcCB	202	50	70-130	40	60-140	50-150	
2,3,3',4,4',5,5',6-OcCB	205	50	70-130	40	60-140	50-150	
2,2',3,3',4,4',5,5',6-NoCB	206	50	70-130	40	60-140	50-150	
2,2',3,3,'4,5,5',6,6'-NoCB	208	50	70-130	40	60-140	50-150	
DeCB	209	50	70-130	40	60-140	50-150	
Extraction standard							
13C12-2-MoCB	1L	100	50-150	50	35-135	30-140	25-150
13C12-4-MoCB	3L	100	50-150	50	35-135	30-140	25-150
13C12-2,2'-DiCB	4L	100	50-150	50	35-135	30-140	25-150
13C12-4,4'-DiCB	15L	100	50-150	50	35-135	30-140	25-150
13C12-2,2',6-TrCB	19L	100	50-150	50	35-135	30-140	25-150
13C12-3,4,4'-TrCB	37L	100	50-150	50	35-135	30-140	25-150
13C12-2,2',6,6'-TeCB	54L	100	50-150	50	35-135	30-140	25-150
13C12-3,3',4,4'-TCB	77L	100	50-150	50	35-135	30-140	25-150
13C12-3,4,4',5-TeCB	81L	100	50-150	50	35-135	30-140	25-150
13C12-2,2',4,6,6'-PeCB	104L	100	50-150	50	35-135	30-140	25-150
13C12-2,3,3',4,4'-PeCB	105L	100	50-150	50	35-135	30-140	25-150
13C12-2,3,4,4',5-PeCB	114L	100	50-150	50	35-135	30-140	25-150
13C12-2,3',4,4',5-PeCB	118L	100	50-150	50	35-135	30-140	25-150
13C12-2',3,4,4',5-PeCB	123L	100	50-150	50	35-135	30-140	25-150
13C12-3,3',4,4',5-PeCB	126L	100	50-150	50	35-135	30-140	25-150
13C12-2,2',4,4',6,6'-HxCB	155L	100	50-150	50	35-135	30-140	25-150
13C12-2,3,3',4,4',5 -HxCB (3)	156L	100	50-150	50	35-135	30-140	25-150
13C12-2,3,3',4,4',5'-HxCB (3)	157L	100	50-150	50	35-135	30-140	25-150
13C12-2,3',4,4',5,5'-HxCB	167L	100	50-150	50	35-135	30-140	25-150
13C12-3,3',4,4',5,5'-HxCB	169L	100	50-150	50	35-135	30-140	25-150
13C12-2,2',3,4',5,6,6'-HpCB	188L	100	50-150	50	35-135	30-140	25-150
13C12-2',3,3',4,4',5,5'-HpCB	189L	100	50-150	50	35-135	30-140	25-150
13C12-2,2',3,3',5,5',6,6'-OcCB	202L	100	50-150	50	35-135	30-140	25-150
13C12-2,3,3',4,4',5,5',6-OcCB	205L	100	50-150	50	35-135	30-140	25-150
13C12-2,2',3,3',4,4',5,5',6-NoCB	206L	100	50-150	50	35-135	30-140	25-150
13C12-2,2',3,3',4,5,5',6,6'-NoCB	208L	100	50-150	50	35-135	30-140	25-150
13C12-2,2',3,3',4,4',5,5',6,6'-DeCB	209L	100	50-150	50	35-135	30-140	25-150
Cleanup standard							
13C12-2,4,4'-TrCB	28L	100	60-130	45	45-120	40-125	30-135
13C12-2,3,3',5,5'-PeCB	111L	100	60-130	45	45-120	40-125	30-135
13C12-2,2',3,3',5,5',6-HpCB	178L	100	60-130	45	45-120	40-125	30-135

1. QC acceptance criteria for IPR, LCS, and samples based on a 20 ul extract final volume
2. Suffix "L" indicates labelled compound.
3. PCBs 156 and 157 are tested as the sum of two concentrations

Table 2B. QC acceptance crtiteria for chlorinated biphenyls in VER, IPR, LCS, and samples for Method 1668C [1,2]

Congener Name	Congener No. [3]	Test Conc (ng/mL)[4]	VER (%)[5]	IPR		LCS Recovery (%)	Labeled Compound Recovery in samples (%)
				RSD (%)	X (%)		
2-MoCB	1	50	75 - 125	25	70 - 130	60 - 135	
4-MoCB	3	50	75 - 125	25	70 - 130	60 - 135	
2,2'-DiCB	4	50	75 - 125	25	70 - 130	60 - 135	
4,4'-DiCB	15	50	75 - 125	25	70 - 130	60 - 135	
2,2'6-TrCB	19	50	75 - 125	25	70 - 130	60 - 135	
3,4,4'-TrCB	37	50	75 - 125	25	70 - 130	60 - 135	
2,2'6,6'TeCB	54	50	75 - 125	25	70 - 130	60 - 135	
3,3',4,4'-TeCB	77	50	75 - 125	25	70 - 130	60 - 135	
3,4,4',5-TeCB	81	50	75 - 125	25	70 - 130	60 - 135	
2,2',4,6,6'-PeCB	104	50	75 - 125	25	70 - 130	60 - 135	
2,3,3',4,4'-PeCB	105	50	75 - 125	25	70 - 130	60 - 135	
2,3,4,4',5-PeCB	114	50	75 - 125	25	70 - 130	60 - 135	NA
2,3',4,4',5-PeCB	118	50	75 - 125	25	70 - 130	60 - 135	
2',3,4,4',5-PeCB	123	50	75 - 125	25	70 - 130	60 - 135	
3,3',4,4',5-PeCB	126	50	75 - 125	25	70 - 130	60 - 135	
2,2',4,4',6,6'-HxCB	155	50	75 - 125	25	70 - 130	60 - 135	
2,3,3',4,4',5-HxCB 6	156	50	75 - 125	25	70 - 130	60 - 135	
2,3,3',4,4',5'-HxCB 6	157	50	75 - 125	25	70 - 130	60 - 135	
2,3',4,4',5,5'-HxCB	167	50	75 - 125	25	70 - 130	60 - 135	
3,3',4,4',5,5'-HxCB	169	50	75 - 125	25	70 - 130	60 - 135	

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2,2',3,4',5,6,6'-HpCB	188	50	75 - 125	25	70 - 130	60 - 135	
2,3,3',4,4',5,5'-HpCB	189	50	75 - 125	25	70 - 130	60 - 135	
2,2',3,3',5,5',6,6'-OcCB	202	50	75 - 125	25	70 - 130	60 - 135	
2,3,3',4,4',5,5',6-OcCB	205	50	75 - 125	25	70 - 130	60 - 135	
2,2',3,3',4,4',5,5',6-NoCB	206	50	75 - 125	25	70 - 130	60 - 135	
2,2',3,3,'4,5,5',6,6'-NoCB	208	50	75 - 125	25	70 - 130	60 - 135	
DeCB	209	50	75 - 125	25	70 - 130	60 - 135	
Extraction standards							
13C12-2-MoCB	1L	100	50 - 145	70	20 - 135	5 - 145	5 - 145
13C12-4-MoCB	3L	100	50 - 145	70	20 - 135	5 - 145	5 - 145
13C12-2,2'-DiCB	4L	100	50 - 145	70	20 - 135	5 - 145	5 - 145
13C12-4,4'-DiCB	15L	100	50 - 145	70	20 - 135	5 - 145	5 - 145
13C12-2,2',6-TrCB	19L	100	50 - 145	70	20 - 135	5 - 145	5 - 145
13C12-3,4,4'-TrCB	37L	100	50 - 145	70	20 - 135	5 - 145	5 - 145
13C12-2,2',6,6'-TeCB	54L	100	50 - 145	70	20 - 135	5 - 145	5 - 145
13C12-3,3',4,4'-TeCB	77L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-3,4,4',5-TeCB	81L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2,2',4,6,6'-PeCB	104L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2,3,3',4,4'-PeCB	105L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2,3,4,4',5-PeCB	114L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2,3',4,4',5-PeCB	118L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2',3,4,4',5-PeCB	123L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-3,3',4,4',5-PeCB	126L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2,2',4,4',6,6'-HxCB	155L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2,3,3',4,4',5 -HxCB [6]	156L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2,3,3',4,4',5'-HxCB [6]	157L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2,3',4,4',5,5'-HxCB	167L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-3,3',4,4',5,5'-HxCB	169L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2,2',3,4',5,6,6'-HpCB	188L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2',3,3',4,4',5,5'-HpCB	189L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2,2',3,3',5,5',6,6'-OcCB	202L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2,3,3',4,4',5,5',6-OcCB	205L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2,2',3,3',4,4',5,5',6-NoCB	206L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2,2',3,3',4,5,5',6,6'-NoCB	208L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
13C12-2,2',3,3',4,4',5,5',6,6'-DeCB	209L	100	50 - 145	50	45 - 135	10 - 145	10 - 145
Cleanup standards							
13C12-2,4,4'-TrCB	28L	100	65 - 135	70	20 - 135	5 - 145	5 - 145
13C12-2,3,3',5,5'-PeCB	111L	100	75 - 125	50	45 - 135	10 - 145	10 - 145
13C12-2,2',3,3',5,5',6-HpCB	178L	100	75 - 125	50	45 - 135	10 - 145	10 - 145

1. Reference 22 describes how interlaboratory results were pooled from analyses of wastewater, biosolids, and fish tissue samples.
2. QC acceptance criteria for IPR, LCS, and samples based on a 20-µL extract final volume
3. Suffix "L" indicates labeled compound.
4. See Table 3.
NA = Not applicable
5. Section 8.2.1.3.
6. CBs 156/157 and 156L/157L are tested as the sum of the two congeners

Table 3. Concentration of CB congeners in calibration and calibration verification standards
Solution concentration (ng/mL)

CB congener	IUPAC1	CS-0.2 (Hi sens)2	CS-1	CS-2	CS-3 (VER)	CS-4	CS-5
Native Toxics/LOC							
2-MoCB	1	0.2	1.0	5.0	50	400	2000
4-MoCB	3	0.2	1.0	5.0	50	400	2000
2,2'-DiCB	4	0.2	1.0	5.0	50	400	2000
4,4'-DiCB	15	0.2	1.0	5.0	50	400	2000
2,2',6'-TrCB	19	0.2	1.0	5.0	50	400	2000
3,4,4'-TrCB	37	0.2	1.0	5.0	50	400	2000
2,2',6,6'-TeCB	54	0.2	1.0	5.0	50	400	2000
3,3',4,4'-TeCB	77	0.2	1.0	5.0	50	400	2000
3,4,4',5-TeCB	81	0.2	1.0	5.0	50	400	2000
2,2',4,6,6'-PeCB	104	0.2	1.0	5.0	50	400	2000
2,3,3',4,4'-PeCB	105	0.2	1.0	5.0	50	400	2000
2,3,4,4',5-PeCB	114	0.2	1.0	5.0	50	400	2000
2,3',4,4',5-PeCB	118	0.2	1.0	5.0	50	400	2000
2',3,4,4',5-PeCB	123	0.2	1.0	5.0	50	400	2000
3,3',4,4',5-PeCB	126	0.2	1.0	5.0	50	400	2000
2,2',4,4',6,6'-HxCB	155	0.2	1.0	5.0	50	400	2000
2,3,3',4,4',5-HxCB	156	0.2	1.0	5.0	50	400	2000
2,3,3',4,4',5'-HxCB	157	0.2	1.0	5.0	50	400	2000
2,3',4,4',5,5'-HxCB	167	0.2	1.0	5.0	50	400	2000

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3,3',4,4',5,5'-HxCB	169	0.2	1.0	5.0	50	400	2000
2,2',3,4',5,6,6'-HpCB	188	0.2	1.0	5.0	50	400	2000
2,3,3',4,4',5,5'-HpCB	189	0.2	1.0	5.0	50	400	2000
2,2',3,3',5,5',6,6'-OcCB	202	0.2	1.0	5.0	50	400	2000
2,3,3',4,4',5,5',6-OcCB	205	0.2	1.0	5.0	50	400	2000
2,2',3,3',4,4',5,5',6-NoCB	206	0.2	1.0	5.0	50	400	2000
2,2',3,3',4',5,5',6,6'-NoCB	208	0.2	1.0	5.0	50	400	2000
DeCB 209	209	0.2	1.0	5.0	50	400	2000
Labelled Toxics/LOC/window-defining							
13C12-2-MoCB	1L	100	100	100	100	100	100
13C12-4-MoCB	3L	100	100	100	100	100	100
13C12-2,2'-DiCB	4L	100	100	100	100	100	100
13C12-4,4'-DiCB	15L	100	100	100	100	100	100
13C12-2,2',6'-TrCB	19L	100	100	100	100	100	100
13C12-3,4,4'-TrCB	37L	100	100	100	100	100	100
13C12-2,2',6,6'-TeCB	54L	100	100	100	100	100	100
13C12-3,3',4,4'-TeCB	77L	100	100	100	100	100	100
13C12-3,4,4',5-TeCB	81L	100	100	100	100	100	100
13C12-2,2',4,6,6'-PeCB	104L	100	100	100	100	100	100
13C12-2,3,3',4,4'-PeCB	105L	100	100	100	100	100	100
13C12-2,3,4,4',5-PeCB	114L	100	100	100	100	100	100
13C12-2,3',4,4',5-PeCB	118L	100	100	100	100	100	100
13C12-2',3,4,4',5-PeCB	123L	100	100	100	100	100	100
13C12-3,3',4,4',5-PeCB	126L	100	100	100	100	100	100
13C12-2,2',4,4',6,6'-HxCB	155L	100	100	100	100	100	100
13C12-2,3,3',4,4',5-HxCB	156L	100	100	100	100	100	100
13C12-2,3,3',4,4',5'-HxCB	157L	100	100	100	100	100	100
13C12-2,3',4,4',5,5'-HxCB	167L	100	100	100	100	100	100
13C12-3,3',4,4',5,5'-HxCB	169L	100	100	100	100	100	100
13C12-2,2',3,4',5,6,6'-HpCB	188L	100	100	100	100	100	100
13C12-2,3,3',4,4',5,5'-HpCB	189L	100	100	100	100	100	100
13C12-2,2',3,3',5,5',6,6'-OcCB	202L	100	100	100	100	100	100
13C12-2,3,3',4,4',5,5',6-OcCB	205L	100	100	100	100	100	100
13C12-2,2',3,3',4,4',5,5',6-NoCB	206L	100	100	100	100	100	100
13C12-2,2',3,3',4',5,5',6,6'-NoCB	208L	100	100	100	100	100	100
13C12-DeCB 209L	209L	100	100	100	100	100	100
Labelled clean-up							
13C12-2,4,4'-TrCB	28L	100	100	100	100	100	100
13C12-2,3,3',5,5'-PeCB	111L	100	100	100	100	100	100
13C12-2,2',3,3',5,5',6-HpCB	178L	100	100	100	100	100	100
Labelled injection internal							
13C12-2,5-DiCB	9L	100	100	100	100	100	100
13C12-2,2',5,5'-TeCB	52L	100	100	100	100	100	100
13C12-2,2',4',5,5'-PeCB	101L	100	100	100	100	100	100
13C12-2,2',3',4,4',5'-HxCB	138L	100	100	100	100	100	100
13C12-2,2',3,3',4,4',5,5'-OcCB	194L	100	100	100	100	100	100

1. Suffix "L" indicates labelled compound
2. Additional concentration used for calibration of high sensitivity HRGC/HRMS systems
Solution concentration (ng/mL)

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Table 4. Concentrations of native and labelled chlorinated biphenyls in stock solutions, spiking solutions, and final extracts

CB congener	Solution concentrations	
Native Toxics/LOC	**Spiking (ng/mL)**	**Extract (ng/mL)**
1	50	50
3	50	50
4	50	50
5	50	50
19	50	50
37	50	50
54	50	50
77	50	50
81	50	50
104	50	50
105	50	50
114	50	50
118	50	50
123	50	50
126	50	50
155	50	50
156	50	50
157	50	50
167	50	50
169	50	50
188	50	50
189	50	50
202	50	50
205	50	50
206	50	50
208	50	50
209	50	50
Native congener mix stock solutions		
MoCB thru TrCB 2.5		
TeCB thru HpCB 5.0		
OcCB thru DeCB 7.5		
Labelled Toxics/LOC/window-defining		
1L	2.0	100
3L	2.0	100
4L	2.0	100
15L	100	100
19L	100	100
37L	100	100
54L	100	100
77L	100	100
81L	100	100
104L	100	100
105L	100	100
114L	100	100
118L	100	100
123L	100	100
126L	100	100
155L	100	100
156L	100	100
157L	100	100
167L	100	100
169L	100	100
188L	100	100
189L	100	100
202L	100	100
205L	100	100
206L	100	100
208L	100	100

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209L	100	100
Labelled clean-up		
28L	100	100
111L	100	100
178L	100	100
Labelled injection internal		
9L	400	100
52L	400	100
101L	400	100
138L	400	100
194L	400	100
Diluted combined 209 congener	**Solution concentration (ug/mL)**	
Standard	**Native**	**Labelled**
Native congeners		
MoCB thru TrCB	50	
TeCB thru HpCB	100	
OcCB thru DeCB	150	
Labelled Toxics/LOC/window-defining		100
Labelled Cleanup		100
Labelled Injection internal		100

Table 5. Theoretical ion abundance ratios and QC limits

Chlorine atoms	m/z's forming ratio	Theoretical ratio	Lower QC limit	Upper QC limit
1	m/(m+2)	3.13	2.66	3.6
2	m/(m+2)	1.56	1.33	1.79
3	m/(m+2)	1.04	0.88	1.2
4	m/(m+2)	0.77	0.65	0.89
5	(m+2)/(m+4)	1.55	1.32	1.78
6	(m+2)/(m+4)	1.24	1.05	1.43
7	(m+2)/(m+4)	1.05	0.89	1.21
8	(m+2)/(m+4)	0.89	0.76	1.02
9	(m+2)/(m+4)	0.77	0.65	0.89
10	(m+4)/(m+6)	1.16	0.99	1.33

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Table 6. Suggested Scan descriptors, levels of chlorination, m/z information, and substances monitored by HRGC/HRMS
NOTE: This table is an example only and to be used for guidance.

Function and chlorine level	m/z	m/z type	m/z formula	Substance
Fn-1; Cl-1	188.0393	M	12C12 H9 35Cl	Cl-1 CB
	190.0363	M+2	12C12 H9 37Cl	Cl-1 CB
	200.0795	M	13C12 H9 35Cl	13C12 Cl-1 CB
	202.0766	M+2	13C12 H9 37Cl	13C12 Cl-1 CB
	218.9856	lock	C4 F9	PFK
Fn-2; Cl-2,3	222.0003	M	12C12 H8 35Cl2	Cl-2 PCB
	223.9974	M+2	12C12 H8 35Cl 37Cl	Cl-2 PCB
	225.9944	M+4	12C12 H8 37Cl2	Cl-2 PCB
	234.0406	M	13C12 H8 35Cl2	13C12 Cl-2 PCB
	236.0376	M+2	13C12 H8 35Cl 37 Cl	13C12 Cl-2 PCB
	242.9856	lock	C6 F9	PFK
	255.9613	M	12C12 H7 35Cl3	Cl-3 PCB
	257.9584	M+2	12C12 H7 35Cl2 37Cl	Cl-3 PCB
Fn-3	255.9613	M	12C12 H7 35Cl3	Cl-3 PCB
Cl-3,4,5	257.9584	M+2	12C12 H7 35Cl2 37Cl	Cl-3 PCB
	259.9554	M+4	12C12 H7 35Cl 37Cl2	Cl-3 PCB
	268.0016	M	13C12 H7 35Cl3	13C12 Cl-3 PCB
	269.9986	M+2	13C12 H7 35Cl2 37Cl 13C12	Cl-3 PCB
	280.9825	lock	C6 F11	PFK
	289.9224	M	12C12 H6 35Cl4	Cl-4 PCB
	291.9194	M+2	12C12 H6 35Cl3 37Cl	Cl-4 PCB
	293.9165	M+4	12C12 H6 35Cl2 37Cl2	Cl-4 PCB
	301.9626	M	13C12 H6 35Cl4	13C12 Cl-4 PCB
	303.9597	M+2	13C12 H6 35Cl3 37Cl	13C12 Cl-4 PCB
	323.8834	M	12C12 H5 35Cl5	Cl-5 PCB
	325.8804	M+2 1	2C12 H5 35Cl4 37Cl	Cl-5 PCB
	327.8775	M+4	12C12 H5 35Cl3 37Cl2	Cl-5 PCB
	337.9207	M+2	13C12 H5 35Cl4 37Cl	13C12 Cl-5 PCB
	339.9178	M+4	13C12 H5 35Cl3 37Cl2	13C12 Cl-5 PCB
Fn-4	289.9224	M	12C12 H6 35Cl4	Cl-4 PCB
Cl-4,5,6	291.9194	M+2	12C12 H6 35Cl3 37Cl	Cl-4 PCB
	293.9165	M+4	12C12 H6 35Cl2 37Cl2	Cl-4 PCB
	301.9626	M+2	13C12 H6 35Cl3 37Cl	13C12 Cl-4 PCB
	303.9597	M+4	13C12 H6 35Cl2	13C12 Cl-4 PCB
	323.8834	M	12C12 H5 35Cl5	Cl-5 PCB
	325.8804	M+2	12C12 H5 35Cl4 37Cl	Cl-5 PCB
	327.8775	M+4	12C12 H5 35Cl3 37Cl2	Cl-5 PCB
	330.9792	lock	C7 F15	PFK
	337.9207	M+2	13C12 H5 35Cl4 37Cl 13C12	Cl-5 PCB
	339.9178	M+4	13C12 H5 35Cl3 37Cl2	13C12 Cl-5 PCB
	359.8415	M+2	13C12 H4 35Cl5 37Cl	Cl-6 PCB
	361.8385	M+4	13C12 H4 35Cl4 37Cl2	Cl-6 PCB
	363.8356	M+6	13C12 H4 35Cl3 37Cl2	Cl-6 PCB
	371.8817	M+2	13C12 H4 35Cl5 37Cl	13C12 Cl-6 PCB
	373.8788	M+4	13C12 H4 35Cl4 37Cl2	13C12 Cl-6 PCB
Fn-5	323.8834	M	12C12 H5 35Cl5	Cl-5 PCB
Cl-5,6,7	325.8804	M+2	12C12 H5 35Cl4 37Cl	Cl-5 PCB
	327.8775	M+4	12C12 H5 35Cl3 37Cl2	Cl-5 PCB
	337.9207	M+2	13C12 H5 35Cl4 37Cl	13C12 Cl-5 PCB
	339.9178	M+4	13C12 H5 35Cl3 37Cl2	13C12 Cl-5 PCB
	354.9792	lock	C9 F13	PFK
	359.8415	M+2	12C12 H4 35Cl5 37Cl	Cl-6 PCB
	361.8385	M+4	12C12 H4 35Cl4 37Cl2	Cl-6 PCB
	363.8356	M+6	12C12 H4 35Cl3 37Cl3	Cl-6 PCB
	371.8817	M+2	13C12 H4 35Cl5 37Cl	13C12 Cl-6 PCB
	373.8788	M+4	13C12 H4 35Cl4 37Cl2	13C12 Cl-6 PCB
	393.8025	M+2	12C12 H3 35Cl6 37Cl	Cl-7 PCB
	395.7995	M+4	12C12 H3 35Cl5 37Cl2	Cl-7 PCB
	397.7966	M+6	12C12 H3 35Cl4 37Cl3	Cl-7 PCB
	405.8428	M+2	13C12 H3 35Cl6 37Cl	13C12 Cl-7 PCB

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	407.8398	M+4	13C12 H3 35Cl5 37Cl2	13C12 Cl-7 PCB
	454.9728	QC	C11 F17	PFK
Fn-6	393.8025	M+2	12C12 H3 35Cl6 37Cl	Cl-7 PCB
Cl-7,8,9,10	395.7995	M+4	12C12 H3 35Cl5 37Cl2	Cl-7 PCB
	397.7966	M+6	12C12 H3 35Cl4 37Cl3	Cl-7 PCB
	405.8428	M+2	13C12 H3 35Cl6 37Cl 13C12	Cl-7 PCB
	407.8398	M+4	13C12 H3 35Cl5 37Cl2	13C12 Cl-7 PCB
	427.7635	M+2	12C12 H2 35Cl7 37Cl	Cl-8 PCB
	429.7606	M+4	12C12 H2 35Cl6 37Cl2	Cl-8 PCB
	431.7576	M+6	12C12 H2 35Cl5 37Cl3	Cl-8 PCB
	439.8038	M+2	13C12 H2 35Cl7 37Cl	13C12 Cl-8 PCB
	441.8008	M+4	13C12 H2 35Cl6 37Cl2	13C12 Cl-8 PCB
	442.9728	QC	C10 F13	PFK
	454.9728	lock	C11 F13	PFK
	461.7246	M+2	12C12 H1 35Cl8 37Cl	Cl-9 PCB
	463.7216	M+4	12C12 H1 35Cl7 37Cl2	Cl-9 PCB
	465.7187	M+6	12C12 H1 35Cl6 37Cl3	Cl-9 PCB
	473.7648	M+2	13C12 H1 35Cl8 37Cl	13C12 Cl-9 PCB
	475.7619	M+4	13C12 H1 35Cl7 37Cl2	13C12 Cl-9 PCB
	495.6856	M+2	13C12 H4 35Cl9 37Cl	Cl-10 PCB
	497.6826	M+4	12C12 35Cl8 37Cl2	Cl-10 PCB
	499.6797	M+6	12C12 35Cl7 37Cl3	Cl-10 PCB
	507.7258	M+2	13C12 H4 35Cl9 37Cl	13C12 Cl-10 PCB
	509.7229	M+4	13C12 H4 35Cl8 37Cl2	13C12 Cl-10 PCB
	511.7199	M+6	13C12 H4 35Cl8 37Cl4	13C12 Cl-10 PCB

1. Isotopic masses used for accurate mass calculation

1H	1.0078
12C	12.0000
13C	13.0034
35Cl	34.9689
37Cl	36.9659
19F	18.9984

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Table 7: LOD/LOQ Levels (DoD PJLA)

Target Analytes	Soils/Sediments/Solids pg/g	Tissues pg/g	Air (Stack/Ambient) pg	Water pg/L
PCB 1	2	2	20	20
PCB 3	2	2	20	20
PCB 4	10	10	100	100
PCB 15	10	10	100	100
PCB 19	2	2	20	20
PCB 37	2	2	20	20
PCB 54	2	2	20	20
PCB 81	2	2	20	20
PCB 77	2	2	20	20
PCB 104	2	2	20	20
PCB 123	2	2	20	20
PCB 118	2	2	20	20
PCB 114	2	2	20	20
PCB 105	2	2	20	20
PCB 126	2	2	20	20
PCB 155	2	2	20	20
PCB 167	2	2	20	20
PCB 156/157	4	4	40	40
PCB 169	2	2	20	20
PCB 188	2	2	20	20
PCB 189	2	2	20	20
PCB 202	2	2	20	20
PCB 205	2	2	20	20
PCB 208	2	2	20	20
PCB 206	2	2	20	20
PCB 209	2	2	20	20

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Table 8: Congeners, IUPAC numbers, CAS registry number, and labelled analogs.

CB congener(1)	IUPAC number	CAS registry number	Labelled analog	IUPAC number	CAS registry number
2-MoCB	1	2051-60-7	13C12-2-MoCB(2)	1L	234432-85-0
3-MoCB	2	2051-61-8			
4-MoCB	3	2051-62-9	13C12-4-MoCB(2)	3L	208263-77-8
2,2'-DiCB	4	13029-08-8	13C12-2,2'-DiCB(2)	4L	234432-86-1
2,3-DiCB	5	16605-91-7			
2,3'-DiCB	6	25569-80-6			
2,4-DiCB	7	33284-50-3			
2,4'-DiCB(3)	8	34883-43-7			
2,5-DiCB	9	34883-39-1	13C12-2,5-DiCB(4)	9L	250694-89-4
2,6-DiCB	10	33146-45-1			
3,3'-DiCB	11	2050-67-1			
3,4-DiCB	12	2974-92-7			
3,4'-DiCB	13	2974-90-5			
3,5-DiCB	14	34883-41-5			
4,4'-DiCB	15	2050-68-2	13C12-4,4'-DiCB(2)	15L	208263-67-6
2,2',3-TrCB	16	38444-78-9			
2,2',4-TrCB	17	37680-66-3			
2,2',5-TrCB(3)	18	37680-65-2			
2,2',6-TrCB	19	38444-73-4	13C12-2,2',6-TrCB(2)	19L	234432-87-2
2,3,3'-TrCB	20	38444-84-7			
2,3,4-TrCB	21	55702-46-0			
2,3,4'-TrCB	22	38444-85-8			
2,3,5-TrCB	23	55720-44-0			
2,3,6-TrCB	24	55702-45-9			
2,3',4-TrCB	25	55712-37-3			
2,3',5-TrCB	26	38444-81-4			
2,3',6-TrCB	27	38444-76-7			
2,4,4'-TrCB(3)	28	7012-37-5	13C12-2,4,4'-TriCB(5)	28L	208263-76-7
2,4,5-TrCB	29	15862-07-4			
2,4,6-TrCB	30	35693-92-6			
2,4',5-TrCB	31	16606-02-3			
2,4',6-TrCB	32	38444-77-8			
2',3,4-TrCB	33	38444-86-9			
2',3,5-TrCB	34	37680-68-5			
3,3',4-TrCB	35	37680-69-6			
3,3',5-TrCB	36	38444-87-0			
3,4,4'-TrCB	37	38444-90-5	13C12-3,4,4'-TrCB(2)	37L	208263-79-0
3,4,5-TrCB	38	53555-66-1			
3,4',5-TrCB	39	38444-88-1			
2,2',3,3'-TeCB	40	38444-93-8			
2,2',3,4-TeCB	41	52663-59-9			
2,2',3,4'-TeCB	42	36559-22-5			
2,2',3,5-TeCB	43	70362-46-8			
2,2',3,5'-TeCB(3)	44	41464-39-5			
2,2',3,6-TeCB	45	70362-45-7			
2,2',3,6'-TeCB	46	41464-47-5			
2,2',4,4'-TeCB	47	2437-79-8			
2,2',4,5-TeCB	48	70362-47-9			
2,2',4,5'-TeCB	49	41464-40-8			
2,2',4,6-TeCB	50	62796-65-0			
2,2',4,6'-TeCB	51	68194-04-7			
2,2',5,5'-TeCB(3)	52	35693-99-3	13C12-2,2',5,5'-TeCB(4)	52L	208263-80-3
2,2',5,6'-TeCB	53	41464-41-9			
2,2',6,6'-TeCB	54	15968-05-5	13C12-2,2',6,6'-TeCB(2)	54L	234432-88-3
2,3,3',4'-TeCB	55	74338-24-2			
2,3,3',4-TeCB	56	41464-43-1			
2,3,3',5-TeCB	57	70424-67-8			
2,3,3',5'-TeCB	58	41464-49-7			
2,3,3',6-TeCB	59	74472-33-6			
2,3,4,4'-TeCB	60	33025-41-1			
2,3,4,5-TeCB	61	33284-53-6			
2,3,4,6-TeCB	62	54230-22-7			
2,3,4',5-TeCB	63	74472-34-7			
2,3,4',6-TeCB	64	52663-58-8			
2,3,5,6-TeCB	65	33284-54-7			
2,3',4,4'-TeCB(3)	66	32598-10-0			
2,3',4,5-TeCB	67	73575-53-8			
2,3',4,5'-TeCB	68	73575-52-7			

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CB congener(1)	IUPAC number	CAS registry number	Labelled analog	IUPAC number	CAS registry number
2,3',4,6-TeCB	69	60233-24-1			
2,3',4',5-TeCB	70	32598-11-1			
2,3',4',6-TeCB	71	41464-46-4			
2,3',5,5'-TeCB	72	41464-42-0			
2,3',5',6-TeCB	73	74338-23-1			
2,4,4',5-TeCB	74	32690-93-0			
2,4,4',6-TeCB	75	32598-12-2			
2',3,4,5-TeCB	76	70362-48-0			
3,3',4,4'-TeCB(3,6)	77	32598-13-3	13C12-3,3',4,4'-TeCB(2,7)	77L	105600-23-5
3,3',4,5-TeCB	78	70362-49-1			
3,3',4,5'-TeCB	79	41464-48-6			
3,3',5,5'-TeCB	80	33284-52-5			
3,4,4',5-TeCB(6)	81	70362-50-4	13C12-3,4,4',5-TeCB(7)	81L	208461-24-9
2,2',3,3',4-PeCB	82	52663-62-4			
2,2',3,3',5-PeCB	83	60145-20-2			
2,2',3,3',6-PeCB	84	52663-60-2			
2,2',3,4,4'-PeCB	85	65510-45-4			
2,2',3,4,5-PeCB	86	55312-69-1			
2,2',3,4,5'-PeCB	87	38380-02-8			
2,2',3,4,6-PeCB	88	55215-17-3			
2,2',3,4,6'-PeCB	89	73575-57-2			
2,2',3,4',5-PeCB	90	68194-07-0			
2,2',3,4',6-PeCB	91	68194-05-8			
2,2',3,5,5'-PeCB	92	52663-61-3			
2,2',3,5,6-PeCB	93	73575-56-1			
2,2',3,5,6'-PeCB	94	73575-55-0			
2,2',3,5',6-PeCB	95	38379-99-6			
2,2',3,6,6'-PeCB	96	73575-54-9			
2,2',3',4,5-PeCB	97	41464-51-1			
2,2',3',4,6-PeCB	98	60233-25-2			
2,2',4,4',5-PeCB	99	38380-01-7			
2,2',4,4',6-PeCB	100	39485-83-1			
2,2',4,5,5'-PeCB(3)	101	37680-73-2	13C12-2,2',4,5,5'-PeCB(4)	101L	104130-39-4
2,2',4,5,6'-PeCB	102	68194-06-9			
2,2',4,5,'6-PeCB	103	60145-21-3			
2,2',4,6,6'-PeCB	104	56558-16-8	13C12-2,2',4,6,6'-PeCB(2)	104L	234432-89-4
2,3,3',4,4'-PeCB(3,6)	105	32598-14-4	13C12-2,3,3',4,4'-PeCB(7)	*105L*	208263-62-1
2,3,3',4,5-PeCB	106	70424-69-0			
2,3,3',4',5-PeCB	107	70424-68-9			
2,3,3',4,5'-PeCB	108	70362-41-3			
2,3,3',4,6-PeCB	109	74472-35-8			
2,3,3',4',6-PeCB	110	38380-03-9			
2,3,3',5,5'-PeCB	111	39635-32-0	13C12-2,3,3',5,5'-PeCB(5)	111L	235416-29-2
2,3,3',5,6-PeCB	112	74472-36-9			
2,3,3',5',6-PeCB	113	68194-10-5			
2,3,4,4',5-PeCB(6)	114	74472-37-0	13C12-2,3,4,4',5-PeCB(7)	114L	208263-63-2
2,3,4,4',6-PeCB	115	74472-38-1			
2,3,4,5,6-PeCB	116	18259-05-7			
2,3,4',5,6-PeCB	117	68194-11-6			
2,3',4,4',5-PeCB(3,6)	118	31508-00-6	13C12-2,3',4,4',5-PeCB(7)	118L	104130-40-7
2,3',4,4',6-PeCB	119	56558-17-9			
2,3',4,5,5'-PeCB	120	68194-12-7			
2,3',4,5,'6-PeCB	121	56558-18-0			
2',3,3',4,5-PeCB	122	76842-07-4			
2',3,4,4',5-PeCB(6)	123	65510-44-3	13C12-2',3,4,4',5-PeCB(7)	123L	208263-64-3
2',3,4,5,5'-PeCB	124	70424-70-3			
2',3,4,5,6'-PeCB	125	74472-39-2			
3,3',4,4',5-PeCB(3,6)	126	57465-28-8	13C12-3,3',4,4',5-PeCB(2,7)	126L	208263-65-4
3,3',4,5,5'-PeCB	127	39635-33-1			
2,2',3,3',4,4'-HxCB(3)	128	38380-07-3			
2,2',3,3',4,5-HxCB	129	55215-18-4			
2,2',3,3',4,5'-HxCB	130	52663-66-8			
2,2',3,3',4,6-HxCB	131	61798-70-7			
2,2',3,3',4,6'-HxCB	132	38380-05-1			
2,2',3,3',5,5'-HxCB	133	35694-04-3			
2,2',3,3',5,6-HxCB	134	52704-70-8			
2,2',3,3',5,6'-HxCB	135	52744-13-5			
2,2',3,3',6,6'-HxCB	136	38411-22-2			
2,2',3,4,4',5-HxCB	137	35694-06-5			
2,2',3,4,4',5'-HxCB(3)	138	35065-28-2	13C12-2,2',3,4,4',5'-HxCB(4)	138L	208263-66-5

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CB congener(1)	IUPAC number	CAS registry number	Labelled analog	IUPAC number	CAS registry number
2,2',3,4,4',6-HxCB	139	56030-56-9			
2,2',3,4,4',6'-HxCB	140	59291-64-4			
2,2',3,4,5,5'-HxCB	141	52712-04-6			
2,2',3,4,5,6-HxCB	142	41411-61-4			
2,2',3,4,5,6'-HxCB	143	68194-15-0			
2,2',3,4,5',6-HxCB	144	68194-14-9			
2,2',3,4,6,6'-HxCB	145	74472-40-5			
2,2',3,4',5,5'-HxCB	146	51908-16-8			
2,2',3,4',5,6-HxCB	147	68194-13-8			
2,2',3,4',5,6'-HxCB	148	74472-41-6			
2,2',3,4',5',6-HxCB	149	38380-04-0			
2,2',3,4',6,6'-HxCB	150	68194-08-1			
2,2',3,5,5',6-HxCB	151	52663-63-5			
2,2',3,5,6,6'-HxCB	152	68194-09-2			
2,2',4,4',5,5'-HxCB(3)	153	35065-27-1			
2,2',4,4',5',6-HxCB	154	60145-22-4			
2,2',4,4',6,6'-HxCB	155	33979-03-2	13C12-2,2',4,4',6,6'-HxCB(2)	155L	234432-90-7
2,3,3',4,4',5-HxCB(6)	156	38380-08-4	13C12-2,3,3',4,4',5-HxCB(7)	156L	208263-68-7
2,3,3',4,4',5'-HxCB(6)	157	69782-90-7	13C12-2,3,3',4,4',5'-HxCB(7)	157L	235416-30-5
2,3,3',4,4',6-HxCB	158	74472-42-7			
2,3,3',4,5,5'-HxCB	159	39635-35-3			
2,3,3',4,5,6-HxCB	160	41411-62-5			
2,3,3',4,5',6-HxCB	161	74472-43-8			
2,3,3',4',5,5'-HxCB	162	39635-34-2			
2,3,3',4',5,6-HxCB	163	74472-44-9			
2,3,3',4',5',6-HxCB	164	74472-45-0			
2,3,3',5,5',6-HxCB	165	74472-46-1			
2,3,4,4',5,6-HxCB	166	41411-63-6			
2,3',4,4',5,5'-HxCB(6)	167	52663-72-6	13C12-2,3',4,4',5,5'-HxCB(7)	167L	208263-69-8
2,3',4,4',5',6-HxCB	168	59291-65-5			
3,3',4,4',5,5'-HxCB(3,6)	169	32774-16-6	13C12-3,3',4,4',5,5'-HxCB(2,7)	169L	208263-70-1
2,2',3,3',4,4',5-HpCB(3)	170	35065-30-6			
2,2'3,3',4,4',6-HpCB	171	52663-71-5			
2,2',3,3',4,5,5'-HpCB	172	52663-74-8			
2,2',3,3',4,5,6-HpCB	173	68194-16-1			
2,2',3,3',4,5,6'-HpCB	174	38411-25-5			
2,2',3,3',4,5',6-HpCB	175	40186-70-7			
2,2',3,3',4,6,6'-HpCB	176	52663-65-7			
2,2',3,3',4',5,6-HpCB	177	52663-70-4			
2,2',3,3',5,5',6-HpCB	178	52663-67-9	13C12-2,2',3,3',5,5',6-HpCB(5)	178L	232919-67-4
2,2',3,3',5,6,6'-HpCB	179	52663-64-6			
2,2',3,4,4',5,5'-HpCB(3)	180	35065-29-3			
2,2',3,4,4',5,6-HpCB	181	74472-47-2			
2,2',3,4,4',5,6'-HpCB	182	60145-23-5			
2,2',3,4,4',5',6-HpCB	183	52663-69-1			
2,2',3,4,4',6,6'-HpCB	184	74472-48-3			
2,2',3,4,5,5',6-HpCB	185	52712-05-7			
2,2',3,4,5,6,6'-HpCB	186	74472-49-4			
2,2',3,4',5,5',6-HpCB(3)	187	52663-68-0			
2,2',3,4',5,6,6'-HpCB	188	74487-85-7	13C12-2,2',3,4',5,6,6'-HpCB(2)	188L	234432-91-8
2,3,3',4,4',5,5'-HpCB(6)	189	39635-31-9	13C12-2,3,3',4,4',5,5'-HpCB(2,7)	189L	208263-73-4
2,3,3',4,4',5,6-HpCB	190	41411-64-7			
2,3,3',4,4',5',6-HpCB	191	74472-50-7			
2,3,3',4,5,5',6-HpCB	192	74472-51-8			
2,3,3',4',5,5',6-HpCB	193	69782-91-8			
2,2',3,3',4,4',5,5'-OcCB	194	35694-08-7	13C12-2,2',3,3',4,4',5,5'-OcCB(4)	194L	208263-74-5
2,2',3,3',4,4',5,6-OcCB(3)	195	52663-78-2			
2,2',3,3',4,4',5,6'-OcCB	196	42740-50-1			
2,2',3,3',4,4',6,6'-OcCB	197	33091-17-7			
2,2',3,3',4,5,5',6-OcCB	198	68194-17-2			
2,2',3,3',4,5,5',6'-OcCB	199	52663-75-9			
2,2',3,3',4,5,6,6'-OcCB	200	52663-73-7			
2,2',3,3',4,5',6,6'-OcCB	201	40186-71-8			
2,2',3,3',5,5',6,6'-OcCB	202	2136-99-4	13C12-2,2',3,3',5,5',6,6'-OcCB(2)	202L	105600-26-8
2,2',3,4,4',5,5',6-OcCB	203	52663-76-0			
2,2',3,4,4',5,6,6'-OcCB	204	74472-52-9			
2,3,3',4,4',5,5',6-OcCB	205	74472-53-0	13C12-2,3,3',4,4',5,5',6-OcCB(2)	205L	234446-64-1
2,2',3,3',4,4',5,5',6-NoCB(3)	206	40186-72-9	13C12-2,2',3,3',4,4',5,5',6-NoCB(2)	206L	208263-75-6
2,2',3,3',4,4',5,6,6'-NoCB	207	52663-79-3			
2,2',3,3',4,5,5',6,6'-NoCB	208	52663-77-1	13C12-2,2',3,3',4,5,5',6,6'-NoCB(2)	208L	234432-92-9

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CB congener(1)	IUPAC number	CAS registry number	Labelled analog	IUPAC number	CAS registry number
DeCB(3)	209	2051-24-3	13C12-DeCB(2)	209L	105600-27-9

1. Abbreviations for chlorination levels
MoCB = monochlorobiphenyl
DiCB = dichlorobiphenyl
TrCB = trichlorobiphenyl
TeCB = tetrachlorbiphenyl
PeCB = pentachlorobiphenyl
HxCB = hexachlorobiphenyl
HpCB = heptachlorobiphenyl
OcCB = octachlorobiphenyl
NoCB = nonachlorobiphenyl
DeCB = decachlorobiphenyl
2. Labelled level of chlorination (LOC) window-defining congener
3. National Oceanic and Atmospheric Administration (NOAA) congener of interest
4. Labelled injection internal standard
5. Labelled clean-up standard
6. World Health Organization (WHO) toxic congener
7. Labelled analog of WHO toxic congener

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Table 9a: PCB LCS Limits for Solid/Tissue/Air Matrices (DoD Analysis Only)

Analyte	Lower Control Limit	UpperControl Limit
PCB 1	48	136
PCB 3	58	137
PCB 4	57	140
PCB 15	64	148
PCB 19	74	125
PCB 37	61	148
PCB 54	67	124
PCB 77	75	118
PCB 81	78	124
PCB 104	81	118
PCB 105	84	127
PCB 114	87	124

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PCB 118	79	137
PCB 123	81	134
PCB 126	79	123
PCB 155	76	121
PCB 156	84	125
PCB 167	82	132
PCB 169	77	121
PCB 188	78	117
PCB 189	85	119
PCB 202	76	118
PCB 205	72	128
PCB 206	74	121
PCB 208	80	120
PCB 209	83	133

* Sporadic marginal exceedances are allowed for those analytes outside the 3 standard deviation control limits but still within 4 standard deviations. Marginal exceedances are not allowed for those analytes determined by a project to be target analytes without project specific approval.

Table 9b: PCB LCS Limits for Water Matrix (DoD Analysis Only)

Analyte	Lower Control Limit	Upper Control Limit
PCB 1	58	115
PCB 3	64	123
PCB 4	62	128
PCB 15	74	133
PCB 19	79	118
PCB 37	60	134
PCB 54	67	123
PCB 77	75	113
PCB 81	77	125
PCB 101	75	132
PCB 104	79	120
PCB 105	77	133
PCB 114	81	133
PCB 118	82	128
PCB 123	76	138
PCB 126	78	119
PCB 128	79	126

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PCB 155	69	126
PCB 156	78	137
PCB 167	78	143
PCB 169	71	123
PCB 170	78	138
PCB 188	76	115
PCB 189	81	124
PCB 202	77	114
PCB 205	69	122
PCB 206	74	113
PCB 208	79	118
PCB 209	80	128

* Sporadic marginal exceedances are allowed for those analytes outside the 3 standard deviation control limits but still within 4 standard deviations. Marginal exceedances are not allowed for those analytes determined by a project to be target analytes without project specific approval.

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PCDD/F BY HRMS

Document Authorization

Authorized By: _____ Date: _____
Quality Authorization

Authorized By: _____ Date: _____
Technical Authorization

Implementation Authorization

Authorized By: _____ Date: _____
Laboratory Manager

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1.0 SCOPE AND APPLICATION

1.1 Scope

This method provides procedures for the instrumental detection and quantitative measurement of polychlorinated dibenzo(p)dioxins (tetra- through octachlorinated homomlogues; PCDD) and polychlorinated dibenzofurans (tetra- through octachlorinated homologues; PCDF) from extracts via isotope dilution high resolution GC/MS. For the sample extraction, clean-up and preparation of extracts for this instrumental method see standard method BU-TM-1110 Overall HR Prep and procedures referenced therein. Variations of this instrumental method herein and the preparative method BU-TM-1110 incorporate the concurrent analysis of selected PCB congeners (see BU-TM-1113 & BU-TP-2113).

The method covers the ultra-trace analysis (ppq to ppt) of extracts prepared from most environmental, tissues and edible materials including matrices such as air (stack and ambient), soils, sediments, other solids, potable water, non-potable water, and food (including but not limited to dairy, eggs, meat, fresh fruits and vegetables, edible oils, processed foods), blood serum and other tissues.

It incorporates the analysis of PCDD/F via the following reference methods:

US EPA Office of Water, Method 1613B
US EPA Office of Solid Waste, SW846 Methods 8290A and 0023/8290A
US EPA Office of Research & Development Method TO-9A.
US EPA Office of Air Quality Planning & Standards Method 23.

1.2 Parameters and Limits of Reporting

Instrument calibration range is located in Table 2.

For PCDD/F analyses as defined by this method, the MDLs are not normally used to define the reporting limits. The most common reporting approach is to report each congener or isomer group to 2.5:1 signal to noise (an estimated detection limit; EDL) as described in OSW SW846 Method 8290A.

1.2.1 Environmental Matrices

Reporting limits for PCDD/F in environmental matrices are presented in the table below.

Parameters	Solid (Mat#45) Soil (Mat# 2) Tissue (Mat# 20) [pg/g] Based on 10g sample size	Air (Mat# 44) Puf (Mat# 16) [pg/sample]	Water (Mat# 1) Waste (Mat# 24) [pg/L]
2,3,7,8-Tetrachlorodibenzo(p)dioxin (TCDD)	0.4	4.00	2.00

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1,2,3,7,8-Pentachlorodibenzo(p)dioxin (PeCDD	0.4	4.00	2.00
1,2,3,4,7,8-Hexachlorodibenzo(p)dioxin (HxCDD)	0.4	4.00	2.00
1,2,3,6,7,8-Hexachlorodibenzo(p)dioxin (HxCDD)	0.4	4.00	2.00
1,2,3,7,8,9-Hexachlorodibenzo(p)dioxin (HxCDD)	0.4	4.00	2.00
1,2,3,4,6,7,8-Heptachlorodibenzo(p)dioxin (HpCDD)	0.4	4.00	5.00
Octachlorodibenzo(p)dioxin (OCDD)	1.00	10.00	10.00
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	0.4	4.00	2.00
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	0.4	4.00	2.00
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	0.4	4.00	2.00
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	0.4	4.00	2.00
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	0.4	4.00	2.00
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	0.4	4.00	2.00
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	0.4	4.00	2.00
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	0.4	4.00	2.00
1,2,3,4,7,8,9-Heptapchlorodibenzofuran (HpCDF)	0.4	4.00	2.00
Octachlorodibenzofuran (OCDF)	1.00	10.00	10.00

These reflect EVOLIMS posted reporting limits based upon NA-SP-0102 Method Revalidation and MU - Chemistry.

ALS Burlington typically reports to EDLs (see Section 10.2) based upon the observed 2.5:1 signal to noise.

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1.2.2 Food Matrices

Detection limits (DL) and limits of Quantification (LOQ) for PCDD/F in food matrices with validated analyses are presented in the table below.

In regards to the CFIA RFSO #39903-150123/B and standing offers derived from this solicitation, all of the DL and LOQ listed below meet the respective individual food group towards the minimum requirements as referenced in Annex "A" of the RFSO.

	CFIA CRDL	LOQ	Dairy		Eggs		Meat		Fresh FV		Processed Foods	
			Soxhlet	Base Digestion	Soxhlet	Base Digestion	Soxhlet	Base Digestion	Soxhlet	Base Digestion	Soxhlet	Base Digestion
Sample Size (g):		**20**	**20**	**100**	**20**	**100**	**20**	**100**	**20**	**100**	**20**	**100**
DL Determination Method:			**MDL**	**EDL**	**MDL**	**EDL**	**MDL**	**EDL**	**MDL**	**EDL**	**MDL**	**EDL**
Target Analytes	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g	ng/Kg or pg/g
2,3,7,8-TCDD	0.1	0.5	0.01	0.013	0.01	0.013	0.01	0.013	0.01	0.013	0.01	0.013
1,2,3,7,8-PeCDD	0.1	2.5	0.05	0.010	0.05	0.010	0.05	0.010	0.05	0.010	0.05	0.010
1,2,3,4,7,8-HxCDD	0.2	2.5	0.03	0.010	0.03	0.010	0.03	0.010	0.03	0.010	0.03	0.010
1,2,3,6,7,8-HxCDD	0.2	2.5	0.02	0.010	0.02	0.010	0.02	0.010	0.02	0.010	0.02	0.010
1,2,3,7,8,9-HxCDD	0.2	2.5	0.04	0.009	0.04	0.009	0.04	0.009	0.04	0.009	0.04	0.009
1,2,3,4,6,7,8-HpCDD	0.2	2.5	0.03	0.073	0.03	0.073	0.03	0.073	0.03	0.073	0.03	0.073
OCDD	0.5	5	0.31	0.017	0.31	0.017	0.31	0.017	0.31	0.017	0.31	0.017
2,3,7,8-TCDF	0.1	0.5	0.01	0.010	0.01	0.010	0.01	0.010	0.01	0.010	0.01	0.010
1,2,3,7,8-PeCDF	0.2	2.5	0.03	0.008	0.03	0.008	0.03	0.008	0.03	0.008	0.03	0.008
2,3,4,7,8-PeCDF	0.1	2.5	0.06	0.008	0.06	0.008	0.06	0.008	0.06	0.008	0.06	0.008
1,2,3,4,7,8-HxCDF	0.1	2.5	0.03	0.006	0.03	0.006	0.03	0.006	0.03	0.006	0.03	0.006
1,2,3,6,7,8-HxCDF	0.2	2.5	0.03	0.006	0.03	0.006	0.03	0.006	0.03	0.006	0.03	0.006
2,3,4,6,7,8-HxCDF	0.2	2.5	0.01	0.008	0.01	0.008	0.01	0.008	0.01	0.008	0.01	0.008
1,2,3,7,8,9-HxCDF	0.2	2.5	0.05	0.008	0.05	0.008	0.05	0.008	0.05	0.008	0.05	0.008
1,2,3,4,6,7,8-HpCDF	0.2	2.5	0.03	0.029	0.03	0.029	0.03	0.029	0.03	0.029	0.03	0.029
1,2,3,4,7,8,9-HpCDF	0.2	2.5	0.04	0.048	0.04	0.048	0.04	0.048	0.04	0.048	0.04	0.048
OCDF	0.2	5	0.09	0.033	0.09	0.033	0.09	0.033	0.09	0.033	0.09	0.033
Average PCDD/F Target			0.051	0.018	0.051	0.018	0.051	0.018	0.051	0.018	0.051	0.018

CRDL = Contract required detection limits as defined in the CFIA RFSO 39903-150123/B
MDL = DL as determined by the method defined in the US Code of Registry Volume 40 Appendix B to Part 136.
EDL = DL as determined by an average set of detection limits determined from 2.5:1 signal to noise on real sample analyses.
LOQ = Limit of Quantification as defined by the level equivalent to the low calibration standard

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1.3 Method Performance Data and Measurement Uncertainty

Refer to NA-SP-0100 Initial Method Validation – Chemistry and NA-SP-0102 Method Revalidation and MU – Chemistry for policies on determination and storage of method performance data.

2.0 PRINCIPLE OF METHOD

Extracts are analyzed via HRMS at resolution greater than 1 in 10,000 and quantified via isotope dilution technique.

3.0 DEFINITIONS

Refer to NA-SP-0001 Definitions of Key Terms for definitions of common terms.

1. **Bias** – Characterizes the systematic error in a given analytical procedure and is the (positive or negative) deviation of the mean analytical result from the (known or assumed) true value. (Eurachem Guide 1998 - IUPAC Compendium of Chemical Technology, 1985)

2. **Limit of Quantitation (LOQ)** – The lower limit of concentration or amount of substance that must be present before a method is considered to provide quantitative results. By convention, LOQ = $10s_0$, where s_0 is the estimate of the standard deviation at the lowest level of measurement. (Talyor 1987). Alternatively, LOQ can be defined as the concentration equivalent to the low concentration calibration standard.

3. **Laboratory Duplicates (LD1 and LD2)** - Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of LD1 and LD2 indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures. (EPA 1994)

For isotope dilution analytical methods, there is inconsistent use of terms within the literature and reference methods. This inconsistency is avoided herein by applying terms that are defined by the timing of their addition rather than by any other the use in the calculation of the data.

Extraction Standards: are the labelled analytes added just prior to extraction. For the data calculations, these are true internal standards - used for determining analyte concentration.

Clean-up Standard(s): is (are) the labeled analyte(s) added just prior to clean-up. Recoveries of the clean-up standard(s) provide an indication on the losses that occur during the extract clean-up and preparation for instrumental analysis.

Injection Standards: are the labelled analytes added just prior to instrumental analysis. These are used as an internal standard reference to determine the recoveries of the extraction and clean-up standards.

Sampling Standard: (also called field spikes) are the labelled analytes added, where

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applicable, prior to sampling onto sorbent media used for analyte capture during the sampling event.

4.0 INTERFERENCES

Prudent application of appropriate clean-ups generally provides extracts that are interference free. Occasionally, despite reasonable efforts during clean-up interferences can be observed. Chlorinated and brominated species (e.g. polychlorinated diphenyl ethers, polybrominated dioxins/furans and polybrominated diphenyl ethers) are commonly observed interferences. Phthalate esters can contaminate extracts through handling and provide interferences in PCDD/F instrumental analyses.

5.0 SAMPLE HANDLING AND STORAGE

Parameter(s) and Matrix: All parameters – Stack	
Container	Pre-cleaned amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE). Glass Petri dishes are not recommended for transportation of filters to and from the field since these are susceptible to breakage during transportation. Reference: EPA SW 846 Chapter 4, Rev 5, July 2014
Lab Storage Conditions	≤6˚C in the dark Reference: EPA Method 8290A Section 8.3
Maximum Sample Holding Time (Provinces / Reference)	30 days Reference: EPA Method 23 Section 5, 0023A Section 7.3.3, 8290A Section 8.3
Maximum Extract / Digest Holding Time (Provinces / References)	45 days Reference: EPA Method 23 Section 5, 0023A Section 7.3.3, 8290A Section 8.3

Parameter(s) and Matrix: All parameters – Ambient	
Container	Pre-cleaned amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE). Glass Petri dishes are not recommended for transportation of filters to and from the field since these are susceptible to breakage during transportation. Reference: EPA SW 846 Chapter 4, Rev 5, July 2014
Lab Storage Conditions	4˚C Reference: EPA TO-9A Section 11.3.4.9
Maximum Sample Holding Time (Provinces / Reference)	7 days Reference: EPA TO-9A Section 11.3.4.9
Maximum Extract / Digest Holding Time (Provinces / References)	40 days Reference: EPA TO-9A Section 11.3.4.9

Parameter(s) and Matrix: All parameters via 1613B – Water	
Container	Pre-cleaned amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE) Reference: EPA SW 846 Chapter 4, Rev 5, July 2014
Preservative	Ontario Drinking Waters: unpreserved Reference: MOE DW Practices Waters (others): 80mg/L of sodium thiosulphate to remove residual

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Parameter(s) and Matrix: All parameters via 1613B – Water	
	chlorine by ALS upon receipt Reference: EPA 1613B Section 8.2
Sample Pre-treatment	Waters (non-drinking waters): Tested for pH and adjusted if necessary. See BU-WI-0325 Water Preservation.
Lab Storage Conditions	Ontario Drinking Waters: 2-8°C (Ref: MOE DW Practices) Waters (others): >0 - 6°C (Ref: US EPA 40CFR 2012)
Maximum Sample Holding Time (Provinces / Reference)	Ontario Drinking Waters: 30 days (Ref: MOE DW Practices) Waters (others): 365 days (Ref: 1613B Section 8.4.1)
Maximum Extract / Digest Holding Time (Provinces / References)	Ontario Drinking Waters: 30 days (Ref: MOE DW Practices) Waters (others): 365 days @ ≤ -10°C (Ref: 1613B Section 8.4.2)

Parameter(s) and Matrix: All parameters via 1613B – Soil/Sediment/Solid/Waste (including Non-perishable Feed/Foods/Nutraceuticals)	
Container	Pre-cleaned amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE) Reference: EPA SW 846 Chapter 4, Rev 5, July 2014
Lab Storage Conditions	>0 - 6°C Ref: US EPA 40CFR 2012
Maximum Sample Holding Time (Provinces / Reference)	365 Days Reference: 1613B Section 8.4.1
Maximum Extract / Digest Holding Time (Provinces / References)	365 Days Reference: 1613B Section 8.4.2

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Parameter(s) and Matrix: All parameters via 1613B – Tissue (including perishable Feed/Foods/Nutraceuticals)	
Container	Pre-cleaned amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE). Solid tissues such as whole fish can be wrapped in solvent cleaned aluminum foil. Reference: EPA SW 846 Chapter 4, Rev 5, July 2014
Lab Storage Conditions	≤ -10˚C Reference: 1613B Section 8.2.3
Maximum Sample Holding Time (Provinces / Reference)	365 days Reference: 1613B Section 8.4.1
Maximum Extract / Digest Holding Time (Provinces / References)	365 days @ ≤ -10˚C Reference: 1613B Section 8.4.2

ALS requirements are: to store all samples and extracts in the dark and to comply with the method recommended hold times and temperature storage requirements except that solids destined for 8290A analysis may be stored frozen at <-10˚C. Short term storage of extracts (i.e. prior to the completion of analysis) is in a refrigerator at 4˚C. Long term storage of extracts is below -10˚C.

5.1 Sampling Instructions

Sample collection is not conducted by the laboratory.

5.2 Sample Handling and Transportation

Refer to ALS Sample Handling and Preservation Guidelines for further details on the sample handling procedures recommended by ALS.

If holding time violations occur, Account Manager(s) must be notified.

6.0 EQUIPMENT AND SUPPLIES

6.1 Primary Equipment

6.1.1 Autosamplers

Capable of dependable automated injection of liquid extracts down to 1uL, including pre- and post-injection automated syringe rinsing. Examples:

- CTC PAL GC-xt Auto-Sampler or functionally equivalent or superior, as provided with, or upgraded on, Waters Autospec HRMS systems.
- PAL RSI Autosampler, or functionally equivalent as configured for use on Thermo DFS HRMS systems.

6.1.2 Gas Chromatograph

Capable of temperature programming with isothermal hold, and temperature-controlled injection capability. Electronic Pressure Control recommended but not essential. Examples:

- Agilent 7890B GC or functionally equivalent or superior, as provided with, or upgraded on, Waters Autospec HRMS systems.

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- Thermo Trace Ultra GC or functionally equivalent, as configured for use on Thermo DFS HRMS systems.

6.1.3 High Resolution Mass Spectrometer

Capable of repetitive and selective monitoring of at least 12 exact m/z's at high resolution (10,000) during a period of approximately one second. Examples:
- Micromass (now Waters) Autospec Premier series HRMS or functionally equivalent or superior.
- Thermo DFS

6.1.4 GC column

See Section 9.3.4.2 for resolution criteria and column choices. Primary analysis is performed using an Agilent DB5 or DB5MS column. If confirmatory analysis is required, an Agilent DB225 column is used.

- Agilent J&W DB5 or DB5MS 60m x 0.25mm id x 0.1um film thickness (Agilent, Supplier #122-5561, MEOL# 4321064) [primary analytical column] or equivalent capable of uniquely separating 2,3,7,8-TCDD.

- DB225 30m x 0.25mm x 0.15um film thickness (Agilent, Supplier # 122-2231, MEOL# 4320189) [confirmation column]

Specific and detailed instrument operational settings and conditions are listed in the corresponding HRMS application notes BU-WI-3206 PCDD_F DB5MS HRMS-AutoSpec M&S Series, BU-WI-3200 PCDD_F DB5MS HRMS-AutoSpec Premier, BU-WI-3207 PCDD_F DB5MS HRMS-DFS, and BU-WI-3208 PCDD_F DB225 HRMS-AutoSpec M&S Series.

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6.2 Support Equipment

1. Rotovap
2. Centrifuge
3. N-Evap
4. Sonicator

6.3 Software

1. MassLynx/TargetLynx
2. Thermo Xcalibur

6.4 Supplies

6.4.1 Sample Prep Supplies

Refer to BU-TM-1110 Overall HR Prep

6.4.2 Instrument Supplies

1. Double gooseneck liner, Chromatographic Specialities, MEOL# 3080282 (Autospec); gooseneck liner, Chromatographic Specialities, MEOL 4320940 (DFS)
2. Syringe: 26 gauge, Chromtographic Specialities, MEOL#3300213 (Autospec); Mandel Scientific, MEOL# 4321972 (DFS)
3. DB5ms 60mx0.25mmx0.1µm column, Agilent, MEOL# 4321064

6.4.3 Standard Preparation Supplies

1. 10 µL-5000 µL syringes, Hamilton, MEOL # for various syringes can be found in G:\Enviro\Purchasing\Organic Prep\Approved Supplies.xls (Syringes Tab)
2. 2 mL Robovials (MEOL# 4001399) and Lids (MEOL# 4000796) with septa (MEOL# 3300552)
3. 5" pipettes (MEOL# 4430061)
4. 15 mL Centrifuge tubes (C-tubes) MEOL# 4000187
5. 50 mL Centrifuge tubes (C-tubes) MEOL# 4000202
6. 7 mL Graduated screw cap vials with solid PTFE lined caps MEOL# 4000687 (vial), MEOL# 4010593 (cap)
7. 22 mL Graduated screw cap vials with solid PTFE lined caps MEOL# 4000738 (vial), MEOL# 4010609 (cap)

7.0 REAGENTS AND STANDARDS

The following reagents and standards have been shown to meet the performance specifications of this test method. Alternate sources may be substituted where equivalent performance and suitability has been verified. Refer to site-specific reagent and standard preparation logs and analytical records to identify the reagents and standards in current use at each lab. Refer to BU-SP-0250 Control of Reagents and Reference Materials.

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7.1 Reagents

All solvents must be analytical grade or better.

1. Nonane, Glass Distilled, Stored at ambient temperature. Stored in standard fumehood. Expiry date: 3 months following distillation. Supplier: Caledon Laboratories Ltd; nonane, solvent dig 500ml, MEOL# 6010079.

2. Toluene, Glass Distilled, Stored at ambient temperature. Stored in flammable materials storage cabinet. Expiry date: 1 year following date of receipt. Supplier: Caledon, Supplier # 9201-2-40; MEOL # 6010007

3. Perfluorokerosene, either high-boiling or low-boiling may be used. Stored at ambient temperature. Expiry date: 5 years after opening. Supplier: Cambridge Isotope Laboratories; PFK-HIGH-1, MEOL# 5005748.

4. Helium, Ultra High Purity, Expiry date: 6 months. Supplier: Praxair; HE 5.0UH-T, ordered through Praxair website.

5. Perfluorotributylamine (PFTBA). Store at ambient temperature. Expiry: 5 years following the year of receipt. Supplier: Agilent, Supplier # 8500-0656, MEOL# 6040032

7.2 Standards

All standards are stored in the fridge in screw cap vials with PTFE lids. Unless otherwise indicated, the default expiry date is 1 year, or longer if revalidation is done. Refer to 'BU-WI-3002 Standards Prep, Spiking and Witnessing' for required protocols for standard preparation.

7.2.1 Extraction Standard Preparation

7.2.1.1 Extraction Standard Supplier Information

Expiry dates are stated on the supplier certificate of analysis.

7.2.1.2 Individual Stock Extraction Standards

Extraction Standard Compound	MEOL No.	Supplier	Cat. No.	D%
13C12-2,3,7,8-TCDD	5005098	Wellington Laboratories	MDD-2378	>99%
13C12-1,2,3,7,8-PCDD	5005096	Wellington Laboratories	MDD-12378	>99%
13C12-1,2,3,4,7,8-HxCDD	5005069	Wellington Laboratories	MDD123478	>99%
13C12-1,2,3,6,7,8-HxCDD	5005095	Wellington Laboratories	MDD-123678	>99%
13C12-1,2,3,4,6,7,8-HpCDD	5004759	Wellington Laboratories	MDD-1234678	>99%
13C12-OCDD	5004423	Wellington Laboratories	MDD-12346789	>99%
13C12-2,3,7,8-TCDF	5005101	Wellington Laboratories	MDF-2378	>99%
13C12-1,2,3,7,8-PCDF	5005099	Wellington Laboratories	MDF-12378	>99%
13C12-2,3,4,7,8-PCDF	5005072	Wellington Laboratories	MDD-23478	>99%
13C12-1,2,3,4,7,8-HxCDF	5005070	Wellington Laboratories	MDF-123478	>99%

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13C12-1,2,3,6,7,8-HxCDF	5004760	Wellington Laboratories	MDF-123678	>99%
13C12-1,2,3,7,8,9-HxCDF	5005100	Wellington Laboratories	MDF-123789	>99%
13C12-2,3,4,6,7,8-HxCDF	5005196	Wellington Laboratories	MDF-234678	>99%
13C12-1,2,3,4,6,7,8-HpCDF	5005071	Wellington Laboratories	MDF-1234678	>99%
13C12-1,2,3,4,7,8,9-HpCDF	5005071	Wellington Laboratories	MDF-1234789	>99%
13C12-OCDF	5004424	Wellington Laboratories	MDF-12346789	>99%

7.2.1.3 Extraction Stock Standard Solution Preparation (1613B-ES$1)

Standard	Concentration (ng/mL)	Amount Added (µl)	Final Concentration (ng/mL)
MDD-2378	50000	200.0	1000
MDD-12378	50000	200.0	1000
MDD123478	50000	200.0	1000
MDD-123678	50000	200.0	1000
MDD-1234678	50000	200.0	1000
MDD-12346789	50000	400.0	2000
MDF-2378	50000	200.0	1000
MDF-12378	50000	200.0	1000
MDD-23478	50000	200.0	1000
MDF-123478	50000	200.0	1000
MDF-123678	50000	200.0	1000
MDF-123789	50000	200.0	1000
MDF-234678	50000	200.0	1000
MDF-1234678	50000	200.0	1000
MDF-1234789	50000	200.0	1000

1. Using a syringe, add 6800 µL of nonane to a c-tube.
2. Using syringes of appropriate size, add the amount shown of each standard to the nonane.
3. The final volume should be 10.0 mL and the final concentration for each target is listed.
4. Aliquot into labeled robovials.
5. Name the standard 1613B-ES$1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.1.4 Extraction Working Standard Solution Preparation (1613B-ES#1)

Standard	Concentration (ng/mL)	Amount Added (µl)	Final Concentration (ng/mL)
1613B-ES$1-xxx	1000-2000	2000.0	100-200

1. Using a syringe, add 18.0 ml of nonane to a c-tube.
2. Using syringes of appropriate size, add the amount shown of each standard to the nonane.
3. The final volume should be 20.0 mL and the final concentration for each target is listed.
4. Aliquot into labeled robovials.

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5. Name the standard 1613B-ES#1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.1.5 Extraction Stock Standard Solution Preparation for Calibration Standards (DTOXM-ES$2)

Standard	Concentration (ng/mL)	Amount Added (µl)	Final Concentration (ng/mL)
MDD-2378	50000	300.0	1000
MDD-12378	50000	300.0	1000
MDD-123478	50000	300.0	1000
MDD-123678	50000	300.0	1000
MDD-123789	50000	300.0	1000
MDD-1234678	50000	300.0	1000
MDD-12346789	50000	600.0	2000
MDF-2378	50000	300.0	1000
MDF-12378	50000	300.0	1000
MDD-23478	50000	300.0	1000
MDF-123478	50000	300.0	1000
MDF-123678	50000	300.0	1000
MDF-123789	50000	300.0	1000
MDF-234678	50000	300.0	1000
MDF-1234678	50000	300.0	1000
MDF-1234789	50000	300.0	1000
MDF-12346789	50000	600.0	2000

1. Using a syringe, add 9300 µL of nonane to a c-tube.
2. Using syringes of appropriate size, add the amount shown of each standard to the nonane.
3. The final volume should be 15.0 mL and the final concentration for each target is listed.
4. Aliquot into labeled robovials.
5. Name the standard DTOXM-ES$2-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.2 Injection Standard Preparation

Expiry dates are stated on the supplier certificate of analysis.

7.2.2.1 Individual Stock Injection Standards

Injection Standard Compound	MEOL No.	Supplier	Cat. No.	D%
13C12-1,2,3,7,8,9-HxCDD	5005097	Wellington Laboratories	MDD-123789	>99%
13C12-1,2,3,4-TCDD	5005094	Wellington Laboratories	MDD-1234	>99%
13C12-1,2,7,8-TCDF	5006526	Wellington Laboratories	MDF-1278	>99%
13C12-1,2,4,7,8-PeCDD	5004972	Wellington Laboratories	MDD-12478	>99%
13C12-1,2,3,4,6,7,9-HpCDD	5004973	Wellington Laboratories	MDD-1234679	>99%

7.2.2.2 Injection Working Standard Solution Preparation (1613B-IS#1)

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Standard	Concentration (ng/mL)	Amount Added (µl)	Final Concentration (ng/mL)
MDD-123789	50000	40.0	100
MDD-1234	50000	40.0	100

1. Using a syringe, add 199200 µL of nonane to a c-tube.
2. Using syringes of appropriate size, add the amount shown of each standard to the nonane.
3. The final volume should be 20.0 mL and the final concentration for each target is listed.
4. Aliquot into labeled robovials.
5. Name the standard 1613B-IS#1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.2.3 Injection Stock Standard Solution Preparation for Calibration Standards (1613B-IS$1)

Standard	Concentration (ng/mL)	Amount Added (µl)	Final Concentration (ng/mL)
MDD-123789	50000	60.0	2000
MDD-1234	50000	60.0	2000
MDF-1278	50000	60.0	2000
MDD-12478	50000	60.0	2000
MDD-1234679	50000	60.0	2000

1. Using a syringe, add 1200 µL of nonane to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard to the nonane.
3. The final volume should be 1.5 mL and the final concentration for each target is listed.
4. Name the standard 1613B-IS$1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.3 Cleanup Standard Preparation

Expiry dates are stated on the supplier certificate of analysis.

7.2.3.1 Individual Stock Cleanup Standard

Cleanup Standard Compound	MEOL No.	Supplier	Cat. No.	D%
37Cl4-2,3,7,8-TCDD	5005068	Wellington Laboratories	MCDD-2378	>94%

7.2.3.2 Cleanup Stock Standard Solution Preparation (1613B-CL$1)

Standard	Concentration (ng/mL)	Amount Added (µl)	Final Concentration (ng/mL)
MCDD-2378	50000	100.0	5000

1. Using a syringe, add 900 µL of nonane to a labeled robovial.

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2. Using syringes of appropriate size, add the amount shown of each standard to the nonane.
3. The final volume should be 1.0 mL and the final concentration for each target is listed.
4. Name the standard 1613B-CL$1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.3.3 Cleanup Working Standard Solution Preparation (1613B-CL#2)

Standard	Concentration (ng/mL)	Amount Added (µl)	Final Concentration (ng/mL)
1613B-CL$1-xxx	5000	10.0	2.0

1. Using a syringe, add 24990 µL of nonane to a c-tube.
2. Using syringes of appropriate size, add the amount shown of each standard to the nonane.
3. The final volume should be 25.0 mL and the final concentration for each target is listed.
4. Aliquot into labeled robovials
5. Name the standard 1613B-CL#2-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.3.4 Cleanup Stock Standard Solution Preparation for Calibration Standards (1613B-CL$2)

Standard	Concentration (ng/mL)	Amount Added (µl)	Final Concentration (ng/mL)
1613B-CL$1-xxx	100	20.0	100

1. Using a syringe, add 980 µL of nonane to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard to the nonane.
3. The final volume should be 1.0 mL and the final concentration for each target is listed.
4. Name the standard 1613B-CL$2-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.4 Native Standard Preparation

Expiry dates are stated on the supplier certificate of analysis.

7.2.4.1 Cambridge Isotope Laboratories Native Mix Catalogue# EDF-7999-10X, MEOL# 5003388

Compound	Concentration (ng/mL)
2,3,7,8-TCDD	400
2,3,7,8-TCDF	400
1,2,3,7,8-PCDD	2000
1,2,3,7,8-PCDF	2000
2,3,4,7,8-PCDD	2000

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1,2,3,4,7,8-HxCDD	2000
1,2,3,6,7,8-HxCDD	2000
1,2,3,7,8,9-HxCDD	2000
1,2,3,4,7,8-HxCDF	2000
1,2,3,6,7,8-HxCDF	2000
1,2,3,7,8,9-HxCDF	2000
2,3,4,6,7,8-HxCDF	2000
1,2,3,4,6,7,8-HpCDD	2000
1,2,3,4,6,7,8-HpCDF	2000
1,2,3,4,7,8,9-HpCDF	2000
OCDD	4000
OCDF	4000

7.2.4.2 Native Working Standard Solution Preparation (1613B-NS#3)

Standard	Concentration (ng/mL)	Amount Added (µl)	Final Concentration (ng/mL)
EDF-7999-10X	400-4000	250.0	10-100

1. Using a syringe, add 9750 µL of nonane to a c-tube.
2. Using syringes of appropriate size, add the amount shown of each standard to the nonane.
3. The final volume should be 10.0 mL and the final concentration for each target is listed.
4. Aliquot into labeled robovials
5. Name the standard 1613B-NS#3-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.4.3 Wellington Native Mix Catalogue# EPA-1613STOCK, MEOL# 5002872

Compound	Concentration (ng/mL)
2,3,7,8-TCDD	400
2,3,7,8-TCDF	400
1,2,3,7,8-PCDD	2000
1,2,3,7,8-PCDF	2000
2,3,4,7,8-PCDD	2000
1,2,3,4,7,8-HxCDD	2000
1,2,3,6,7,8-HxCDD	2000
1,2,3,7,8,9-HxCDD	2000
1,2,3,4,7,8-HxCDF	2000
1,2,3,6,7,8-HxCDF	2000
1,2,3,7,8,9-HxCDF	2000
2,3,4,6,7,8-HxCDF	2000
1,2,3,4,6,7,8-HpCDD	2000
1,2,3,4,6,7,8-HpCDF	2000
1,2,3,4,7,8,9-HpCDF	2000
OCDD	4000
OCDF	4000

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7.2.44 Native Stock Standard Solution Preparation for Low Level Calibration Standards (1613B-NS$1)

Standard	Concentration (ng/mL)	Amount Added (µl)	Final Concentration (ng/mL)
EPA-1613STOCK	400-4000	10.0	4-40

1. Using a syringe, add 990 µL of nonane to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard to the nonane.
3. The final volume should be 1.0 mL and the final concentration for each target is listed.
4. Name the standard 1613B-NS$1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.4.5 Natives for CS#3+DB5ms_PERF Standard

The CS#3+DB5ms_PERF standard contains all 2,3,7,8-substituted PCDD and PCDF highlighted in section 7.2.4.3. It also contains all first and last eluting congeners for each level of chlorination as well as congeners used for demonstration of chromatography performance. These are not used quantitatively. WD Standards are for windowing, GC are for chromatography demonstration.

7.2.4.6 Individual Native Standards in CS#3+DB5ms_PERF Standard

Native Standard Compound	MEOL No.	Supplier	Cat. No.
1,3,6,8-TCDD (WD)	5005102	CIL/Cerilliant	ED-2518
1,2,8,9-TCDD (WD)	5005074	Wellington Laboratories	DD-1289-S
1,3,6,8-TCDF (WD)	5005111	CIL/Cerilliant	EF-944
1,2,8,9-TCDF (WD)	5005109	CIL/Cerilliant	EF-939
1,2,4,6,8/1,2,4,7,9-PeCDD (WD)	5005106	CIL/Cerilliant	ED-927
1,2,3,8,9-PeCDD (WD)	5005105	CIL/Cerilliant	ED-924
1,3,4,6,8-PeCDF (WD)	5005110	CIL/Cerilliant	EF-942-50
1,2,3,8,9-PeCDF (WD)	5005076	Wellington Laboratories	DF-12389-S
1,2,4,6,7,9/1,2,4,6,8,9-HxCDD (WD)	5005073	Wellington Laboratories	DD-124679-S
1,2,3,4,6,8-HxCDF (WD)	5005075	Wellington Laboratories	DF-123468-S
1,2,3,4,6,7,9-HpCDD (WD)	5005108	CIL/Cerilliant	ED-976
1,2,3,4-TCDD (GC)	5005104	CIL/Cerilliant	ED-912
1,2,3,7/1,2,3,8-TCDD (GC)	5005103	CIL/Cerilliant	ED-905
1,2,3,9-TCDD (GC)	5005107	CIL/Cerilliant	ED-948
1,2,3,9-TCDF (GC)	5004219	CIL/Cerilliant	EF-4030
2.3.4.7-TCDF (GC)	5004086	Wellington Laboratories	DF-2347-S
2.3.4.8-TCDF (GC)	5004088	Wellington Laboratories	DF-2348-S

7.2.4.7 Native Stock Standard Solution Preparation for CS#3+DB5ms_PERF Standard (1613-NS$2)

Standard	Concentration (ng/mL)	Amount Added	Final Concentration (ng/mL)

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		(µl)	
ED-2518	50000	7.5	250
DD-1289-S	50000	7.5	250
EF-944	50000	7.5	250
EF-939	50000	7.5	250
ED-927	5000	375.0	1250
ED-924	5000	375.0	1250
EF-942-50	50000	37.5	1250
DF-12389-S	50000	37.5	1250
DD-124679-S	50000	37.5	1250
DF-123468-S	50000	37.5	1250
ED-976	50000	37.5	1250
ED-912	50000	7.5	250
ED-905	50000	7.5	250
ED-948	50000	7.5	250
EF-4030	50000	7.5	250
DF-2347-S	50000	7.5	250
DF-2348-S	50000	7.5	250

1. Using a syringe, add 487.5 µL of nonane to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard to the nonane.
3. The final volume should be 1.5 mL and the final concentration for each target is listed.
4. Name the standard 1613-NS$2-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.4.8 Individual Native Standards in 1613B-CS#0 Standard

Native Standard Compound	MEOL No.	Supplier	Cat. No.
2,3,7,8-TCDD	5005692	Wellington Laboratories	DD-2378-S
2,3,7,8-TCDF	5006556	Wellington Laboratories	DF-2378-S

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7.2.4.9 Native Stock Standard Solution Preparation for 1613B-LOD Standard (1613-NS$3)

Standard	Concentration (ng/mL)	Amount Added (µl)	Final Concentration (ng/mL)
DD-2378-S	50000	3.0	25
DF-2378-S	50000	3.0	25

1. Using a syringe, add 5994 µL of nonane to a c-tube.
2. Using syringes of appropriate size, add the amount shown of each standard to the nonane.
3. The final volume should be 6.0 mL and the final concentration for each target is listed.
4. Aliquot into labeled robovials.
5. Name the standard 1613B-NS$3-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.4.10 Native Stock Standard Solution Preparation for DoD LOD/LOQ Verification (1613B-LOD_LOD#1)

Standard	Concentration (ng/mL)	Amount Added (µl)	Final Concentration (ng/mL)
1613B-NS#3-xxx	10-100	60.0	0.5-5.0

1. Using a syringe, add 1140 µL of nonane to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard to the nonane.
3. The final volume should be 1.2 mL and the final concentration for each target is listed.
4. Name the standard 1613B-LOD_LOQ#1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.4.11 Native Stock Standard Solution Preparation for Quarterly Limit of Quantitation Verification (1613B-MDL#3)

Standard	Concentration (ng/mL)	Amount Added (µl)	Final Concentration (ng/mL)
1613B-NS#3-xxx	10-100	60.0	0.5-5.0
1613-NS$3-xxx	25	24	0.4

1. Using a syringe, add 1461 µL of nonane to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard to the nonane.
3. The final volume should be 1.5 mL and the final concentration for each target is listed.

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4. Name the standard 1613B-MDL#3-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.5 Calibration Standards Preparation

7.2.5.1 CS#0 Calibration Standard (1613B-CS#0)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	1613-NS$1	25	0.1-1.0
Extraction	DTOXM-ES$2	100	100-200
Injection	1613B-IS$2	50	100
Cleanup	1613-CL$2	1.0	0.1

1. Using a syringe, add 824 µl of nonane to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard source to the nonane.
3. The final volume should be 1.0 mL and the final concentrations are listed.
4. Name the standard 1613B-CS#0-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.5.2 CS#1 Calibration Standard (1613B-CS#1)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	1613-NS$1	125	0.5-5.0
Extraction	DTOXM-ES$2	100	100-200
Injection	1613B-IS$2	50	100
Cleanup	1613-CL$2	5.0	0.5

1. Using a syringe, add 720 µl of nonane to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard source to the nonane.
3. The final volume should be 1.0 mL and the final concentrations are listed.
4. Name the standard 1613B-CS#1-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

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7.2.5.3 CS#2 Calibration Standard (1613B-CS#2)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	1613-NS$1	500	2.0-20
Extraction	DTOXM-ES$2	100	100-200
Injection	1613B-IS$2	50	100
Cleanup	1613-CL$2	5.0	2.0

1. Using a syringe, add 330 µl of nonane to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard source to the nonane.
3. The final volume should be 1.0 mL and the final concentrations are listed.
4. Name the standard 1613B-CS#2-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.5.4 CS#4 Calibration Standard (1613B-CS#4)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	EPA-1613STOCK	100	40-400
Extraction	DTOXM-ES$2	100	100-200
Injection	1613B-IS$2	50	100
Cleanup	1613-CL$1	8.0	40

1. Using a syringe, add 742 µl of nonane to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard source to the nonane.
3. The final volume should be 1.0 mL and the final concentrations are listed.
4. Name the standard 1613B-CS#4-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.5.5 CS#5 Calibration Standard (1613B-CS#5)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	EPA-1613STOCK	500	200-2000
Extraction	DTOXM-ES$2	100	100-200
Injection	1613B-IS$2	50	100
Cleanup	1613-CL$1	40	200

1. Using a syringe, add 310 µl of nonane to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard source to the nonane.
3. The final volume should be 1.0 mL and the final concentrations are listed.
4. Name the standard 1613B-CS#5-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.5.6 CS#3/Window/GC Resolution Calibration Standard (CS3+DB5ms_PERF)

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Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	EPA-1613STOCK	37.5	10-100
	1613-NS$2	60	10-50
Extraction	DTOXM-ES$2	150	100-200
Injection	1613B-IS$2	75	100
Cleanup	1613-CL$1	3.0	10

1. Using a syringe, add 1174.5 µl of nonane to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard source to the nonane.
3. The final volume should be 1.5 mL and the final concentrations are listed.
4. Name the standard CS3+DB5ms_PERF-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.5.7 Limit of Detection Calibration Standard (1613B-LOD)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	1613-NS$1	12.5	0.05-0.50
	1613-NS$3	8.0	0.20
Extraction	DTOXM-ES$2	100	100-200
Injection	1613B-IS$2	50	100
Cleanup	1613-CL$1	2.5	0.25

1. Using a syringe, add 827 µl of nonane to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard source to the nonane.
3. The final volume should be 1.0 mL and the final concentrations are listed.
4. Name the standard 1613B-LOD-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

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7.2.6 Reference Standards (CVS)

There are 2 different reference standards, one for all 2,3,7,8-substituted dibenzo-p-dioxins (PCDD) and 2,3,7,8-substituted dibenzofurans (PCDF). The second is for 2,3,7,8-TCDD only. Expiry dates are stated on the supplier certificate of analysis.

7.2.6.1 Reference Standard for all PCDD and PCDF (1613B-RS#1)

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	EDF-7999-10X	25	10-100
Extraction	DTOXM-ES$2	100	100-200
Injection	1613B-IS$2	50	100
Cleanup	1613-CL$2	100	10

1. Using a syringe, add 725 µl of nonane to a labeled robovial.
2. Using syringes of appropriate size, add the amount shown of each standard source to the nonane.
3. The final volume should be 1.0 mL and the final concentrations are listed.
4. Name the standard CS3+DB5ms_PERF-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

7.2.6.2 Reference Standard for 2,3,7,8-TCDD

The standard used for the reference standard is SRM-1614 purchased from National Institute of Standards and Technology (MEOL No. 5005992) containing 2,3,7,8-TCDD only. The concentration varies per batch and is accounted for when quantifying the data. A full vial of SRM-1614 is used each time the standard is made.

Type of Standard	Source	Amount Added (µl)	Final Concentration (ng/mL)
Native	NIST SRM-1614	1000	varies
Extraction	DTOXM-ES$2	117.5	100-200
Injection	1613B-IS$2	59	100

1. Using syringes of appropriate size, add the amount shown of each standard source a labeled robovial.
2. The final volume should be 1177 µL and the final concentrations are listed.
3. Name the standard 1613B-RS#2-xxx, where xxx represents a sequential lot number, and refers to a specific preparation.

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8.0 QUALITY CONTROL

8.1 Method QC

Maximum (Sample Preparation) Batch Size =		20 client samples
Method QC	**Min. Frequency**	**Data Quality Objective (units)**
Method Blank (MB)[1]	1 per batch	< instrument response of lowest calibration standard (CS1 or CS0 depending on project)
Laboratory Control Sample (LCS)[2] [aka Clean Matrix Spike or On-going Precision and Recovery (OPR)]	1 per batch	See Table 1 Non-2,3,7,8-substitued PCDD/F are not quantified as individual targets by this method and are not included in the Native Standard spiking solution.
Reference Material (RM)	Upon client request	As per supplier
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)[3]	Upon Client Request	Apply the Table 1 LCS limits where the sample concentration is <25% of the spiking level; otherwise no criterion
Extraction Standards (SURR)	In all samples, including QC	See Table 1.
Clean-up Standards	In all samples, including QC	See Table 1.
Sampling Standards	In all samples, including QC	See Table 1.
Sample Duplicate (DUP)	All matrices expect water: 1 per batch, or as specified in contract or client request	< 50% RPD for targets within quantifiable range of the method
Sample Duplicate (DUP) – Quebec Waters	If 1-10 samples in batch, no duplicate required; 11-20 samples in batch, LCS duplicate required	< 50% RPD for targets within quantifiable range of the method
Procedural Blank (PB)	1 per batch*	Visual Assessment of Contamination
Limit of Quantitation verification	4 times annually, 2 samples each quarter	All targets must meet requirements in section 10.1

*If all samples from a batch require further cleanup, the QC samples must be included. If only selected samples require further cleanup, a procedural blank must be created which will receive the prescribed cleanup to ensure no contamination has been acquired through the additional steps.

8.2 Instrument QC

Maximum Batch Size =	Every 12 hours (every shift)

1 For DoD sample analysis, MB criteria is <1/2 x LOQ or 1/10th of what is in the samples or 1/10th of the regulatory limit whichever is largest.
2 PCDD/F LCS control limits for DoD analysis are listed in Table 7a/b.
3 MS/MSD is to be evaluated relative to the acceptance criteria listed in Table 7a/b, for DoD analysis.

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Instrument QC	Minimum Frequency	Control Limit
Initial Calibration Curve (IC)[4] [CS1-CS5 or CS0-CS5]	As required to maintain a valid initial calibration as demonstrated by required CCV injections	See Table 3
Calibration Verification Standard (CVS) (Section 7.2.8)	Run together with every initial calibration	See Table 3
Continuing Calibration Verification (CCV)[5] (CS3)	Every 12 hours	See Table 3
Post run calibration	End of run for 8290A runs	See Table 3
Instrument Blank	Every analytical batch. Run before Method Blank.	Visually assessed to be free of carry-over contamination from previous injection of standard or Laboratory Control Sample

Corrective Actions for Recovery Limit Failures:

a) Extraction Standards:

Evaluate source of the recovery failure. Actions may include, re-injection, clean-up/analysis of an archived portion of the raw extract, re-extraction or flagging of the data. Unless there is clear evidence for a matrix bias, every reasonable effort should be taken to resolve a failed extraction recovery issue. When simpler corrective actions such as re-injection do not resolve the issue, re-analysis or, where there is insufficient sample available, analysis of an archived portion of the raw extract should be the appropriate course. If the recovery bias is repeatable, then this constitutes clear evidence for a matrix bias, no further action is required and the data can be flagged appropriately and reported.

b) Clean-up Standards:

Clean-up recoveries represent a tool to help assess whether a difficulty originates from the extraction of the sample, or the clean-up of the extract. Other than evaluation of the data set, failures in clean-up recoveries do not force a corrective action.

c) Injection Standards:

A low injection standard response indicates a missed injection. A repeat injection is the most common course of action. A high response indicates extract concentration. A review of the extract final volume is required. Otherwise, a low or high recovery indicates a potential compromise to the handling of the sample extract or interference. Unless there is a clear explanation for the failure, then re-extraction/ re-analysis or analysis of an archived portion of the extract is necessary.

d) Sampling Standards:

4 If any calibration criteria are not met, system must be evaluated and corrective action must be taken; the entire initial calibration procedure must be repeated before sample analysis begins.

5 If all acceptance criteria are met for the Continuing Calibration Verification Standard, then the initial calibration can be used for the analysis of additional samples. In the event of a CCV failure during DoD sample analysis, proceed with the following corrective action:

- Immediately analyze 2 additional consecutive CCVs, both must meet acceptance criteria in order for samples to be reported without reanalysis.
- If these do not pass, recalibration must occur and all samples since the last acceptable CCV must be reanalyzed.

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For stack and ambient sampling, the entire sample is consumed in the extraction. Therefore re-extraction/re-analysis is not normally an option. Re-injection and/or analysis of an archived portion of the extract are the appropriate corrective actions.

9.0 TEST PROCEDURE

9.1 Sample Preparation

See BU-TM-1110 Overall HR Prep.

9.2 Dilution Procedure

1) The target analyte concentrations in the sample extract must be within the calibrated range of the instrument.

    a) See BU-SP-0263 for Dilution policy and general procedures.

2) If a sample is diluted 100x or greater, then spike with ES and IS at the regular amount. The results will no longer be recovery corrected; therefore, a comment will need to be made on the report.

3) If the sample is in a microvial and the final volume will be 500 µL or less, then a measured amount of solvent (toluene or distilled nonane) is added directly to the uncapped microvial. Re-cap and vortex the sample

4) If a greater dilution is required or the sample is for more than one analyses, then vortex the sample. Remove the cap. Using a clean syringe, put an aliquot into a new, labeled microvial. Cap and vortex the new microvial.

NOTE: If a sample is suspected of being high level prior to analysis, an instrument blank is run immediately after it or the high-level sample can be put at the end of the run.

9.3 Additional Cleanups & Reworks (Procedural Blank Generation)

If a sample requires additional work in the lab, a procedural blank is generated at the instrument, which accompanies the samples and the rework document as it goes back out into the prep lab. The procedure below outlines how to generate a procedural blank.

1) Refer to the sample batch sheet to identify the injection and extraction standard types and amounts used in generating the batch.
2) Retrieve the corresponding injection and extraction standards from the standards fridge.
3) A new vial is obtained, and labelled as PB-xxx (where xxx is a sequential, unique numerical identifier)
4) Using proper syringe techniques, spike the appropriate amount of each standard into the procedural blank vial.

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5) Submit the procedural blank vial, alongside the sample(s) that require additional work, to the prep lab.

9.4 Instrumentation

1) System Initialization & Maintenance

Refer to BU-WI-3007 GCMS Operation.

2) Instrument Operating Parameters

The operation of the HRMS instrument is described in Work Instruction BU-WI-3007 GCMS Operation. Specific MS conditions and parameters may vary slightly for each instrument, and can be found in BU-WI-3206 PCDD_F DB5MS HRMS-AutoSpec M&S Series, BU-WI-3200 PCDD_F DB5MS HRMS-AutoSpec Premier, BU-WI-3207 PCDD_F DB5MS HRMS-DFS, and BU-WI-3208 PCDD_F DB225 HRMS-AutoSpec M&S Series.

The exact masses monitored, and suggested acquisition descriptor parameters are shown in Table 5. Note that depending on the instrument used, alternate or additional PFK masses may be used (for example, on the Thermo DFS instrument, each analytical segment requires three PFK masses – one each for lock and calibration masses, and another to be monitored by the data processing software for lock mass and response drift. Also, depending on the GC column and conditions, it may not be possible to separate the last eluting tetrachlorodibenzofuran from the first eluting pentachlorodibenzofuran. In that case tetrafuran and pentafuran masses may be used in more than one analytical segment to cover the entire chromatographic range in which that level of chlorination elutes. The exact MS acquisition methods used by each instrument are referenced in BU-WI-3206 PCDD_F DB5MS HRMS-AutoSpec M&S Series, BU-WI-3200 PCDD_F DB5MS HRMS-AutoSpec Premier, BU-WI-3207 PCDD_F DB5MS HRMS-DFS, and BU-WI-3208 PCDD_F DB225 HRMS-AutoSpec M&S Series.

Lock mass drift correction must be used within every descriptor to adjust for small changes in mass peak location.

The response of the mass calibration standard (typically perfluorokerosene, PFK) is also monitored to determine the presence of interferences. Deviations to the average response must be less than or equal 20%. Deviations outside of this may indicate the presence of chemical interference(s) which may impact data quality. Where lock mass deviations exceed the 20% value within the target window, an assessment on the impact of data quality is required. Whenever the deviation co-elutes with and compromises quantitation of any calibrated target analyte, then one or more of the following options in remedial actions must be implemented: (a) re-injection (b) dilution/re-injection (c) re-clean of the extract followed by re-analysis (c) additional cleanups of the extract followed by re-analysis or (d) re-extraction/re-analysis. If re-extraction is in order, re-extraction of a smaller sample size should be considered. Ultimately, any remedial action needs to provide data within lock

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mass control and that meets the method and project data quality objectives.

GC Conditions:

GC Temperature profiles and conditions must be designed so that the performance meets all method RT, RRT and chromatographic resolution criteria as required by the applicable reference method (see Section 9.3.4 and 10.1). GC operating conditions are optimized on each HRGC/HRMS system. Once optimized, the same conditions must be used for analysis of all calibration standards, Quality Control Samples, and samples. The specific GC operating conditions for each system can be found in BU-WI-3206 PCDD_F DB5MS HRMS-AutoSpec M&S Series, BU-WI-3200 PCDD_F DB5MS HRMS-AutoSpec Premier, BU-WI-3207 PCDD_F DB5MS HRMS-DFS, and BU-WI-3208 PCDD_F DB225 HRMS-AutoSpec M&S Series.

3) Run Processing

a) Samples in autosampler

1. Place samples extracts and standards in microvials within the autosampler on the GC/MS instrument.
2. The autosampler injects 1 µL into the instrument for each instrumental run. Volumes up to 2 µL are permitted in order to meet sensitivity requirements. The injection volume and run conditions must be the same for all injections within each 12-hour run sequence. The specific GC and MS operating conditions for each system can be found in BU-WI-3206 PCDD_F DB5MS HRMS-AutoSpec M&S Series, BU-WI-3200 PCDD_F DB5MS HRMS-AutoSpec Premier, BU-WI-3207 PCDD_F DB5MS HRMS-DFS, and BU-WI-3208 PCDD_F DB225 HRMS-AutoSpec M&S Series.

b) Run sequence set up

A typical continuous run sequence is listed below.

1A) Window defining mix and GC Performance Mix[1]
2A) CS3[1]
3A) QC samples spiked at the CS3 level or higher
4A) One or more system blanks
5A) Method blank(s)
6A) Samples
1B) Window defining mix and GC Performance Mix[1]
2B) CS3[1]
3B) QC samples spiked at the CS3 level or higher
4B) One or more system blanks
5B) Method blanks(s)
6B) Samples
1C)…..

1. Optionally, solutions for Items 1A and 2A or 1B and 2B can be combined for a single injection.

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Additional Considerations

- The 1A to 5A portion of the continuous sequence represents the first 12-hour run sequence. The timing of the last injection of each 12-hour sequence must not be greater than 12 hours following the injection of the calibration verification CS3. Before and after each 12-hour sequence a mass resolution check must demonstrate 1 in 10,000 resolution. Instrument conditions must remain the same during each 12-hour sequence and no other injections can take place between the last injection of the 12-hour sequence and the ending mass resolution check.

- For 8290A and many contracts, a CS3 verification must be run and be in control at the end of the 12-hour run sequence (i.e. a post run verification). In this case, that post-run injection must be within the 12-hour sequence. Provided instrument calibration verification is achieved, the post-run calibration can be the acceptable CS3 start of the following 12-hour sequence.

- When a multi-point calibration initiates a 12-hour run sequence, then the injection of the first standard represents the start of the 12-hour sequence.

4) System Performance

These are aspects of instrument control that are mandatory before analysis of samples can proceed. If there is any failure on any one or more of these criteria, then instrument control must be regained by re-injection or instrument maintenance before samples can be analyzed.

a) **Window Performance Mix:** There must be confirmation that instrument conditions are set to reliably capture and quantify all PCDD and PCDF of interest. When the targets are for all PCDD/F (tetra- through octa-) a window performance mixture, set to define the first and last eluting member of each homologue group, must be run at the beginning of each 12-hour run sequence. This run must demonstrate that the descriptor windows are set to capture all targeted analytes. If the targeted analyte listing is limited (e.g. analysis of 2,3,7,8-TCDF only), then the requirement is to demonstrate capture of the limited listing only.

The Window Performance Mix may be analyzed in a separate analytical run or in combination with the CS3 and/or the appropriate GC Performance mix.

b) **GC Performance:** For a DB5, DB5MS column or equivalent, a demonstration of 2,3,7,8-TCDD specificity is required. The GC peak resolution between 2,3,7,8-TCDD and its closest eluting isomers must be less than or equal to 25% valley as shown in Figure 1 [x/y must be less than or equal to 0.25].

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This must be demonstrated at the beginning of each 12-hour run sequence for which this column is being used.

For a DB225 column or equivalent, a demonstration of 2,3,7,8-TCDF specificity is required. The GC peak resolution between 2,3,7,8-TCDF and its closest eluting isomers must be less than or equal to 25% valley as shown in Figure 2 [x/y must be less than or equal to 0.25]. This must be demonstrated at the beginning of each 12-hour run sequence for which this column is being used.

For a DB5MS column or equivalent, a demonstration of 2,3,7,8-TCDF specificity is required. The GC peak resolution between 2,3,7,8-TCDF and its closest eluting isomers must be less than or equal to 80% valley as shown in Figure 2 [x/y must be less than or equal to 0.80]. This must be demonstrated at the beginning of each 12-hour run sequence for which this column is being used.

The GC Performance mix maybe analyzed as a separate analytical run or in combination with the CS3 and/or the appropriate Window Performance mix.

FIGURE 1: DB5 GC Performance Check

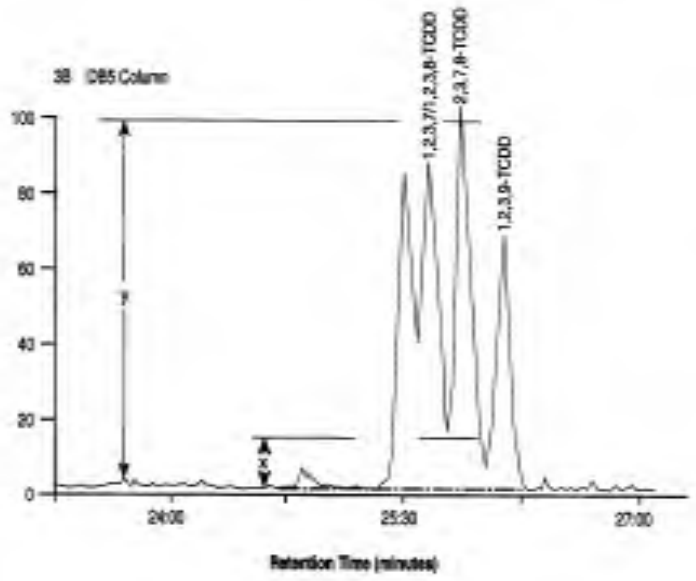

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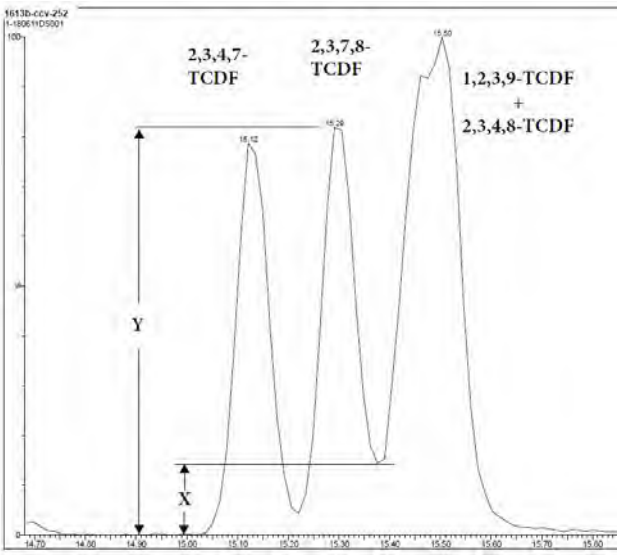
FIGURE 2: DB225 GC Performance Check



c) **Mass Resolution:** At the beginning of and just following the end of each 12-hour run sequence, the instrument must be checked to demonstrate a resolution of 10,000 at 10% valley for each quantification window. The 10% valley definition for resolution requires mass peak width to be determined at 5% of peak height. If the resolution check fails at the end of the sequence, then the data from the sequence should be reviewed carefully for a) chemical interferences and b) deterioration in instrument sensitivity. If upon review there remains concern for real compromise to data quality, then re-injection of the impacted samples under full instrument control is required. If during the evaluation, the data assessment concludes that there has been no significant compromise to the data quality from the drift in resolution, then the data may be reported while including a comment on the report identifying the control failure and the absence of impact on data quality.

d) **Relative Retention Times (RRT):** For method 1613B, suggested relative retention times (RRT) of the compounds in the daily CS3 calibration verification are presented in Table 4 for a DB5 GC column. The RT specifications in section 10.1.4 must be met.

e) **Absolute Retention Times:** For method 1613B, for all calibrations, QC samples and field samples, the absolute retention time for 1,2,3,4-TCDD-$^{13}C_{12}$ must be >25.0 min on a DB5 or DB5MS column and the absolute retention time for 2,3,7,8-TCDF-$^{13}C_{12}$ must be >15.0 min on a DB225 column.

f) **Retention Time Match between Initial and Verification Standards:** The RT in the daily CS3 verification standards must be within 15 seconds of the CS3 in the initial calibration run.

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g) **Maximum Scan Time:** The maximum time between scans within a descriptor is 1 second.

10.0 DATA CALCULATION AND REPORTING

10.1 Qualitative Analysis

The following criteria must be met for a positively identified response to labelled or unlabelled analytes on all field samples, QC samples and calibration runs. Failures on any one of these items for a calibration run means that the calibration run is out of control for that analyte. For calibration runs, instrument control must be regained by re-injection or instrument maintenance before samples can be analyzed. Sample failures indicate the presence of interferences or responses inadequate for qualitative or quantitative determination.

1) Ion Ratio Criteria

For all compounds except for 2,3,7,8-TCDD-$^{37}Cl_4$, a pair of ions with a specific ^{37}Cl :^{35}Cl isotopic ratio are being monitored. To have a confirmed positive response to a native or labelled PCDD/F, that ratio must be within 15% of the theoretical value or within 10% of the observed values on the most recent CS3.

2) Signal to Noise Criteria (Calibration Runs Only)

The signal to noise ratio for each quantification ion for labelled and non-labelled analytes must be greater than or equal to 10:1 for the initial calibration CS1 and for each calibration verification CS3.

3) Matched RT on Peak Maxima

The retention time (RT) of the peak maxima for each pair of quantification ions must be no more than 2 seconds (i.e. 2 scans) difference.

4) Retention Time (RT) Match between Native to Labelled Analyte

The retention time (RT) of the peak maxima of all 2,3,7,8- substituted native analytes must be within -1 to +3 seconds of the RT of corresponding $^{13}C_{12}$-labelled isomer of that injection run. For those native analytes without a corresponding labelled isomer, the relative retention time (RRT) must be within 0.005 of the relative retention time observed in the daily CS3 run.

5) Polychlorinated Diphenyl Ether (PCDPE) Interferences [Samples only]

This issue is limited to sample extract native PCDF responses that meet all other applicable criteria within section 10.1. If polychlorinated diphenyl ethers (PCDPE) have not been completely removed during extract clean-up, then major mass fragment ions (i.e. m/z M^+-70) of PCDPE can provide positive interferences into the corresponding PCDF channels. For this reason, appropriate PCDPE parent ions are monitored during the

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instrumental analysis. Significant PCDPE interference is identified when there is a PCDPE parent ion peak 10% or more of the response of a peak at the same RT (i.e. within 2 seconds) in the corresponding PCDF channel. When there is a significant interference observed, then the peak in the PCDF channel is not confirmed to be PCDF.

6) Peaks within Pre-Defined Windows

For any peak to be identified as a positive PCDD/F response, that peak must be within the retention time windows defined by the daily analysis of Window Performance Mixture (see Section 9.2.5.1).

10.2 Isotope Dilution and Internal Standard Calibration

Calculation of Concentrations

The relative response factor of each target relative to the standard against which it is to be calculated is determined using the area responses of both quantification ions via equation 10.1.

In cases where a native target is calculated against an exact labelled analogue, the quantification will be considered to be by isotope dilution. In other cases, the quantification will be considered to be by internal standard.

For the case of the homologue group totals, concentrations are calculated using Equation 10.1 where the target areas represent the sums of the areas for each peak in the retention time window, where the peak has met the peak identification criteria. Others are excluded. For example, a peak where the ratio between the areas of the two quantification ions does not meet the method control limit will be excluded from the calculation. The areas of the labelled compound are the average of the areas for the extraction standards within the homologue group.

$$RRF = \frac{(A1_t + A2_t)\ C_s}{(A1_s + A2_s)\ C_t} \qquad \textbf{Equ. 10.1}$$

Where,
$A1_t + A2_t$ = The areas of the two quantification ions for the target analyte
$A1_s + A2_s$ = The areas of the two quantification ions for the labelled compound against which the target analyte will be calculated.
C_t = The concentration in the calibration standard of the target analyte.
C_s = The concentration in the calibration standard of the labelled compound against which the target will be calculated.

For all analytes to be quantified and from the initial calibration series of standard injections, a table of RRFs is prepared. The relative standard deviation (%RSD, or the coefficient of variance) is checked to confirm that the appropriate method criteria has

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been met as listed in Table 3. The average of the five or six levels of standard for each analyte, RRF_{av} is applied for quantification of samples according to Equations 10.2 and 10.3 below.

$$\text{Amount in sample (pg)} = \frac{(A1_n + A2_n)\ Q_l}{(A1_l + A2_l)\ (RRF_{av})} \quad \textbf{Equ. 10.2}$$

$$\text{Concentration in sample (pg/g or pg/l)} = \frac{(A1_n + A2_n)\ Q_l}{(A1_l + A2_l)\ (RRF_{av})\ (W_s)} \quad \textbf{Equ. 10.3}$$

Where,
Q_l = The amount (pg) of labelled compound added to the sample
W_s = The weight (g) or volume (l) of sample

Extraction, Clean-up, and Sampling Standard Recovery Calculation:

The extraction, clean-up, and sampling standard recoveries are determined by Equation 10.4 below.

% Recovery = (Amount in sample)/(Amount added to sample) X 100 **Equ. 10.4**

Estimated Detection Limit (EDL)

Noise-based EDLs are calculated for each target for each sample, every time the extract is analysed using Equation 10.5 below for all 2,3,7,8-substituted targets. For homologue group totals, EDLs are also calculated using Equation 10.5, using the average of all of the labelled extraction standards in the homologue group. Data are reported to the noise-based Estimated Detection Limit (EDL). As this is a measured value, it does not require further adjustment for final volume, sample size, split ratio, or dilution factor

$$EDL = \frac{2.5 \times H_x \times Q_{es}}{H_{es} \times W \times RFF_{av}} \quad \textbf{Equ. 10.5}$$

Where,
EDL = estimated detection limit for homologous 2,3,7,8-Substituted PCDD/Fs
H_x = sum of the height of the noise level for each quantification ions for the unlabelled PCDD/Fs.
H_{es} = Sum of the heights of responses of both quantification ions for the labelled extraction standard.
W = weight of volume of sample

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RRF_{av} = average relative response factor
Q_{es} = Amount of extraction standard added

Toxic Equivalency (TEQ)

BU-WI-3025 TEQ Calculations details the calculation of toxic equivalency (TEQ) values

10.3 Data Calculation and Report Generation

BU-WI-3225 Reporting HRMS Results details the process for quantifying results, preparing reports and uploading results to LIMS

BU-WI-3019 Manual Integration details the policy and procedures regarding manual peak integration

10.4 Data Validation

Peer review and sign off is a requirement, refer to NA-SP-0175 Data Validation and BU-SP-0175 Report Template and Data Validation for details.

10.4.1 Method Blank Failure

If the blank level exceeds the DQO (see Section 8.0) for any analyte, then the corrective action is to re-analyze any sample within the batch that has a positively determined analyte with up to 10-fold higher response of the blank level and, for regulatory compliance, if the biased analyte in the sample is within a factor of 10 of the regulatory compliance limit.

10.4.2 LCS Failure

When the LCS recoveries are outside the recommended ranges, an evaluation of the impact on the data quality is required. Failure(s) may indicate compromise to one or more of the standards or to the extraction/clean-up process. If the evaluation shows significant compromise to the reported data quality for one or more samples in the batch, then the affected samples should be re-injected and/or repeated for PCDD/F analysis from archived extract and/or from another portion of the original sample. If repeat analysis is desired and additional sample is not available for repeat, then a final evaluation of the pooled results is required to evaluate appropriate data flagging or non-reporting of the results.

10.4.3 MS and MSD Failure

Corrective actions from failure of this criterion include, at the least, re-evaluation of the data set with respect to calculation errors or analytical compromise. Re-injection or re-analysis of the sample for MS/MSD may be considered. Re-extraction and re-analysis of the entire batch may be considered.

10.4.4 DUP Failure

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Corrective actions from failure of this criterion include, at the least, examination of the sample for evidence of inhomogeneity or evaluation of the data set with respect to calculation errors and analytical compromise. For solids, sample inhomogeneity is a common source of the problem. Documented evidence for inhomogeneity may resolve a criterion failure. Re-injection or re-analysis of the sample and duplicate, or the entire batch may be considered.

10.4.5 Annual Verification of MDL_b and MDL_s

At least every thirteen months, the MDLs for the low-level spiked samples (MDL_s) and method blanks (MDL_b) are recalculated from the Limit of Quantification verification samples and method blanks run in the last 24 months. The laboratory has the option to use only the last 6 months of method blank data or the fifty most recent method blanks, whichever criteria yields the greatest number of method blanks.

10.5 Data Reporting

10.5.1 Modifying Limits of Reporting

Method Blank subtraction is not performed for this method. Where Method Blank contamination above the method control limit occurs, and re-extraction is not possible or likely to reduce the blank level, a report comment is required detailing the potential impact to data quality. Instrument subtraction is not done for this method.

10.5.2 Units and Significant Figures

Results are reported to three significant figures for the PCDD/Fs found in all blanks and samples. Extraction, Clean-up, and Sampling Standards are reported as a percent recovery to the nearest integer.

Aqueous samples—Report results in pg/L (parts-per-quadrillion).

Samples containing greater than 1% solids (soils, sediments, filter cake, compost)—Report results in pg/g (parts-per-trillion) based on the dry weight of the sample. Report the percent moisture so that the result may be converted to aqueous units.

Tissues—Report results in pg/g of wet tissue, not on the basis of the lipid content of the tissue. Report the percent lipid content, so that the data user can calculate the concentration on a lipid basis if desired.

Air/Stack/Swabs—Report results in pg.

10.5.3 Data Qualification

The following Qualifiers are routinely used:

M	Indicates that a peak has been manually integrated
U	Indicates that this compound was not detected above the EDL
E	indicates that a target analyte was detected above the calibrated range

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J	indicates that a target analyte was detected below the calibrated range but above the EDL
B	Indicates that this compound was detected in the method blank at greater than 10% of the sample concentration
R	Indicates that the ion abundance ratio for this compound did not meet the acceptance criterion.

Additional Qualifiers may be applied to results are they are uploaded to LIMS.

The qualifiers U, J, E, B, and R are applied automatically by the Excel report template. The qualifier M is applied by the template to all data identified as being derived from a manually-integrated peak.

The qualifier E is used only in cases where dilution and re-analysis is not possible.

The level at which the qualifier J is applied is calculated from the concentration of the lowest level calibration standard, the final volume, sample size, split ratio, and dilution factor.

All DQO failures require qualification and/or comment on the Title Page and/or in the data package Case Narrative.

For all detected blank levels, the associated samples with positive hits below 10x the blank contamination level are suspect. The measured level in the method blank is reported and the affected sample data are flagged with a B.

10.6 Estimated Maximum Possible Concentration

An estimated maximum possible concentration (EMPC) is a worst-case estimate of the concentration of PCDD/F analyte under a chemical interference. It is calculated assuming theoretical isotopic ratio and applying equations 10.2 and 10.3.

11.0 REFERENCES

11.1 Primary Reference

1. EPA Method 1613B, Analysis of Dioxins and Furans in Wastewater, Revision B, October, 1994.

11.2 Secondary References

The following reference methods are also supported by this Test Method for all applicable matrices by employing the appropriate control limits, standard spiking and calibration, as described, and subject to the deviations listed below.

1. EPA Method 8290A, Polychlorinated Dibenzo-p-dioxins (PCDDs) and Polychlorinated dibensofurans (PCDFs) by high-resolution gas chromatography/high-resolution mass spectrometry (HRGC/HRMS), Revision A, January, 1998. [Also referenced for the treatment of noise-based estimated detection limits (EDLs) and provides

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guidance on noise determination.]

2. EPA Method 23, Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans from Municipal Waste Combustors. [re Stack Analyses]

3. EPA Method 0023A, Sampling Method for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofuran emissions from stationary sources, Revision 1, December, 1996. [Method 0023A is a version of Method 23 used by the EPA OSW and references Method 23. Analytical details are directed to Method 8290A.]

4. EPA TO-9A, Determination of Polychlorinated, Polybrominated and Brominated/Chlorinated Dibenzo-p-Dioxins and Dibenzofurans in ambient air, Edition 2, January, 1999. [re Ambient Analyses]

5. Code of the Federal Registry, Volume 40, Part 60 App. A; Method 23, December, 2016. [used for calculation of MDLs]

6. Talyor 1987: Quality Assurance of Chemical Measurements, John Keenan Taylor, Lewis Publishers Inc., 1987. [LOQ definition]

7. EPA Method 218.6 Determination of Dissolved Hexavalent Chromium in Drinking Water, Groundwater, and Industrial Wastewater Effluents by Ion Chromatography, Revision 3.3, 1994. [laboratory duplicate definition]

8. Eurachem Guide 1998 - IUPAC Compendium of Chemical Technology, 1985. [bias definition]

9. Ontario MOE Laboratory Services Branch, Practices for the Collection and Handling of Drinking Water Samples, Version 2, April 2009

11.3 Instrument Manuals

1. AutoSpec Instrument Manual (HRMS1, 2)
2. G:\Enviro\Library\Equipment diagrams and manuals\Thermo DFS (HRMS4)
3. AutopSpec Premier: Operators Guide. 71500089202. Revision B (HRMS5, 6, 7)

11.4 Software Manuals

1. TargetLynx/MassLynx v4.1 Getting Started Guide
2. Thermo Xcalibur

11.5 Supporting ALS Documents and Forms

1. NA-SP-0001 Definition of Key Terms
2. NA-SP-0100 Initial Method Validation – Chemistry
3. NA-SP-0102 Method Revalidation and MU – Chemistry
4. NA-SP-0104 QC Protocols
5. NA-SP-0175 Data Validation

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6. BU-TM-1110 Overall HR Prep
7. BU-TP-1110 Overall HR Prep for Serum
8. BU-WI-3000 Glassware Cleaning, Proofing and Maintenance
9. BU-WI-3007 GCMS Operation
10. BU-WI-3019 Manual Integration
11. BU-WI-3025 Calculation of TEQs
12. BU-WI-3200 PCDD_F DB5MS HRMS-Autospec Premier
13. BU-WI-3225 Reporting HRMS Results
14. BU-WI-3206 PCDD_F DB5MS HRMS-AutospecM&S Series
15. BU-WI-3207 PCDD_F DB5MS HRMS-DFS
16. BU-WI-3208 PCDD_F DB225 HRMS-Autospec M&S Series
17. BU-SP-0021 Waste Handling and Sample Disposal
18. BU-SP-0250 Control of Reagents and Reference Materials
19. BU-SP-0263 Dilution
20. BU-SP-0175 Report Template Validation & Data Validation
21. BU-FM-1107j PCDD_F-1613B-HRMSTL-2.0
22. BU-FM-1107k PCDD_F-8290A-HRMSTL-1.0
23. BU-FM-1107l PCDD_F-M23-HRMSTL-1.0
24. BU-FM-1110j CFIA_DXPCB-HRMSTL-BU-2.0

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12.0 DEVIATIONS FROM THE PRIMARY REFERENCE

The following changes and clarifications apply:

1) The calibration standards as listed in Table 2 are applied appropriately to all of the reference methods listed above. Such an application of one standard calibration series to all of these methods is within the scope of each and every one of the methods. The calibration standard set CS1 through CS5 is consistent with the standards concentration listing in method 1613B Table 4. The CS0 extends the calibration range below what is required by all of the methods. Table 4 defines the use of each of the labelled standards relative to each of the methods.

a) Method 1613B lists a larger suite of labelled extraction standards than does method 8290A. Additional labelled extraction standards have been added into the 8290A analysis to enhance the method and the data quality. These additions to the method constitute performance-based enhancements and are within the scope of SW846 Method 8290A.

b) The levels presented in the calibration table of method 8290A are recommended values only. Changes to these concentrations, especially to expand the range, are within the scope of the method. Therefore, application of the 1613B calibration standards to method 8290A is compliant with the scope of the method.

c) TO-9A is also a performance-based method. It specifically states that different extraction standards and different concentrations of standards from those listed in TO-9A Table 3 is acceptable (see Section 6.8 of reference method).

d) Although OAQPS reference method 23 is not a performance-based method, application of the 1613B standards has been defined as within the scope of the method. (see Appendix B)

2) Chlorinated Diphenyl Ether interferences: Both methods 1613B and 8290A indicate that any instrumental response showing the presence of a chlorinated diphenyl ether response and that coelutes with a PCDF represents an interference on that analyte (see Sections 18.3 and 11.8.4.4 respectively). This apparent zero tolerance does not take into account that the response in the diphenyl ether channel may be trivial relative to the corresponding PCDF. For this 'Standard Method', we have defined a chlorinated diphenyl ether interference as the presence of a **significant** response within the chlorinated diphenyl ether channel (rather than zero response) and defined significant as a response equal to or greater than 10% of the peak response in the PCDF channel.

3) The method 1613B and method 23 listed requirement for secondary column analysis to resolve 2,3,7,8-TCDF only is seriously flawed since there are other TEQ biases from primary column co-elutions that are, in all likelihood, more impactful. A full understanding of congener co-elutions was fully understood long after the publication of these methods.[1] Furthermore, 2,3,7,8-TCDF can be uniquely resolved from other

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tetrachlorodibenzofuran co-elutions with the use of a DB5MS primary column.

ALS Burlington's default analytical procedure for all PCDD/F methods is to perform a DB5MS single column-analysis. This single-column approach is superior to the 1613B & method 23 dual-column approach, as it uniquely resolves 2,3,7,8-TCDF, while providing a similar TEQ bias potential when compared to the DB5 and DB225 combination (where the DB225 is used to resolve 2,3,7,8-TCDF only). A minimum separation criterion of <80% valley from 2,3,7,8-TCDF from its closest eluting congeners has been included for this single-column analysis.

Secondary column confirmation for all potential co-elutions may be performed, in cases where any and all potential TEQ bias need to be eliminated (i.e. separation of 2378-subsituted co-elutions from co-eluting congener). Column pairs of DB5 and DB225, or DB5MS and SP-2331, are recommended to provide unique resolution for all 2,3,7,8-PCDD/F targets. This comprehensive secondary column approach is consistent in design with Method 8290A where such a secondary analysis is a listed option. This comprehensive secondary column option is available on special request.

2,3,7,8-Substituted Congeners Not Uniquely Resolved from Other Congeners2

DB5	DB5MS	DB-225	SP-2331
1,2,3,7,8,9-HxCDD	2,3,4,7,8-PnCDF	2,3,7,8-TCDD	2,3,7,8-TCDD
2,3,7,8-TCDF	2,3,4,6,7,8-HxCDF	1,2,3,7,8-PnCDD	1,2,3,7,8-PnCDD
2,3,4,7,8-PnCDF	1,2,3,7,8,9-HxCDF	1,2,3,7,8-PnCDF	2,3,7,8-TCDF
1,2,3,4,7,8-HxCDF		2,3,4,6,7,8-HxCDF	1,2,3,7,8-PnCDF
		1,2,3,7,8,9-HxCDF	1,2,3,4,7,8-HxCDF

[1] Vyacheslav N. Fishman, Gregory D. Martin, Lester L. Lamparski; Journal of Chromatography A, 1139 (2007) 285–300

[2] Fishman VN, Martin GD, Wilken M; Dow Chemical Company Internal Publication

4) Although categorically stated in many associated PCDD/F methods, but not all, we maintain that each and every individual clean-up procedure is, by definition, performance-based and optional. There is not an expectation within the industry to follow exactly the descriptions of clean-ups in reference methods. Adaptations which meet or exceed the required performance criteria are therefore acceptable within the scope of each reference method. The reference method descriptions are intended as guidelines or templates available to help the laboratory to define effective in-house clean-up methods. The objective within the laboratory is to provide quality clean extracts to the instrument for analysis. Each individual clean-up is part of the laboratory's capabilities in order to achieve this objective.

5) There are differences within the individual reference methods as to the precise spiking protocols for adding extraction standards and native spikes (for LCS, MS and MSD). To ensure consistency within the laboratory between PCDD/F and related methods, the PCDD/F preparative 'Specific Method' requires solids (including stack

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and ambient sorbents/filters) to be spiked in the soxhlet thimble from a nonane solution and waters are spiked before filtering from an acetone solution. This is consistent with the 8290A approach.

6) Sub-sampling of solids and pre-extraction processing is done in a manner that minimizes potential for cross-contamination. These processes are designed around SW846 protocols rather than 1613B protocols. Solids are sub-sampled directly from the bottle as submitted to the laboratory wherever practical. If the sample is submitted such that homogenization in the bottle is impractical (e.g. the bottle is too full or lumps cannot be broken down), then transferring the sample to a tray or another bottle maybe in order.

7) The concentrations of labelled and native spiking solutions are not consistent with those listed in all the reference methods. These concentrations are prepared at levels convenient and expedient for accurate laboratory processing.

8) With respect to extraction standard recovery limits on non-stack samples analyzed via method 8290A, the limits are based upon the inter-laboratory performance limits defined in method 1613B rather than the relatively arbitrary limits of 35-140% suggested in Section 9.7 of method 8290A.

9) Method 8290A allows for cases where the post-run CCV has failures >35%, that a new Ical be run within 2 hours, implying that the prior data could be reported. Our approach for failures >35% is to rectify the instrument performance issue and re-analyze any negatively affected samples (Section 9.6.2.4)

10) EPA methods 1613B and 8290A state for qualitative determination the signal-to-noise ratio for the GC peak at each m/z must be greater than or equal to 2.5 for each analyte detected. Many Canadian and European regulatory bodies require a signal-to-noise of 3:1. It is our view that it is not specified to be 3.0:1, and that 2.5 rounds up to 3 and meets this requirement.

13.0 REVISION HISTORY

Version #	Date	Summary of Major Changes Made / By
01	11 July 2005	Method creation and definition. Stephen Kennedy/ Ron McLeod
02	24 Jan 2008	Refer to v02
03	17 Sept 2010	Refer to v03
04	14 Nov 2012	Refer to v04
05	15 Nov 2012	Refer to v05
06	15 Feb 2013	Refer to v06
07	29-Aug-2014	Refer to v07
08	9-Feb-2015	Refer to v08
09	25-Feb-2015	Refer to v09
10	19-Mar-2015	Refer to v10
11	10-Nov-2015	Refer to v11
12	1-Feb-2016	Refer to v12
13	2-Feb-2017	Refer to v13

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Version #	Date	Summary of Major Changes Made / By
14	3-Oct-2018	Refer to v14
15	14-May-2020	Refer to v15
16	22-Mar-2021	Updated Section 12.3 to include column and method clarification /RM/CM5

14.0 WASTE MANAGEMENT

Refer to 'BU-SP-0021 Waste Handling and Sample Disposal'. Waste information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

15.0 SAFETY

This task may include CHEMICAL, BIOLOGICAL, OPERATIONAL and/or EQUIPMENT hazards. Staff must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

Refer to Material Safety Data Sheets (MSDS) for detailed information about safe handling guidelines and hazards associated with chemicals used in this method.

HAZARD ASSESSMENT		
Job Task #1:	**Hazards**	**Preventative Measures**
Sample Preparation	Solvent exposure	Wear gloves, safety glasses and lab coat. Work in fume hood. Avoid skin contact with solvents.
Job Task #2:	**Hazards**	**Preventative Measures**
Preparing Reagents and Standards	Although only dilute solutions of standards are used in the laboratory to minimize handling risks, the intrinsic risks in handling PCDD/F standards and extracts must be recognized. The 2,3,7,8-TCDD isomer has been found to be carcinogenic and teratogenic in laboratory animal studies.	Wear appropriate PPE. On the basis available toxicological information, all tetra- to octachlorinated dibenzo(p)dioxins/furans should be handled only by highly trained personnel thoroughly familiar with the handling and cautionary procedures and the associated risks.

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16.0 APPENDICES

Table 1: Data Quality Objective / Acceptance Criteria
Table 2: Calibration Standards
Table 3: Calibration Control Limits
Table 4: Quantitation References and Method 1613B RT References and RRT
Table 5: HRMS Suggested Acquisition Descriptor Parameters
Table 6: LOD/LOQ Levels (DoD PJLA)
Table 7a: PCDD/F LCS Limits for Solid/Tissue/Air Matrices (DoD Analysis Only)
Table 7b: PCDD/F LCS Limits for Water Matrix (DoD Analysis Only)
Table 8: CAS Numbers for Non-2378 Substituted Native Compounds and Labeled Compounds

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Table 1: Data Quality Objective / Acceptance Criteria (in % recovery)

Compound	1613 Sample	1613 LCS	M23/TO9A Sample	M23/TO9A LCS	8290 Sample	8290 LCS
Native Standard						
2,3,7,8-TCDD		67-158		70-130		67-158
1,2,3,7,8-PeCDD		70-142		70-130		70-142
1,2,3,4,7,8-HxCDD		70-164		70-130		70-164
1,2,3,6,7,8-HxCDD		76-134		70-130		76-134
1,2,3,7,8,9-HxCDD		64-162		70-130		64-162
1,2,3,4,6,7,8-HpCDD		70-140		70-130		70-140
OCDD		78-144		70-130		78-144
2,3,7,8-TCDF		75-158		70-130		75-158
1,2,3,7,8-PeCDF		80-134		70-130		80-134
2,3,4,7,8-PeCDF		68-160		70-130		68-160
1,2,3,4,7,8-HxCDF		72-134		70-130		72-134
1,2,3,6,7,8-HxCDF		84-130		70-130		84-130
2,3,4,6,7,8-HxCDF		70-156		70-130		70-156
1,2,3,7,8,9-HxCDF		78-130		70-130		78-130
1,2,3,4,6,7,8-HpCDF		82-122		70-130		82-122
1,2,3,4,7,8,9-HpCDF		78-138		70-130		78-138
OCDF		63-170		70-130		63-170
Extraction Standard						
13C12-2,3,7,8-TCDD	25-164	20-175	40-130	40-130	40-130	40-130
13C12-1,2,3,7,8-PeCDD	25-181	21-227	40-130	40-130	40-130	40-130
13C12-1,2,3,4,7,8-HxCDD	32-141	21-193	-	-	40-130	40-130
13C12-1,2,3,6,7,8-HxCDD	28-130	25-163	40-130	40-130	40-130	40-130
	-	-	-	-	-	-
13C12-1,2,3,4,6,7,8-HpCDD	23-140	26-166	25-130	25-130	25-130	25-130
13C12-OCDD	17-157	13-138	25-130	25-130	25-130	25-130
13C12-2,3,7,8-TCDF	24-169	22-152	40-130	40-130	40-130	40-130
13C12-1,2,3,7,8-PeCDF	24-185	21-192	40-130	40-130	40-130	40-130
13C12-2,3,4,7,8-PeCDF	21-178	13-328	-	-	40-130	40-130
13C12-1,2,3,4,7,8-HxCDF	26-152	19-202	-	-	40-130	40-130
13C12-1,2,3,6,7,8-HxCDF	26-123	21-159	40-130	40-130	40-130	40-130
13C12-2,3,4,6,7,8-HxCDF	29-147	17-205	-	-	40-130	40-130
13C12-1,2,3,7,8,9-HxCDF	28-136	22-176	-	-	40-130	40-130
13C12-1,2,3,4,6,7,8-HpCDF	28-143	21-158	25-130	25-130	25-130	25-130
13C12-1,2,3,4,7,8,9-HpCDF	26-138	20-186	-	-	25-130	25-130
	-	-	-	-	-	-
Clean-up Standard						
37Cl4-2,3,7,8-TCDD	35-197	31-191			40-130	40-130
13C12-1,2,3,7,8,9-HxCDF			40-130	40-130		
Sampling Standard						
37Cl4-2,3,7,8-TCDD			70-130	70-130		
13C12-1,2,3,4,7,8-HxCDD			70-130	70-130		
13C12-2,3,4,7,8-PeCDF			70-130	70-130		
13C12-1,2,3,4,7,8-HxCDF			70-130	70-130		
13C12-1,2,3,4,7,8,9-HpCDF			70-130	70-130		

Table 2: Calibration Standards

		CS0	CS1	CS2	CS3	CS4	CS5
Natives	2,3,7,8-TCDD	0.1	0.5	2	10	40	200
	2,3,7,8-TCDF	0.1	0.5	2	10	40	200
	1,2,3,7,8-PeCDD	0.5	2.5	10	50	200	1000
	1,2,3,7,8-PeCDF	0.5	2.5	10	50	200	1000
	2,3,4,7,8-PeCDF	0.5	2.5	10	50	200	1000
	1,2,3,4,7,8-HxCDD	0.5	2.5	10	50	200	1000
	1,2,3,6,7,8-HxCDD	0.5	2.5	10	50	200	1000
	1,2,3,7,8,9-HxCDD	0.5	2.5	10	50	200	1000
	1,2,3,4,7,8-HxCDF	0.5	2.5	10	50	200	1000

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	1,2,3,6,7,8-HxCDF	0.5	2.5	10	50	200	1000
	1,2,3,7,8,9-HxCDF	0.5	2.5	10	50	200	1000
	2,3,4,6,7,8-HxCDF	0.5	2.5	10	50	200	1000
	1,2,3,4,6,7,8-HpCDD	0.5	2.5	10	50	200	1000
	1,2,3,4,6,7,8-HpCDF	0.5	2.5	10	50	200	1000
	1,2,3,4,7,8,9-HpCDF	0.5	2.5	10	50	200	1000
	OCDD	1	5	20	100	400	2000
	OCDF	1	5	20	100	400	2000
Labeled	2,3,7,8-TCDD-$^{13}C_{12}$	100	100	100	100	100	100
	2,3,7,8-TCDF-$^{13}C_{12}$	100	100	100	100	100	100
	1,2,3,7,8-PeCDD-$^{13}C_{12}$	100	100	100	100	100	100
	1,2,3,7,8-PeCDF-$^{13}C_{12}$	100	100	100	100	100	100
	2,3,4,7,8-PeCDF-$^{13}C_{12}$	100	100	100	100	100	100
	1,2,3,4,7,8-HxCDD-$^{13}C_{12}$	100	100	100	100	100	100
	1,2,3,6,7,8-HxCDD-$^{13}C_{12}$	100	100	100	100	100	100
	1,2,3,4,7,8-HxCDF-$^{13}C_{12}$	100	100	100	100	100	100
	1,2,3,6,7,8-HxCDF-$^{13}C_{12}$	100	100	100	100	100	100
	1,2,3,7,8,9-HxCDF-$^{13}C_{12}$	100	100	100	100	100	100
	2,3,4,6,7,8-HxCDF-$^{13}C_{12}$	100	100	100	100	100	100
	1,2,3,4,6,7,8-HpCDD-$^{13}C_{12}$	100	100	100	100	100	100
	1,2,3,4,6,7,8-HpCDF-$^{13}C_{12}$	100	100	100	100	100	100
	1,2,3,4,7,8,9-HpCDF-$^{13}C_{12}$	100	100	100	100	100	100
	OCDD-$^{13}C_{12}$	200	200	200	200	200	200
	2,3,7,8-TCDD-$^{37}Cl_4$	0.1	0.5	2	10	40	200
Injection	1,2,3,4-TCDD-$^{13}C_{12}$	100	100	100	100	100	100
	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	100	100	100	100	100	100

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Table 3: Calibration Control Limits

	1613B		M23 & TO-9A		8290A		ALL
	Initial Cal.	CCV	Initial Cal.	CCV	Initial Cal.	CCV	CVS***
	%RSD	% rec	%RSD	% Diff	%RSD	% Diff	% Diff
Natives							
2,3,7,8-TCDD	20	78-129	25	25	20	20*	15
2,3,7,8-TCDF	20	84-120	25	25	20	20*	15
1,2,3,7,8-PeCDD	20	78-130	25	25	20	20*	15
1,2,3,7,8-PeCDF	20	82-120	25	25	20	20*	15
2,3,4,7,8-PeCDF	20	82-122	25	25	20	20*	15
1,2,3,4,7,8-HxCDD	20	78-128	25	25	20	20*	15
1,2,3,6,7,8-HxCDD	20	78-128	25	25	20	20*	15
1,2,3,7,8,9-HxCDD	35	82-122	25	25	20	20*	15
1,2,3,4,7,8-HxCDF	20	90-112	25	25	20	20*	15
1,2,3,6,7,8-HxCDF	20	88-114	25	25	20	20*	15
1,2,3,7,8,9-HxCDF	20	90-112	25	25	20	20*	15
2,3,4,6,7,8-HxCDF	20	88-114	25	25	20	20*	15
1,2,3,4,6,7,8-HpCDD	20	86-116	25	25	20	20*	15
1,2,3,4,6,7,8-HpCDF	20	90-110	25	25	20	20*	15
1,2,3,4,7,8,9-HpCDF	20	86-116	25	25	20	20*	15
OCDD	20	79-126	25	25	20	20*	15
OCDF	35	63-159	30	30	20	20*	15
Labels							
2,3,7,8-TCDD-$^{13}C_{12}$	35	82-121	25	25	30	30**	15
2,3,7,8-TCDF-$^{13}C_{12}$	35	71-140	30	30	30	30**	15
1,2,3,7,8-PeCDD-$^{13}C_{12}$	35	62-160	30	30	30	30**	15
1,2,3,7,8-PeCDF-$^{13}C_{12}$	35	76-130	30	30	30	30**	15
2,3,4,7,8-PeCDF-$^{13}C_{12}$	35	77-130	25	25	30	30**	15
1,2,3,4,7,8-HxCDD-$^{13}C_{12}$	35	85-117	25	25	30	30**	15
1,2,3,6,7,8-HxCDD-$^{13}C_{12}$	35	85-118	25	25	30	30**	15
1,2,3,4,7,8-HxCDF-$^{13}C_{12}$	35	76-131	25	25	30	30**	15
1,2,3,6,7,8-HxCDF-$^{13}C_{12}$	35	70-143	30	30	30	30**	15
1,2,3,7,8,9-HxCDF-$^{13}C_{12}$	35	74-135	-	-	30	30****	15
2,3,4,6,7,8-HxCDF-$^{13}C_{12}$	35	73-137	30	30	30	30**	15
1,2,3,4,6,7,8-HpCDD-$^{13}C_{12}$	35	72-138	30	30	30	30**	15
1,2,3,4,6,7,8-HpCDF-$^{13}C_{12}$	35	78-129	30	30	30	30**	15
1,2,3,4,7,8,9-HpCDF-$^{13}C_{12}$	35	77-129	25	25	30	30**	15
OCDD-$^{13}C_{12}$	35	48-207	30	30	30	30**	15
2,3,7,8-TCDD-$^{37}Cl_4$	35	79-127	25	25	30	30**	15

* 25% is allowed for a post-run verification but when the value is above 20%, then the analyte quantification must be as per 8290A Section 8.3.2.4 and corrective action is required before more samples can be analyzed.
**35% is allowed for a post-run verification but when the value is above 30%, then the analyte quantification must be as per 8290A Section 8.3.2.4 and corrective action is required before more samples can be analyzed.
*** Calibration Verification Standard. A second-source standard used to verify the primary source initial calibration standard. (See NA-SP-0104)
**** With the use of the enhanced standard set for method 8290a, the CCV limit has been set to match the other labelled standards.

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Table 4: Quantitation References and Method 1613B RT References and RRT

Analyte	Stack/Ambient Quantitation Reference	Method 1613B RT Reference Solids/ Waters Quantitation Reference	Method 1613B RRT
Compounds using 1,2,3,4-TCDD-$^{13}C_{12}$ as injection standard			
2,3,7,8-TCDF	2,3,7,8-TCDF-$^{13}C_{12}$	2,3,7,8-TCDF-$^{13}C_{12}$	0.999-1.003
2,3,7,8-TCDD	2,3,7,8-TCDD-$^{13}C_{12}$	2,3,7,8-TCDD-$^{13}C_{12}$	0.999-1.002
1,2,3,7,8-PeCDF	1,2,3,7,8-PeCDF-$^{13}C_{12}$	1,2,3,7,8-PeCDF-$^{13}C_{12}$	0.999-1.002
2,3,4,7,8-PeCDF	1,2,3,7,8-PeCDF-$^{13}C_{12}$	2,3,4,7,8-PeCDF-$^{13}C_{12}$	0.999-1.002
1,2,3,7,8-PeCDD	1,2,3,7,8-PeCDD-$^{13}C_{12}$	1,2,3,7,8-PeCDD-$^{13}C_{12}$	0.999-1.002
2,3,7,8-TCDF-$^{13}C_{12}$	1,2,3,4-TCDD-$^{13}C_{12}$	1,2,3,4-TCDD-$^{13}C_{12}$	0.923-1.103
2,3,7,8-TCDD-$^{13}C_{12}$	1,2,3,4-TCDD-$^{13}C_{12}$	1,2,3,4-TCDD-$^{13}C_{12}$	0.976-1.043
2,3,7,8-TCDD-$^{37}Cl_4$	2,3,7,8-TCDD-$^{13}C_{12}$	1,2,3,4-TCDD-$^{13}C_{12}$	0.989-1.052
1,2,3,7,8-PeCDF-$^{13}C_{12}$	1,2,3,4-TCDD-$^{13}C_{12}$	1,2,3,4-TCDD-$^{13}C_{12}$	1.000-1.425
2,3,4,7,8-PeCDF-$^{13}C_{12}$	1,2,3,7,8-PeCDF-$^{13}C_{12}$	1,2,3,4-TCDD-$^{13}C_{12}$	1.011-1.526
1,2,3,7,8-PeCDD-$^{13}C_{12}$	1,2,3,4-TCDD-$^{13}C_{12}$	1,2,3,4-TCDD-$^{13}C_{12}$	1.000-1.567
Compounds using 1,2,3,7,8,9-HxCDD-$^{13}C_{12}$ as injection standard			
1,2,3,4,7,8-HxCDF	1,2,3,6,7,8-HxCDF-$^{13}C_{12}$	1,2,3,4,7,8-HxCDF-$^{13}C_{12}$	0.999-1.001
1,2,3,6,7,8-HxCDF	1,2,3,6,7,8-HxCDF-$^{13}C_{12}$	1,2,3,6,7,8-HxCDF-$^{13}C_{12}$	0.997-1.005
1,2,3,7,8,9-HxCDF	1,2,3,6,7,8-HxCDF-$^{13}C_{12}$	1,2,3,7,8,9-HxCDF-$^{13}C_{12}$	0.999-1.001
2,3,4,6,7,8-HxCDF	2,3,4,6,7,8-HxCDF-$^{13}C_{12}$	2,3,4,6,7,8-HxCDF-$^{13}C_{12}$	0.999-1.001
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDD-$^{13}C_{12}$	1,2,3,4,7,8-HxCDD-$^{13}C_{12}$	0.999-1.001
1,2,3,6,7,8-HxCDD	1,2,3,6,7,8-HxCDD-$^{13}C_{12}$	1,2,3,6,7,8-HxCDD-$^{13}C_{12}$	0.998-1.004
1,2,3,7,8,9-HxCDD[a]	1,2,3,6,7,8-HxCDD-$^{13}C_{12}$	[a]	1.000-1.019
1,2,3,4,6,7,8-HpCDF	1,2,3,4,6,7,8-HpCDF-$^{13}C_{12}$	1,2,3,4,6,7,8-HpCDF-$^{13}C_{12}$	0.999-1.001
1,2,3,4,7,8,9-HpCDF	1,2,3,4,6,7,8-HpCDF-$^{13}C_{12}$	1,2,3,4,7,8,9-HpCDF-$^{13}C_{12}$	0.999-1.001
1,2,3,4,6,7,8-HpCDD	1,2,3,4,6,7,8-HpCDD-$^{13}C_{12}$	1,2,3,4,6,7,8-HpCDD-$^{13}C_{12}$	0.999-1.001
OCDF	OCDD-$^{13}C_{12}$	OCDD-$^{13}C_{12}$	0.999-1.008
OCDD	OCDD-$^{13}C_{12}$	OCDD-$^{13}C_{12}$	0.999-1.001
1,2,3,4,7,8-HxCDF-$^{13}C_{12}$	1,2,3,6,7,8-HxCDF-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	0.944-0.970
1,2,3,6,7,8-HxCDF-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	0.949-0.975
1,2,3,7,8,9-HxCDF-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	0.977-1.047
2,3,4,6,7,8-HxCDF-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	0.959-1.021
1,2,3,4,7,8-HxCDD-$^{13}C_{12}$	1,2,3,6,7,8-HxCDD-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	0.977-1.000
1,2,3,6,7,8-HxCDD-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	0.981-1.003
1,2,3,4,6,7,8-HpCDF-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	1.043-1.085
1,2,3,4,7,8,9-HpCDF-$^{13}C_{12}$	1,2,3,4,6,7,8-HpCDF-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	1.057-1.151
1,2,3,4,6,7,8-HpCDD-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	1.086-1.110
OCDD-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	1.032-1.311

[a] For solids/waters via 1612B and 8290A, 1,2,3,7,8,9-HXCDD is quantified against the average responses of 1,2,3,4,7,8-HxCDD-$^{13}C_{12}$ and 1,2,3,6,7,8-HxCDD-$^{13}C_{12}$ while 1,2,3,6,7,8-HxCDD-$^{13}C_{12}$ is the RT reference.

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Table 5: HRMS Suggested Acquisition Descriptor Parameters

Descriptor	Exact M/Z	M/Z Type	Elemental Composition	Substance	Type
1	303.9016	M	$^{12}C_{12}$ $^{1}H_4$ $^{35}Cl_4$ ^{16}O	TCDF	Native
	305.8987	M+2	$^{12}C_{12}$ $^{1}H_4$ $^{35}Cl_3$ ^{37}Cl ^{16}O	TCDF	Native
	315.9419	M	$^{13}C_{12}$ $^{1}H_4$ $^{35}Cl_4$ ^{16}O	TCDF	^{13}C
	316.9824	Lock	$^{12}C_9$ $^{19}F_{11}$	PFK	Lock
	317.9389	M+2	$^{13}C_{12}$ $^{1}H_4$ $^{35}Cl_3$ ^{37}Cl ^{16}O	TCDF	^{13}C
	319.8965	M	$^{12}C_{12}$ $^{1}H_4$ $^{35}Cl_4$ $^{16}O_2$	TCDD	Native
	321.8936	M+2	$^{12}C_{12}$ $^{1}H_4$ $^{35}Cl_3$ ^{37}Cl $^{16}O_2$	TCDD	Native
	327.8847	M+8	$^{12}C_{12}$ $^{1}H_4$ $^{37}Cl_4$ $^{16}O_2$	TCDD	Native - ^{37}Cl enriched
	331.9368	M	$^{13}C_{12}$ $^{1}H_4$ $^{35}Cl_4$ $^{16}O_2$	TCDD	^{13}C
	333.9339	M+2	$^{13}C_{12}$ $^{1}H_4$ $^{35}Cl_3$ ^{37}Cl $^{16}O_2$	TCDD	^{13}C
	375.8364	M+2	$^{12}C_{12}$ $^{1}H_4$ $^{35}Cl_5$ ^{37}Cl ^{16}O	HxCDPE	Cl-DPE
2	339.8597	M+2	$^{12}C_{12}$ $^{1}H_3$ $^{35}Cl_4$ ^{37}Cl ^{16}O	PeCDF	Native
	341.8568	M+4	$^{12}C_{12}$ $^{1}H_3$ $^{35}Cl_3$ $^{37}Cl_2$ ^{16}O	PeCDF	Native
	351.9000	M+2	$^{13}C_{12}$ $^{1}H_3$ $^{35}Cl_4$ ^{37}Cl ^{16}O	PeCDF	^{13}C
	353.8970	M+4	$^{13}C_{12}$ $^{1}H_3$ $^{35}Cl_3$ $^{37}Cl_2$ ^{16}O	PeCDF	^{13}C
	355.8546	M+2	$^{12}C_{12}$ $^{1}H_3$ $^{35}Cl_4$ ^{37}Cl $^{16}O_2$	PeCDD	Native
	357.8517	M+4	$^{12}C_{12}$ $^{1}H_3$ $^{35}Cl_3$ $^{37}Cl_2$ $^{16}O_2$	PeCDD	Native
	365.8978				
	366.9792	Lock	$^{12}C_{10}$ $^{19}F_{13}$	PFK	Lock
	367.8949	M+2	$^{13}C_{12}$ $^{1}H_3$ $^{35}Cl_4$ ^{37} Cl $^{16}O_2$	PeCDD	^{13}C
	369.8919	M+4	$^{13}C_{12}$ $^{1}H_3$ $^{35}Cl_3$ ^{37} Cl_2 $^{16}O_2$	PeCDD	^{13}C
	409.7974	M+2	$^{12}C_{12}$ $^{1}H_3$ $^{35}Cl_6$ ^{37}Cl ^{16}O	HpCDPE	Cl-DPE
3	373.8207	M+2	$^{12}C_{12}$ $^{1}H_2$ $^{35}Cl_5$ ^{37}Cl ^{16}O	HxCDF	Native
	375.8178	M+4	$^{12}C_{12}$ $^{1}H_2$ $^{35}Cl_4$ $^{37}Cl_2$ ^{16}O	HxCDF	Native
	380.9760	Lock	$^{12}C_8$ $^{19}F_5$	PFK	Lock
	383.8639	M	$^{13}C_{12}$ $^{1}H_2$ $^{35}Cl_6$ ^{16}O	HxCDF	^{13}C
	385.8610	M+2	$^{13}C_{12}$ $^{1}H_2$ $^{35}Cl_5$ ^{37}Cl ^{16}O	HxCDF	^{13}C
	389.8156	M+2	$^{12}C_{12}$ $^{1}H_2$ $^{35}Cl_5$ ^{37}Cl $^{16}O_2$	HxCDD	Native
	391.8127	M+4	$^{12}C_{12}$ $^{1}H_2$ $^{35}Cl_4$ $^{37}Cl_2$ $^{16}O_2$	HxCDD	Native
	401.8559	M+2	$^{13}C_{12}$ $^{1}H_2$ $^{35}Cl_5$ ^{37}Cl $^{16}O_2$	HxCDD	^{13}C
	403.8530	M+4	$^{13}C_{12}$ $^{1}H_2$ $^{35}Cl_4$ $^{37}Cl_2$ $^{16}O_2$	HxCDD	^{13}C
	445.7555	M+4	$^{12}C_{12}$ $^{1}H_2$ $^{35}Cl_6$ $^{37}Cl_2$ ^{16}O	OCDPE	Cl-DPE
4	407.7818	M+2	$^{12}C_{12}$ ^{1}H $^{35}Cl_6$ ^{37}Cl ^{16}O	HpCDF	Native
	411.7759				
	409.7789	M+4	$^{12}C_{12}$ ^{1}H $^{35}Cl_5$ $^{37}Cl_2$ ^{16}O	HpCDF	Native
	417.8253	M	$^{13}C_{12}$ ^{1}H $^{35}Cl_7$ ^{16}O	HpCDF	^{13}C
	419.8220	M+2	$^{13}C_{12}$ ^{1}H $^{35}Cl_6$ ^{37}Cl ^{16}O	HpCDF	^{13}C
	423.7767	M+2	$^{12}C_{12}$ ^{1}H $^{35}Cl_6$ ^{37}Cl $^{16}O_2$	HpCDD	Native
	425.7737	M+4	$^{12}C_{12}$ ^{1}H $^{35}Cl_5$ $^{37}Cl_2$ $^{16}O_2$	HpCDD	Native
	430.9728	Lock	$^{12}C_9$ $^{19}F_{17}$	PFK	Lock
	435.8169	M+2	$^{13}C_{12}$ ^{1}H $^{35}Cl_6$ ^{37}Cl $^{16}O_2$	HpCDD	^{13}C
	437.8140	M+4	$^{13}C_{12}$ ^{1}H $^{35}Cl_5$ $^{37}Cl_2$ $^{16}O_2$	HpCDD	^{13}C
	454.9728	Lock	$^{12}C_{11}$ $^{19}F_{17}$	PFK	Lock
	479.7165	M+4	$^{12}C_{12}$ ^{1}H $^{35}Cl_7$ $^{37}Cl_2$ ^{16}O	NCDPE	Cl-DPE
5	441.7428	M+2	$^{12}C_{12}$ $^{35}Cl_7$ ^{37}Cl ^{16}O	OCDF	Native
	443.7399	M+4	$^{12}C_{12}$ $^{35}Cl_6$ $^{37}Cl2$ ^{16}O	OCDF	Native
	454.9728	Lock	$^{12}C_{11}$ $^{19}F_{17}$	PFK	Lock
	457.7377	M+2	$^{12}C_{12}$ $^{35}Cl_7$ ^{37}Cl $^{16}O_2$	OCDD	Native
	459.7348	M+4	$^{12}C_{12}$ $^{35}Cl_6$ $^{37}Cl_2$ $^{16}O_2$	OCDD	Native
	469.7780	M+2	$^{13}C_{12}$ $^{35}Cl_7$ ^{37}Cl $^{16}O_2$	OCDD	^{13}C
	471.7750	M+4	$^{13}C_{12}$ $^{35}Cl_6$ $^{37}Cl_2$ $^{16}O_2$	OCDD	^{13}C
	513.6775	M+4	$^{12}C_{12}$ $^{35}Cl_8$ $^{37}Cl_2$ ^{16}O	DCDPE	Cl-DPE

The masses found in descriptor 2 for PeCDF and its corresponding DPE are also in descriptor 1 due to the first PeCDF eluting at approximately the same time as the last eluting TCDD/F.

Alternate abundant PFK fragment masses may be used for the purposes of mass lock in order to best reduce interferences

Table 6: LOD/LOQ Levels (DoD PJLA)

Target Analytes	Soils/Sediments/Solids pg/g	Tissues pg/g	Air (Stack/Ambient) pg	Water pg/L

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2,3,7,8-TCDD	1	1	10	10
1,2,3,7,8-PeCDD	5	5	50	50
1,2,3,4,7,8-HxCDD	5	5	50	50
1,2,3,6,7,8-HxCDD	5	5	50	50
1,2,3,7,8,9-HxCDD	5	5	50	50
1,2,3,4,6,7,8-HpCDD	5	5	50	50
OCDD	10	10	100	100
2,3,7,8-TCDF	1	1	10	10
1,2,3,7,8-PeCDF	5	5	50	50
2,3,4,7,8-PeCDF	5	5	50	50
1,2,3,4,7,8-HxCDF	5	5	50	50
1,2,3,6,7,8-HxCDF	5	5	50	50
2,3,4,6,7,8-HxCDF	5	5	50	50
1,2,3,7,8,9-HxCDF	5	5	50	50
1,2,3,4,6,7,8-HpCDF	5	5	50	50
1,2,3,4,7,8,9-HpCDF	5	5	50	50
OCDF	10	10	100	100

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Table 7a: PCDD/F LCS Limits for Solid/Tissue/Air Matrices (DoD Analysis Only)

Analyte	Lower Control Limit	Upper Control Limit
1,2,3,4,5,6,7,8-Octachlorodibenzo-p- dioxin	73	135
1,2,3,4,6,7,8,9-Octachlorodibenzofuran	66	144
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	76	125
1,2,3,4,6,7,8-Heptachlorodibenzofuran	73	135
1,2,3,4,7,8,9-Heptachlorodibenzofuran	72	131
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	72	131

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1,2,3,4,7,8-Hexachlorodibenzofuran	77	130
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	74	134
1,2,3,6,7,8-Hexachlorodibenzofuran	73	134
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	71	138
1,2,3,7,8,9-Hexachlorodibenzofuran	74	135
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	74	125
1,2,3,7,8-Pentachlorodibenzofuran	77	131
2,3,4,6,7,8-Hexachlorodibenzofuran	74	133
2,3,4,7,8-Pentachlorodibenzofuran	75	128
2,3,7,8-Tetrachlorodibenzo-p-dioxin	70	128
2,3,7,8-Tetrachlorodibenzofuran	75	135

* Sporadic marginal exceedances are allowed for those analytes outside the 3 standard deviation control limits but still within 4 standard deviations. Marginal exceedances are not allowed for those analytes determined by a project to be target analytes without project specific approval.

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Table 7b: PCDD/F LCS Limits for Water Matrix (DoD Analysis Only)

Analyte	Lower Control Limit	Upper Control Limit
1,2,3,4,5,6,7,8-Octachlorodibenzo-p- dioxin	81	135
1,2,3,4,6,7,8,9-Octachlorodibenzofuran	66	150
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	79	122
1,2,3,4,6,7,8-Heptachlorodibenzofuran	81	130
1,2,3,4,7,8,9-Heptachlorodibenzofuran	77	128
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	80	126
1,2,3,4,7,8-Hexachlorodibenzofuran	80	130
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	78	134
1,2,3,6,7,8-Hexachlorodibenzofuran	79	131
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	76	137
1,2,3,7,8,9-Hexachlorodibenzofuran	83	130
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	76	121
1,2,3,7,8-Pentachlorodibenzofuran	82	130
2,3,4,6,7,8-Hexachlorodibenzofuran	81	130
2,3,4,7,8-Pentachlorodibenzofuran	77	129
2,3,7,8-Tetrachlorodibenzo-p-dioxin	71	125
2,3,7,8-Tetrachlorodibenzofuran	72	138

* Sporadic marginal exceedances are allowed for those analytes outside the 3 standard deviation control limits but still within 4 standard deviations. Marginal exceedances are not allowed for those analytes determined by a project to be target analytes without project specific approval.

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Table 8. CAS Numbers for Non-2,3,7,8 Substituted Native Compounds and Labeled Compounds

Compound	CAS No.
1,3,6,8-TCDD	33423-92-6
1,2,8,9-TCDD	62470-54-6
1,3,6,8-TCDF	71998-72-6
1,2,8,9-TCDF	70648-22-5
1,2,4,6,8/1,2,4,7,9-PeCDD	N/A
1,2,3,8,9-PeCDD	N/A
1,3,4,6,8-PeCDF	N/A
1,2,3,8,9-PeCDF	83704-54-5
1,2,4,6,7,9/1,2,4,6,8,9-HxCDD	39227-62-8
1,2,3,4,6,8-HxCDF	69698-60-8
1,2,3,4,6,7,9-HpCDD	58200-70-7
1,2,3,4-TCDD	30746-58-8
1,2,3,7/1,2,3,8-TCDD	N/A
1,2,3,9-TCDD	72669-26-6
1,2,3,9-TCDF	83704-23-8
2.3.4.7-TCDF	83704-31-8
2.3.4.8-TCDF	83704-32-9
13C12-2,3,7,8-TCDD	76523-40-5
13C12-1,2,3,7,8-PCDD	109719-79-1
13C12-1,2,3,4,7,8-HxCDD	109719-80-4
13C12-1,2,3,6,7,8-HxCDD	109719-81-5
13C12-1,2,3,4,6,7,8-HpCDD	109719-83-7
13C12-OCDD	114423-97-1
13C12-2,3,7,8-TCDF	89059-46-1
13C12-1,2,3,7,8-PCDF	109719-77-9
13C12-2,3,4,7,8-PCDF	116843-02-8
13C12-1,2,3,4,7,8-HxCDF	114423-98-2
13C12-1,2,3,6,7,8-HxCDF	116843-03-9
13C12-1,2,3,7,8,9-HxCDF	116843-04-0
13C12-2,3,4,6,7,8-HxCDF	116843-05-1
13C12-1,2,3,4,6,7,8-HpCDF	109719-84-8
13C12-1,2,3,4,7,8,9-HpCDF	109719-94-0
13C12-1,2,3,7,8,9-HxCDD	109719-82-6
13C12-1,2,3,4-TCDD	30746-58-8
37Cl4-2,3,7,8-TCDD	85508-50-5
13C12-OCDF	114423-97-1
13C12-1,2,7,8-TCDF	N/A
13C12-1,2,4,7,8-PeCDD	N/A
13C12-1,2,3,4,6,7,9-HpCDD	N/A

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PCDD/F, PCB & PCN PREPARATIVE METHOD FOR ISOTOPE DILUTION GC/MS

Document Authorization

Authorized By: _____ Date: _____
Quality Authorization

Authorized By: _____ Date: _____
Technical Authorization

Implementation Authorization

BURLINGTON

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Laboratory Location

Implementation Authorization

Authorized By: Effective Date: _____

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1.0 SCOPE AND APPLICATION

1.1 Scope

This method provides procedures for the extraction, clean-up, and general preparation of extracts prior to instrumental detection and quantitative measurement of polychlorinated dibenzo(p)dioxins (tetra- through octachlorinated homologues; PCDD), polychlorinated dibenzofurans (tetra- through octachlorinated homologues; PCDF) and chlorinated biphenyls (PCB) via isotope dilution GC/MS.

1.2 Parameters and Reporting Limits

Refer to corresponding test method.

BU-TM-1107 PCDD_F	(for the analysis of PCDD/F only in all matrices)
BU-TM-1105 PCB Congeners	(for the analysis of PCB in all matrices)
BU-TM-1102 PCN	(for the analysis of PCN in all matrices)

Note: There are circumstances where it can be advantageous to analyze selected PCBs within the dioxin/furan analysis. By the judicious application of column clean-ups, this method provides different approaches to the separation and ultimate analyses of PCDD/F and PCB. The PCB can be separated completely from the tetra- through octa- chlorinated dioxins and furans using florisil columning. Alternatively, the bulk of the PCB can be separated from the dioxins/furans and from selected toxic PCBs (e.g. coplanars) using carbon columning. As a third option, applicable where PCB levels are low enough not to cause significant interference on the dioxin/furan analyses, one may be best served not to separate the PCBs at all.

1.3 Method Performance Data and Measurement Uncertainty

Refer to NA-SP-0100 Initial Method Validation – Chemistry and NA-SP-0102 Method Revalidation and MU – Chemistry for policies on determination and storage of method performance data.

2.0 PRINCIPLE OF METHOD

PCDD/F via HRMS:
Samples are extracted and cleaned in preparation of GC HRMS analysis for PCDD/F.

PCB via GC/HRMS
Samples are extracted and cleaned in preparation of the congener analysis of chlorinated biphenyls via isotope dilution GC/HRMS.

PCN via GC/HRMS
Samples are extracted and cleaned in preparation of the congener analysis of polychlorinated naphthalenes via isotope dilution GC/HRMS.

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3.0 DEFINITIONS

Refer to NA-SP-0001 Definitions of Key Terms.

For isotope dilution analytical methods, there is inconsistent use of terms within the literature and reference methods. Much of the confusion is derived from the timing of the addition of internal standards. For example, for most isotope dilution methods, the internal standards are added prior to extraction while in most other GC or GC/MS applications (i.e. non-isotope dilution analyses) within the environmental arena, the internals are added just prior to analysis. This confusion is avoided by applying terms that are defined by the timing of their addition rather than by any other the use in the calculation of the data.

Extraction Standards: are the labelled analytes added just prior to extraction. For the data calculations, these are true internal standards - used for determining analyte concentration.

Clean-up Standards: are the labelled analytes added just prior to clean-up. Recoveries of the clean-up standard(s) provide an indication on the losses that occur during all the preparative stages that follow the extraction.

Injection Standards: are the labelled analytes added just prior to analysis. These are used as an internal standard reference to determine the recoveries of the extraction standards.

Sampling Standards: (also called field spikes) are the labelled analytes added, where applicable, prior to sampling onto sorbent media used for analyte capture during the sampling event. Sorbent media such as XAD2 resin and polyurethane foam (PUF) are used for stack and ambient air sampling. XAD2 resin is also used for the sampling of large volumes of water.

4.0 INTERFERENCES

Chlorinated and brominated species (e.g. polychlorinated diphenyl ethers, polybrominated dioxins/furans and polybrominated diphenyl ethers) can be particularly troublesome. Phthalate esters can contaminate extracts through handling and provide interferences in instrumental analyses.

Proper cleaning of glassware is extremely important, because glassware may not only contaminate the samples but may also remove the analytes of interest by adsorption on the glass surface.

See BU-WI-3000 Organic Glassware/Equipment Cleaning, Proofing and Maintenance for proper cleaning techniques.

Disposable glassware should be used where possible. Immediately prior to use, the Soxhlet apparatus should be pre-extracted with acetone for approximately 3 hours.

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5.0 SAMPLE HANDLING AND STORAGE

PCDD/F are generally stable within sample matrices and are not usually subject to degradation when protected from light. Recommended hold times of are variable dependent upon the method.

DX:

Parameter(s) and Matrix: All parameters (including PCB) – Stack	
Container	Pre-cleaned amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE). Glass Petri dishes are not recommended for transportation of filters to and from the field since these are susceptible to breakage during transportation. Reference: EPA SW846, Chapter 4, Rev, July 2014
Lab Storage Conditions	4˚C in the dark Reference: EPA Method 8290A Section 8.3
Maximum Sample Holding Time (Provinces / Reference)	30 days Reference: EPA Method 23 Section 5, 0023A Section 7.3.3, 8290A Section 8.3
Maximum Extract / Digest Holding Time (Provinces / References)	45 days Reference: EPA Method 23 Section 5, 0023A Section 7.3.3, 8290A Section 8.3

Parameter(s) and Matrix: All parameters (including PCB) – Ambient Air	
Container	Pre-cleaned amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE). Glass Petri dishes are not recommended for transportation of filters to and from the field since these are susceptible to breakage during transportation. Reference: EPA SW846, Chapter 4, Rev, July 2014
Lab Storage Conditions	4˚C Reference: EPA TO-9A Section 11.3.4.9
Maximum Sample Holding Time (Provinces / Reference)	7 days Reference: EPA TO-9A Section 11.3.4.9
Maximum Extract / Digest Holding Time (Provinces / References)	40 days Reference: EPA TO-9A Section 11.3.4.9

Parameter(s) and Matrix: All parameters via 1613B - Water	
Container	Pre-cleaned amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE) Reference: EPA SW 846 Chapter 4, Rev 5, July 2014
Preservative	Ontario Drinking Waters: unpreserved Reference: MOE DW Practices Waters (others): 80mg/L of sodium thiosulphate to remove residual chlorine by ALS upon receipt Reference: EPA 1613B Section 8.2

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Parameter(s) and Matrix: All parameters via 1613B - Water	
Sample Pre-treatment	Waters (non-drinking waters): Tested for pH and adjusted if necessary. See BU-WI-0325 Water Preservation.
Lab Storage Conditions	Ontario Drinking Waters: 2-8˚C (Ref: MOE DW Practices) Waters (others): >0 - 6˚C (Ref: US EPA 40CFR 2012)
Maximum Sample Holding Time (Provinces / Reference)	Ontario Drinking Waters: 30 days (Ref: MOE DW Practices) Waters (others): 365 days (Ref: 1613B Section 8.4.1)
Maximum Extract / Digest Holding Time (Provinces / References)	Ontario Drinking Waters: 30 days (Ref: MOE DW Practices) Waters (others): 365 days @ ≤ -10˚C (Ref: 1613B Section 8.4.2)

Parameter(s) and Matrix: All parameters via 1613B – Soil/Sediment/Solid/Waste (including Non-perishable Feed/Foods/Nutraceuticals)	
Container	Pre-cleaned amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE) Reference: EPA SW846, Chapter 4, Rev, July 2014
Lab Storage Conditions	>0 - 6˚C Reference: US EPA 40CFR 2012
Maximum Sample Holding Time (Provinces / Reference)	365 Days Reference: 1613B Section 8.4.1
Maximum Extract / Digest Holding Time (Provinces / References)	365 Days Reference: 1613B Section 8.4.2

Parameter(s) and Matrix: All parameters via 1613B – Tissue (including perishable Feed/Foods/Nutraceuticals	
Container	Pre-cleaned amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE). Solid tissues such as whole fish can be wrapped in solvent cleaned aluminum foil. Reference: EPA SW846, Chapter 4, Rev, July 2014
Lab Storage Conditions	≤ -10˚C Reference: 1613B Section 8.3.3
Maximum Sample Holding Time (Provinces / Reference)	365 days Reference: 1613B Section 8.4.1
Maximum Extract / Digest Holding Time (Provinces / References)	365 days @ ≤ -10˚C Reference: 1613B Section 8.4.2

PCBs & PCNs:

Parameter(s) and Matrix: PCBs & PCNs - Water	
Container	Pre-cleaned amber 1L glass bottles with caps or lids that are lined with polytetrafluoroethene (PTFE) Reference: 1668C Section 8.0
Preservative	80mg/L of sodium thiosulphate to remove residual chlorine by ALS upon receipt. See BU-WI-0325 Water Preservation.

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Parameter(s) and Matrix: PCBs & PCNs - Water	
	Reference: 1668C Section 8.2.2
Lab Storage Conditions	>0 - 6˚C Reference: 1668C Section 8.2.3
Maximum Sample Holding Time (Provinces / Reference)	365 days Reference: 1668C Section 8.5.1
Maximum Extract / Digest Holding Time (Provinces / References)	365 days @ ≤ -10˚C Reference: 1668C Section 8.5.2

Parameter(s) and Matrix: PCBs & PCNs – Soil/Sediment/Solid/Waste (including Non-perishable Feed/Foods/Nutraceuticals)	
Container	Pre-cleaned amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE) 250mL preferred if sufficient sample available Reference: 1668C Section 6.1.1
Lab Storage Conditions	>0-6˚C in the dark Reference: 1668C Section 8.3.2
Maximum Sample Holding Time (Provinces / Reference)	365 days Reference: 1668C Section 8.5.1
Maximum Extract / Digest Holding Time (Provinces / References)	365 days @ ≤ -10˚C Reference: 1668C Section 8.5.2

Parameter(s) and Matrix: PCBs & PCNs – Tissue (including perishable Feed/Foods/Nutraceuticals)	
Container	Pre-cleaned amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE). Solid tissues such as whole fish can be wrapped in solvent cleaned aluminum foil. Reference: 1668C Section 6.1.1
Lab Storage Conditions	<-10˚C in the dark Reference: 1668C Section 8.4.3
Maximum Sample Holding Time (Provinces / Reference)	365 days Reference: 1668C Section 8.5.1
Maximum Extract / Digest Holding Time (Provinces / References)	365 days @ ≤ -10˚C Reference: 1668C Section 8.5.2

Parameter(s) and Matrix: PCBs & PCNs – Blood and Blood products	
Container	Plastic test tubes Reference: CDC Laboratory Procedure Manual
Lab Storage Conditions	<-80˚C in the dark Reference: CDC Laboratory Procedure Manual
Maximum Sample Holding Time (Provinces / Reference)	365 days Reference: 1668C Section 8.5.1
Maximum Extract / Digest	365 days @ ≤ -10˚C

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Parameter(s) and Matrix: PCBs & PCNs – Blood and Blood products	
Holding Time (Provinces / References)	Reference: 1668C Section 8.5.2

ALS requirements are: to store all samples and extracts in the dark and to comply with the method recommended hold times and temperature storage requirements except that solids destined for 8290A analysis may be stored frozen at <-10˚C. Short term storage of extracts (i.e. prior to the completion of analysis) is in a refrigerator at 4˚C. Long term storage of extracts is below -10˚C.

5.1 Sampling Instructions

Sample collection is not conducted by the laboratory.

5.2 Sampling Handling and Transportation

Refer to ALS Sample Handling and Preservation Guidelines for further details on the sample handling procedures recommended by ALS.

If holding time violations occur, Account Manager(s) must be notified.

6.0 EQUIPMENT AND SUPPLIES

The following equipment and supplies have been shown to meet the performance specifications of this test method. Refer to site-specific equipment logs, procedures and analytical records to identify the instrumentation and supplies used in each lab.

6.1 Primary Equipment

1. Soxhlet heating mantle
2. Top-loading balance capable of accurately weighing to two decimal places
3. Analytical balance capable of accurately weighing to four decimal places

6.2 Support Equipment

1. Muffle furnace capable of maintain 450˚C ±4˚C for a minimum of 8 hours
2. Reagent oven capable of maintaining 130˚C ±4˚C for a minimum of 6 hours
3. Drying oven
4. Rotary evaporator.
5. N-Evap
6. Stir plate with Teflon stir bar
7. Vortex mixer

6.3 Supplies

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6.3.1 Glassware

Note: All glassware for volumetric measurement in Burlington is Class A.

1. 1000 mL florence flask with 24/40 ground glass joint and stopper, VWR, MEOL# n/a
2. 500 mL florence flasks with 24/40 ground glass joint, VWR, MEOL# 4000463
3. 125 mL florence flask with 24/40 ground glass joint and stopper, VWR, MEOL# n/a
4. Soxhlet extraction apparatuses, Jon Vandenhoff, ordered directly
5. Dean-Stark adapters, Jon Vandenhoff, ordered directly
6. 1000 mL graduated cylinder, VWR, MEOL# n/a
7. 100 mL graduated cylinder, VWR, MEOL# 4001645
8. 10 mL graduated cylinder, VWR, MEOL# 4001647
9. 2000 mL erlenmeyer flask, Fisher, MEOL# n/a
10. 1000 mL erlenmeyer flask, Fisher, MEOL# n/a
11. 250 mL erlenmeyer flasks, Fisher, MEOL# 4000090
12. 500 mL glass beakers, Fisher, MEOL# 4000307
13. 500 mL quartz beakers, VWR, MEOL# 4001474
14. Allihn filters, column length = 11cm, internal diameter = 4cm, contain a coarse fritted disk (40-60 microns), Jon Vandenhoff, ordered directly
15. 2L Glass separatory funnels, Jon Vandenhoff, ordered directly
16. Teflon stopper and stopcock, Jon Vandenhoff, ordered directly

6.3.2 Sample Preparation Supplies

1. 1 mL pipette bulbs, Fisher, MEOL#4430106
2. 5 1/4“disposable pipettes, Fisher, MEOL#4430061
3. 9” disposable pipettes, Fisher, MEOL#4430062
4. 10 mL disposable pipettes, Fisher, MEOL#4430178
5. Glass wool, Fisher, MEOL#4340169
6. Boiling chips, MEOL#6040942
7. 700 µL auto-sampler vials, Canadian Life Science, MEOL#4320149
8. Teflon lined auto-sampler vial caps, Canadian Life Science, MEOL#4320752
9. pH paper, VWR, MEOL#4350001
10. 15 mL disposable c-tubes, VWR, MEOL#4000187
11. 50 mL disposable c-tubes, VWR, MEOL#4000202
12. Disposable glass columns L=40cm I.D.=15mm, John Vandenhoff, ordered directly
13. Aluminum Foil, Whitebird, MEOL#4550248 (30cmx200m) and MEOL#4550376 (45cm x100cm)
14. Nitrogen, ultra-high purity, Praxair, ordered on the Praxair website
15. 1L ‘pre-cleaned’ amber glass bottles with Teflon lined lids, Systems Plus, MEOL#4000432
16. 250 mL ‘pre-cleaned’ amber glass bottles with Teflon lined lids, Systems Plus, MEOL#4000260
17. Polyethylene gloves, Fisher, MEOL#4240474 (small), MEOL#4240156 (medium), MEOL#4240486 (large)
18. Teflon tape, Fisher, MEOL#4030046 (1/2”), MEOL#4030089 (1”)
19. Detergent (Liquinox), Caledon, MEOL#4230093 (1 gallon), MEOL#4230884 (5 gallon)
20. Adhesive labels, MEOL# 8020034

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21. Permanent fine-tip marker
22. Support racks and various clamps
23. Teflon squeeze bottles, MEOL# 4030011
24. Stainless steel spatulas
25. Scissors
26. 5 µL-5000 µL syringes, Hamilton, MEOL # for various syringes can be found in G:\Enviro\Purchasing\Organic Prep\Approved Supplies.xls (Syringes Tab)
27. Mortar and Pestle - glass
28. Cotton extraction thimbles, 43x123mm, MEOL# 4340114

Note: All equipment manuals can be found in "Organic Prep Lab Equipment Manuals" binders located in the Sample Receiving area.

6.3.3 Instrument Supplies

Refer to BU-TM-1102 PCN, BU-TM-1105 PCB Congeners, or BU-TM-1107 PCDD/F.

6.3.4 Standard Preparation Supplies

Refer to BU-TM-1102 PCN, BU-TM-1105 PCB Congeners, or BU-TM-1107 PCDD/F.

7.0 REAGENTS AND STANDARDS

Refer to BU-SP-0250 Control of Reagents and Reference Materials for reagent handling protocols. Unless otherwise specified, all reagents are verified on a batch-by-batch basis through an assessment of Method QC. Refer to Section 8.1 for a description of Method QC performed.

7.1 Reagents

7.1.1 Toluene (Distilled in glass). Purchased from Caledon. MEOL# 601007, Expires 1 year after date received

7.1.2 Hexane (Distilled in glass). Purchased from Fisher. MEOL# 6010005, Expires 1 year after date received

7.1.3 Dichloromethane (Distilled in glass). Purchased from Caledon. MEOL# 6010024, Expires 1 year after date received

7.1.4 Acetone (Distilled in glass). Purchased from Caledon. MEOL# 6010003, Expires 1 year after date received

7.1.5 Nonane (Distilled in glass). Purchased from Caledon. MEOL# 6010102 - Expires 1 year from date received.

7.1.6 80:20 Toluene:Acetone

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a) Prepare a 5:1 solution of Toluene/Acetone in 1 L clear jar. This for cleaning only and does not need to be prepared quantitatively.
b) Do not re-use/store. Dispose of after use.

7.1.7 Sodium Sulfate. A.C.S. Grade, Anhydrous Powder. Purchased from Caledon. Cat # 8220-1-90, MEOL# 6031412 (Sigma-Aldrich), MEOL# 6030433 (Caledon), Expires 1 year after date received

7.1.7.1 Place the desired amount of sodium sulphate in to a quartz beaker.
7.1.7.2 Bake at 450˚C for a minimum of 8 hours.
7.1.7.3 Store in an air-tight jar at room temperature for up to 2 years.
7.1.7.4 Record preparation into the Reagent Binder, label ORG-SSU-###.

7.1.8 Quartz Sand. White-Quartz -50+70 Mesh. Purchased from Sigma-Aldrich. Cat # 274739-5KG.

7.1.9 Baked Quartz Sand

7.1.9.1 Place the desired amount of quartz sand in to a quartz beaker.
7.1.9.2 Bake at 450˚C for a minimum of 8 hours.
7.1.9.3 Store in an air-tight jar at room temperature for up to 2 years.
7.1.9.4 Record preparation into the Reagent Binder, label ORG-QSA-###.

7.1.10 Sodium Chloride. Reagent Grade. Purchased from Fisher. Cat # S642-500.

7.1.11 Reagent Water. Ultra-pure (reverse-osmosis, de-ionized, carbon filtrated). Refer to BU-SP-0253 Laboratory Water for details.

7.1.12 Potassium Hydroxide Pellets. A.C.S. Grade. Purchased from Fisher Scientific. Cat # P250-3

7.1.13 9:1 KOH:MeOH

7.1.13.1 Measure out at 336.0 g ±0.5 g of the KOH pellets into a pre-tared beaker using a two point balance and record to two decimal places.
7.1.13.2 Mix the KOH pellets with 300 mL of DI water in a large Erlenmeyer, measured using a to-deliver, class A graduated cylinder.
7.1.13.3 Once the pellets have completely dissolved transfer this mixture to a 4 L amber bottle containing 2700 mL of Methanol, measured using a to deliver, class A graduated cylinder.
7.1.13.4 Shake well and vent several times, let stand to cool in fume hood prior to use.
7.1.13.5 Store for 3 months at room temperature.
7.1.13.6 Record preparation into the Reagent Binder, label ORG-KOH:MeOH-###.

7.1.14 Corn Oil. Mazola Brand, Purchased from the grocery store.

7.2 Standards

The following standards are used during the extraction procedure:

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Dioxins:

Standard Type	Volume per split (µL)	Volume per Portion (µL)	Name
Extraction in Air	20		M23-ES#2-xxx
Extraction in Edible Oil	20		DX17-ES#2-xxx
Extraction	20		1613B-ES#1-xxx
Native	20		1613B-NS#3-xxx
Cleanup	20		1613B-CL#2-xxx
Injection		20	1613B-IS#1-xxx
Injection in Edible Oil		15	DX5-IS#1-xxx
Cleanup in Air	20		M23-CL#1-xxx

PCBs:

Standard Type	Volume per split (µL)	Volume per Portion (µL)	Name
Extraction	20		1668A-ES#2-xxx
Native	20		1668A-NS#1-xxx
Cleanup	20		1668A-CL#2-xxx
Injection		5	1668A-IS#2-xxx
Injection in Edible Oil		5	1668A-IS#2-4x Diluted-xxx

PCNs:

Standard Type	Volume per split (µL)	Volume per Portion (µL)	Name
Extraction	20		PCN-ES#3-xxx
Native	20		PCN-NS#4-xxx
Injection		5	DXPCB-IS#1-xxx

Syringe tolerance is 2-5%. All standards are prepared by the instrument analysts. Refer to the appropriate instrument test method for standard details.

It is the organic prep department supervisor's responsibility to maintain up-to-date communication with the instrument analysts regarding the most current standards in use. This information is added to each preparative batch sheet before it is released to the prep staff.

For protocols for the identification and control, handling, witnessing and spiking of standards refer to BU-WI-3002 Standards Prep, Spiking, and Witnessing.

8.0 QUALITY CONTROL

8.1 Method QC

Maximum (Sample Preparation) Batch Size =	20 client samples

Method QC	Min. Frequency	Data Quality Objective (units)
Method Blank (MB)	1 per batch	See instrument method

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Laboratory Control Sample (LCS or Clean Matrix Spike) or On-going Precision and Recovery (OPR)	1 per batch	See instrument method
Reference Material (RM)	Upon Client Request	See instrument method
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)1	Upon Client Request	See instrument method
Extraction Standards (SURR)	All samples and QC samples	See instrument method
Clean-up Standard	All samples and QC Samples	See instrument method
Sampling Standard	Air: all samples spiked prior to submission of sampling apparatus to client	See instrument method
Sample Duplicate	Solids: 1 per batch Air: N/A Water: 1 per batch, or as specified in contract or client request	See instrument method
Sample Duplicate – Quebec DX Waters	If 1-10 samples in batch, no duplicate required; 11-20 samples in batch, LCS duplicate required	See instrument method

QC Matrices	
Matrix	**Preparation Details**
Soil	20 grams of sand ± 2 grams
Water	1L of DI Water ± 20 mL
Tissue - Soxhlet	20 grams of sodium sulphate ± 2 grams
Edible Oils	10 grams of Corn Oil ±2 grams
Tissue – Digestion	80 grams of Corn Oil ±2 grams
Stack	30 grams of XAD from the same media prep ± 2 grams

9.0 TEST PROCEDURES

9.1 Extraction and Extract Purification Overview

Note: Not all of the steps in the Extract Purification Procedures are required for all matrices. The decision on which purification steps are required will be made based on matrix type, matrix history, and sample assessment. These decisions will be finalized (by management) and detailed on the preparative batch sheets (by the organic prep department supervisor) prior to sample release to prep staff.

9.1.1 Extraction Procedures Overview

Matrix	Target Analysis	Subsample Size	Pre-Extraction Treatment	Section	Extraction/Typical CleanupMethod[1]	Section

1 For DoD analysis an MS/MSD is required at a minimum for each batch of samples where there is sample available. An LCS Dup is recommended in absence of sufficient sample for MS/MSD.

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Soils & Sediments	PCDD/F	10g Dry Equivalent	Mixed with Quartz Sand	9.3.1.3e)	Dean Stark Soxhlet in Toluene/CU/LS/FL	9.3.1.3
Soils & Sediments	PCB	10g Dry Equivalent	Mixed with Quartz Sand	9.3.1.3 e)	DeanStark Soxhlet in Toluene/CU/LS/AL	9.3.1.3
Soils & Sediments	PCDD/F + Toxic PCB	10g Dry Equivalent	Mixed with Quartz Sand	9.3.1.3 e)	DeanStark Soxhlet in Toluene/CU/LS/CC	9.3.1.3
Water No Visible Solids	PCDD/F	1L	none		Liquid/Liquid with DCM/LS/FL	9.3.2 and 9.3.3
Drinking Water (MOE)	PCDD/F	1L	None – do NOT filter		Liquid/Liquid with DCM/LS/FL	9.3.2 and 9.3.3
Water Visible Solids	PCDD/F	1L	Filter	9.3.4	Soxhlet Filter with Toluene Liquid/Liquid Filtrate with DCM/LS/FL	9.3.2 and 9.3.4
Water Less than 1% Solids	PCB	1L	none		Liquid/Liquid with DCM/LS/AL	9.3.2 and 9.3.3
Water Greater than 1% Solids	PCB	1L	Filter	9.3.4	Soxhlet Filter with Toluene Liquid/Liquid Filtrate with DCM/LS/AL	9.3.2 and 9.3.4
Water Less than 1% Solids	PCDD/F + Toxic PCB	1L	none		Liquid/Liquid with DCM/LS/CC	9.3.2 and 9.3.3
Water Greater than 1% Solids	PCDD/F + Toxic PCB	1L	Filter	9.3.4	Soxhlet Filter with Toluene Liquid/Liquid Filtrate with DCM/LS/CC	9.3.2 and 9.3.4
Ambient Air	PCDD/F	Entire Sample	none		Soxhlet PUF and Filter with Toluene/LS/FL	9.3.5
Stack Effluent	PCDD/F	Entire Sample	none		Filter/XAD Dean Stark Soxhlet in Toluene Impinger contents Liquid/Liquid with DCM/LS/FL	9.3.6
Ambient Air	PCB	Entire Sample	none		Soxhlet PUF and Filter with Toluene/LS/AL	9.3.5
Stack Effluent	PCB	Entire Sample	none		Filter/XAD Dean Stark Soxhlet in Toluene Impinger Contents Liquid/Liquid with DCM/LS/AL	9.3.6
Ambient Air	PCDD/F + Toxic PCB	Entire Sample	none		Soxhlet PUF and Filter with Toluene/LS/CC	9.3.5
Stack Effluent	PCDD/F + Toxic PCB	Entire Sample	none		Filter/XAD Dean Stark Soxhlet in Toluene Impinger Contents Liquid/Liquid with DCM/LS/CC	9.3.6
Waste Materials - Liquid	PCDD/F	1L	none		Liquid/Liquid with DCM/LS/FL	9.3.2 and 9.3.3
Waste Materials - Solids	PCDD/F	10g	Dried with Quartz Sand	9.3.1.3 e)	Routine Dean Stark Soxhlet in Toluene/LS/FL	
Waste Materials - Liquid	PCB	1L	none		Liquid/Liquid with DCM/LS/AL	9.3.2 and 9.3.3
Waste Materials - Solids	PCB	10g	Dried with Quartz Sand	9.3.1.3 e)	Routine Dean Stark Soxhlet in Toluene/LS/AL	
Waste Materials - Liquid	PCDD/F + Toxic PCB	1L	none		Liquid/Liquid with DCM/LS/CC	9.3.2 and 9.3.3

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Waste Materials - Solids	PCDD/F + Toxic PCB	10g	Dried with Quartz Sand	9.3.1.3 e)	Routine Dean Stark Soxhlet in Toluene/LS/CC	
Meat, Fish, Eggs, Cereals, Feeds, Solid Food Additives, other Biota & Tissues	Soxhlet Option: PCDD/F	1g lipid	Dried with Sodium Sulphate	9.3.1.3 e)	Routine Soxhlet in DCM/AW/LS/FL	9.3.1.3
Meat, Fish, Eggs, Cereals, Feeds, Solid Food Additives, other Biota & Tissues	Soxhlet Option: PCB	1g lipid	Dried with Sodium Sulphate	9.3.1.3 e)	Routine Soxhlet in DCM/AW/LS/AL	9.3.1.3
Meat, Fish, Eggs, Cereals, Feeds, Solid Food Additives, other Biota & Tissues	Soxhlet Option: PCDD/F + Toxic PCB	1g lipid	Dried with Sodium Sulphate	9.3.1.3 e)	Routine Soxhlet in DCM/AW/LS/CC	9.3.1.3
Meat, Fish, Eggs, Cereals, Feeds, Solid, Dairy, Edible Oils, other Biota & Tissues	Base Digestion Option: PCDD/F or PCB	80g	none		Base Digestion – methanolic KOH/AW/LS/AL	See BU-TP-2170 Base Digestion
Serum (See BU-TP-1110 for Serum SOP)	Soxhlet Option: PCDD/F, PBDE + Toxic PCB	5g -20g	none		Liquid/Liquid extraction in Hexane /LS/AL	See BU-TP-1110 for Serum SOP
Food Additives - Liquid	PCDD/F	30g dry equivalent	none		Liquid/Liquid with DCM/LS/FL	9.3.2 and 9.3.3
Food Additives - Liquid	PCB	30g dry equivalent	none		Liquid/Liquid with DCM/LS/AL	9.3.2 and 9.3.3
Food Additives - Liquid	PCDD/F + Toxic PCB	30g dry equivalent	none		Liquid/Liquid with DCM/LS/CC	9.3.2 and 9.3.3
Butter & Edible Oils	PCDD/F	10g	none		Dissolve in Hexane/AW/LS/FL	9.3.7
Butter & Edible Oils	PCB	10g	none		Dissolve in Hexane/AW/LS/AL	9.3.7
Butter & Edible Oils	PCDD/F + Toxic PCB	10g	none		Dissolve in Hexane/AW/LS/CC	9.3.7

[1]Cleanup methods:

CU	Copper Treatment	BU-WI-3023 Copper Treatment
AL	Alumina	BU-WI-3011 Alumina Column
AW	Acid washing	
CC	Chromacarb	BU-WI-3006 Chromacarb
LS	Layered Silica (Acid Silica)	BU-WI-3016 Acid Silica Column Preparation and Use
FL	Florisil	BU-WI-3018 Florisil Column

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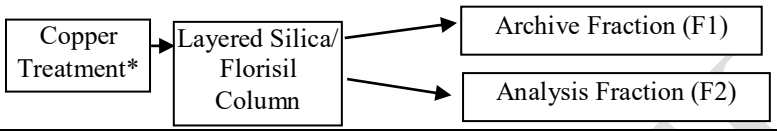
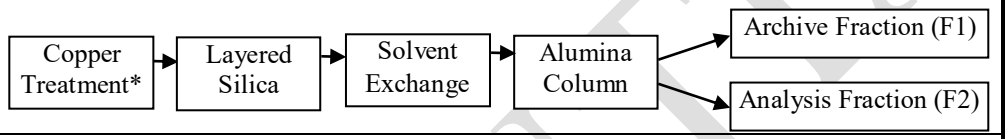
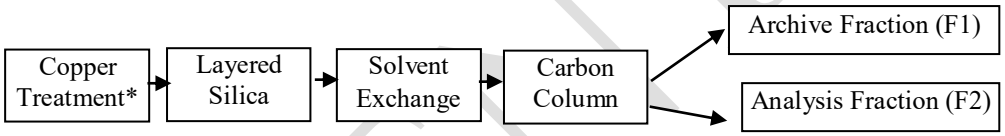
9.1.2 Extract Purification Procedures Overview

Target Analysis	Cleanup and Reduction Stages
PCDD/F only	
PCDD/F, PCB or PBDE	
Both PCDD/F & Coplanar PCB, PCN	

*Copper treatment can be performed before or after column cleanups

9.1.2.1 Gel Permeation Chromatography

Tissues generally require GPC cleanup to remove lipid interferences. Other solids such as contaminated soils and sediments can sometimes benefit from GPC cleanup. For GPC cleanup protocols, please see refer to BU-WI-3017 Gel Permeation Chromatography. GPC cleanup if required, is recommended before other column chromatographic clean-ups

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9.1.3 Handling of Multi-Phase Samples

Please see BU-WI-3001 Subsampling and Homogenization of Samples for Organic Analysis, if applicable.

All targets covered by this method are highly organic soluble and therefore are predominantly found in organic phases. Similarly, the targets are poorly dissolved in aqueous phases. Exceptions on water solubility may occur when there is significant dissolved organic content in the aqueous phase.

When faced with a multi-phase sample, always stop processing and contact your Supervisor before proceeding with the following options:

9.1.3.1 For solids, sediments and soils where there is significant standing water in the sample container, the standing water may be decanted prior to analysis. This must be done in a manner to avoid significant losses of any solid 'fines'. Preparation then proceeds as detailed in Section 9.3.1.

9.1.3.2 The preparation of turbid water samples (< 1% solids) is detailed in Section 9.3.3. Water samples with higher than approximately 1% solids are treated as detailed in Section 9.3.4.

9.1.3.3 Water samples with visible oil but with insufficient oil to separate into an organic phase are handled as whole water samples. Sub-sampling should be avoided if practical but when necessary must be rigorously shaken or otherwise homogenized to ensure representative sub-sampling

9.1.3.4 Other mixed phase samples can be challenging. Consultation with a supervisor and/or customer service is often appropriate and recommended.

9.1.3.5 Two options for the handling of other mixed phase samples are presented below. If option (b) is deemed appropriate, then this must be done in consultation with the client to ensure that the client's project design is not compromised or at least compromised at a minimum. Option (a) may proceed without consultation with the client assuming that this can be accomplished without significant compromise to the project designed data quality objectives.

a) Analysis of a sub-sample of homogenized sample
b) Separation of the phases and analysis of one or more of the appropriate phases (for this option a determination of the relative amounts of the individual phases may be required).

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9.2 General Procedures

9.2.1 Glassware Cleaning

9.2.1.1 General Glassware and Utensils Cleaning

Wash and solvent rinse all glassware and utensils according to BU-WI-3000 Organic Glassware/Equipment Cleaning Proofing and Maintenance. Any high concentration samples processed should have their glassware segregated as per BU-WI-3000 Organic Glassware/Equipment Cleaning Proofing and Maintenance and be treated as per the High-Level Glassware Treatment section.

9.2.1.2 Pre-Extracting Soxhlet Glassware

Wash and solvent rinse all soxhlet apparatus, thimble, and 500 mL florence flasks as outlined in BU-WI-3000 Organic Glassware/Equipment Cleaning Proofing and Maintenance. Section 2.2 Pre-Soxhlet Procedure of BU-WI-3003 Soxhlet Setup Procedure should be completed prior to extracting samples.

9.2.1.3 Preparing Separatory Funnels

Wash and solvent rinse a 2L separatory funnel as outlined in BU-WI-3000 Organic Glassware/Equipment Cleaning Proofing and Maintenance. Also perform the following:

a) Sonicate the stopper and stopcock in 80:20 Toluene:Acetone for 15 minutes, decant off the solvent mixture and perform a hexane shake in the sonicating jar.
b) Assemble the separatory funnel and place it into a ring stand.
c) Rinse the separatory funnel with a DCM shakes of approximately 100 mL for 15 seconds. Discard the DCM rinse.

9.2.2 Subsampling and Homogenization

For protocols on homogenization and subsampling refer to BU-WI-3001 Subsampling and Homogenization of Samples for Organic Analysis and BU-WI-3005 Food Sample Homogenization.

9.3 Sample Extraction Procedures

9.3.1 Soxhlet Extraction of Solids, Sediments, Meat, Fish, Eggs, Cereals, Feeds, Solid Food Additives, other Biota & Tissues:

Note: Please refer to BU-SP-0018 International Soil Handling before prepping sample, if applicable.

9.3.1.1 % Moisture Determination

Refer to BU-TM-1200 Moisture Content by Gravimetry

9.3.1.2 % Lipid Determination

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Refer to BU-TM-1700 Lipids

9.3.1.3 Routine Soxhlet Extraction and Dean-Stark Soxhlet Extraction

a) Method Blank (MB)

i. Prepare a separate MB for each set up of 20 client samples.
ii. Subsample as described in Section 9.3.1.3e "Drying Samples with Sand/Sodium Sulphate".

b) Laboratory Control Sample (LCS)

i. Prepare a separate LCS for each set up of 20 client samples.
ii. Subsample as described in Section 9.3.1.3e "Drying Samples with Sand/Sodium Sulphate".

c) Duplicate Sample (DUP)

i. Prepare a duplicate for each batch consisting of 4 or more samples or one every 5th batch when these batches contain less than 4 samples.
ii. Subsample duplicate as described in Section 9.3.1.3d "Percent Moisture Determination" and 9.3.1.3e "Drying Samples with Sodium Sulphate".

d) Percent (%) Moisture Determination

i. Refer to BU-TM-1200 Moisture Content by Gravimetry

e) Drying Samples with Sand/Sodium Sulphate

i. Sodium Sulphate is to be used for Routine Soxhlet extraction. Quartz Sand is to be used for Dean-Stark Soxhlet Extraction.
ii. Place the sample jar on to a two-place balance, and tare the balance
iii. Subsample the amount of sample as required on the batch sheet ± 1.0 g (if sampling less than 10 grams defer to the Supervisor for acceptable subsampling range criteria) into the jar and record the weight of the subsample to two decimal places. Refer to BU-WI-3001 Subsampling and Homogenization of Samples for Organic Analysis for proper sub-sampling and homogenization procedures and BU-WI-3005 Food Sample Homogenization for food prep instructions.
iv. Thoroughly mix the sample and sand or sodium sulphate (refer to batch sheet for the appropriate packing material to use) mixture with a spatula.
v. Add additional sand or sodium sulphate and mixing until a dry, free-flowing mixture is obtained.

f) Soxhlet Extraction

i. Refer to BU-WI-3003 Soxhlet Setup Procedure for the proper setup of the soxhlet extraction apparatus.
ii. Transfer sample to pre-Soxhlet-extracted cellulose thimble.
iii. Spike, using the appropriate syringe and volume of Extraction Standard (refer to Section 7.2), on to the samples and QC samples. Also, spike the Native Standard into LCS and MS (if required) as per Section 7.2 "Standards"

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NOTE: For pulp mill samples the sample must soak in ethanol overnight prior to proceeding to the next steps. This is done by placing the sample into a soxhlet body, filling the soxhlet body with ethanol to just below the siphon arm. After the soaking is complete, the ethanol is drained into the extraction flask and is used for the sample extraction.

iv. Soxhlet extract sample with the appropriate solvent (Refer to BU-WI-3003 Soxhlet Setup Procedure) for a minimum time of 16 hours. Heat Setting = Medium or less.
NOTE: For soils and sediments extract for 16 hours in Toluene. For tissues, food and feed extract for 16 hours in DCM. For NDMA soils extract for 16 hours in DCM. For ash samples soxhlet extract for 48 hours in toluene followed by 16 hours in DCM with 5 drops of trifluoroacetic acid. For pulp mill samples extract for 16 hours in Toluene after the previously mentioned ethanol soak.

v. Turn the soxhlet off and allow it to cool to room temperature.

vi. Proceed to the Section 9.4 Extract Isolation and Purification Procedures.

9.3.1.4 Extraction for Serum Samples

a) Please refer to BU-TP-1110 PCDD/F, BDPE & PCB HRMS Prep Method for Serum Samples.

9.3.2 Preparation for Extraction of Aqueous Samples

9.3.2.1 Temperature Adjustment of Sample

a) Check that the sample has the label associated with Water Preservation and pH check. If this is not present stop and consult Supervisor or Team Lead.

b) Place the samples on the bench, away from direct heat sources or sunlight.

c) Allow the bottles to come up to ambient room temperature before proceeding with extraction.

9.3.2.2 Mark the Sample Level on the Original Bottle

a) Do not perform this step until the sample has come up to room temperature.

b) Using a fine-point permanent marker, mark the line of the sample meniscus on the bottle.

c) Once the sample has been removed from the bottle, the extraction steps are completed (9.3.3 or 9.3.4), and the residual DCM rinse has evaporated, fill the bottle to the line with tap water.

d) Transfer the tap water to a graduated cylinder. Record the volume on the sample batch sheet

9.3.2.3 Preparing Spike Extraction Standard into 1 mL Acetone

a) Pipette, using a disposable glass pipette, ~1 mL of Acetone into a 15 mL disposable C-tube, this is not quantitative.

b) Spike with a syringe, using the appropriate volume of Extraction Standard, as indicated on the preparative batch sheet into the c-tube containing the acetone. Refer to BU-WI-3002 Standards Prep, Spiking and Witnessing for proper protocols.

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c) Vortex the C-tube for 15 seconds.

d) Quantitatively transfer the spiked acetone into the sample bottle containing the sample with 2x 1mL acetone rinses. Cap the bottle and gently shake to mix.

e) Method Blank (MB)
   i. Prepare a separate MB for each set of up to 20 client samples.
   ii. Measure 1.0 L ±0.2 L of organic-free water using a graduated beaker and place into a 2 L separatory funnel. Record volume on batch sheet.
   iii. Quantitatively transfer the spiked acetone into the separatory funnel with 2x 1mL acetone rinses.
   iv. Proceed to Section 9.3.3 or 9.3.4 where appropriate.

f) Laboratory Control Sample (LCS)
   i. Prepare a separate LCS for each set of up to 20 client samples.
   ii. Measure 1.0 L ±0.2 L of organic-free water using a graduated beaker and place into a 2 L separatory funnel. Record volume on batch sheet.
   iii. Quantitatively transfer the spiked acetone into the separatory funnel with 2x 1mL acetone rinses
   iv. Proceed to Section 9.3.3 or 9.3.4 where appropriate.

9.3.2.4 Prepare Allihn Filters with Sodium Sulphate

a) Wash and solvent rinse an Allihn filter and a 500 mL Florence flask, as outlined in BU-WI-3000 Organic Glassware/Equipment Cleaning, Proofing, and Maintenance.
b) Place the Allihn filter on to the 500mL Florence flask.
c) Fill the Allihn filter half full with sodium sulphate.
d) Rinse the Allihn filter containing the sodium sulphate with at least 50 mL DCM, discarding the DCM from the Florence flask.
e) Cover the top of the Allihn filter with clean foil until needed.

9.3.3 Extraction of Liquids Containing Less than 1% Solids (Containing no Visible Solids)

a) Pour the entire contents of the sample bottle (1L) in to a 2L separatory funnel. Be sure that the sample level has been marked on the bottle before the sample is transferred to the separatory funnel.

b) Add 100 mL ± 5 mL of DCM, measured using a glass graduated beaker, to the original sample bottle and shake for 30 seconds, then add this to the separatory funnel. Place the empty original sample container into the fume hood. This will be used to determine the original volume of the sample.

c) Vigorously shake the separatory funnel for 2 minutes, with periodic venting.

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d) Allow the sample to sit to allow for good phase separation.

e) If good phase separation cannot be achieved after 10 minutes, a small amount of sodium chloride can be added to help break the emulsion. If this is performed, this must also be done to the QC samples.

f) Drain the DCM layer (bottom layer) from the separatory funnel, through the Allihn filter containing sodium sulphate, in to a 500 mL Florence flask.

g) Using 100 mL ± 5 mL of DCM, measured using a glass graduated beaker, repeat the extraction steps two more times. If sample analysis is for DX, PCB, or DX/PCB extracting for 30 seconds for the second and third round. All other analyses or combination of analyses extract for 2 min each round.

h) Using 50 mL ± 5 mL of DCM using a glass graduated beaker, rinse the sodium sulphate and Allihn filter, adding it to the previously collected extract.

i) Proceed to Section 9.4 Extract Isolation and Purification Procedures.

9.3.4 Extraction of Waters Containing Visible Solids

NOTE: Drinking water (MOE) samples are **never** filtered prior to analysis. Unfiltered samples for the measurement of organic compounds are important because many organic compounds adsorb to particulates present in the water sample. If a drinking water sample looks to contain greater than 1% solids, please notify your Supervisor.

a) If the sample contains visible solids, it must be drained through a glass wool filter first. The glass wool is sonicated in DCM prior to use and placed on a funnel. The sample is filtered through the glass wool and collected in a second bottle. Be sure to mark the sample level on the bottle before transferring. This step is also performed for the Method Blank.

b) The original sample bottle is rinsed using 100 mL ± 5 mL of DCM, and added to the separatory funnel through the glass wool filter.

c) Allow the glass wool to air-dry for a minimum of two hours, and then place it in to a pre-extracted soxhlet containing a 1cm layer of glass wool.

d) Soxhlet extract the glass wool with toluene via Dean-stark for a minimum time of 16 hours. Heat Setting = High. Refer to BU-WI-3003 Soxhlet Setup Procedure for the proper setup of the soxhlet extraction apparatus.

e) Pour the entire filtrate in to a 2L separatory funnel.

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f) Shake 100 mL ± 5 mL of DCM, measured using a glass graduated beaker, in the original sample bottle for 30 seconds, and then add this to the separatory funnel. Place the empty original sample container into the fume hood. This will be used to determine the original volume of the sample.

g) Vigorously shake the separatory funnel for 2 minutes, with periodic venting.

h) Allow the sample to sit to allow for good phase separation.

i) If good phase separation cannot be achieved after 10 minutes, a small amount of sodium chloride can be added to help break the emulsion.

j) Drain the DCM layer (bottom layer) from the separatory funnel, through the Allihn filter containing sodium sulphate, in to a 500 mL florence flask.

k) Using 100 mL ± 5 mL of DCM, measured using a glass graduated beaker, repeat the extraction steps two more times. If sample analysis is for DX, PCB, or DX/PCB extracting for 30 seconds for the second and third round. All other analyses or combination of analyses extract for 2 min each round.

l) Using 50 mL ± 5 mL of DCM, measured using a glass graduated beaker, rinse the sodium sulphate and the Allihn filter, adding it to the previously collected extract.

m) Reduce the filtrate extract on the rotovap. Once the level becomes low enough in the Florence flask, combine the Filter extract to the flask.

n) Proceed to Section 9.4 Extract Isolation and Purification Procedures.

9.3.5 Extraction of Ambient Air Samples

a) Place 1cm of clean glass wool into the bottom of a soxhlet body.

b) Place 1cm of sodium sulphate on to the glass wool layer.

c) Method Blank (MB)
   i. Prepare a separate MB for each set of up to 20 client samples.
   ii. MB consists of a puf from the same lot as the samples and/or 30 grams ± 2 grams of the same lot of XAD as the samples, along with one filter from the same lot as the samples.

d) Laboratory Control Sample (LCS)
   i. Prepare a separate LCS for each set of up to 20 client samples.
   ii. LCS consists of a puf from the same lot as the samples and/or 30 grams ± 2 grams of the same lot of XAD that the samples contain, along with one filter from the same lot as the.

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Note: Be sure to carefully check client labels to avoid sample cross-contamination.

e) Transfer the PUF, and/or XAD, and/or Filter to the soxhlet body. Record media prep L# for each sample in the appropriate section of the batch sheet.

f) Rinse the inside surface of the PUF cartridge in to the soxhlet body with acetone.

g) Spike the appropriate volume of Extraction Standard, as indicated on the batch sheet, on to the sample. Refer to BU-WI-3002 Standards Prep, Spiking, and Witnessing for proper spiking procedures.

h) Soxhlet extract sample using Toluene for a minimum time of 16 hours. Heat Setting = High. Refer to BU-WI-3003 Soxhlet Set-up Procedure for the proper setup of the soxhlet extraction apparatus.

i) Proceed to Section 9.4 Extract Isolation and Purification Procedures.

9.3.6 Extraction of Stack Effluent Samples

Note: Stack Effluent Samples may be analyzed in one of two ways:
The Front and Back halves may be combined into a single sample, or each half may be analyzed as separate samples. The protocol to be followed will come from the client, and will be clearly reflected by the manner in which the samples will be logged in to the LIMS system. The front half and Back-Half are rinses collected in the field from train glassware. The Front-Half rinse is of the filter housing, and the Back-Half rinse is of the impingers.

9.3.6.1 Combined Front-Half and Back-Half Extraction

1. Transfer the Front-Half rinse to a Florence flask. Rinse the original container with 3x10 mL of Toluene, shaking each rinse in the bottle and then adding it to the Florence flask.

2. Reduce the Front-Half rinse down to approximately 2 mL on a rotary-evaporator. Refer to BU-WI-3009 Rotary Evaporators for the proper use.

3. Refer to BU-WI-3003 Soxhlet Set-up Procedure for pre-cleaning apparatus.

4. Place 1cm of clean glass wool into the bottom of a soxhlet body.

5. Place 1cm of sodium sulphate on to the glass wool layer.

6. Method Blank (MB)
   i. Prepare a separate MB for each set of up to 20 client samples.

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ii. The MB consists of 30 grams ± 2 grams of the same lot of XAD that the samples contain. Along with one filter from the same lot of filters that the samples contain.

7. Laboratory Control Sample (LCS)
   i. Prepare a separate LCS for each set of up to 20 client samples.
   ii. The LCS consists of 30 grams ± 2 grams of the same lot of XAD that the samples contain. Along with one filter from the same lot of filters that the samples contain.

Note: Be sure to carefully check client labels to avoid sample cross-contamination.

8. Place the Filter on top of the sodium sulphate layer.

Note: Be sure to carefully check client labels to avoid sample cross-contamination.

9. Transfer the XAD from sorbent trap to the soxhlet.

Note: Be sure to carefully check client labels to avoid sample cross-contamination.

10. Rinse the trap in to the soxhlet with Toluene.

Note: Record media prep L# for each sample in the appropriate section of the batch sheet.

11. Transfer the reduced Front-Half rinse to the soxhlet with 3x1 mL Toluene rinses.

12. Spike the appropriate volume of Extraction Standard, as indicated on the batch sheet, on to the sample. Refer to BU-WI-3002 Standards Prep, Spiking, and Witnessing for proper spiking procedures.

13. Soxhlet extract sample using Toluene for a minimum time of 16 hours. Heat Setting = High. Refer to Refer to BU-WI-3003 Soxhlet Set-up Procedure for the proper setup of the soxhlet extraction apparatus.

14. Reduce the extract down to approximately 10 mL on a rotary-evaporator. Refer to BU-WI-3009 Rotary Evaporators for the proper use.

15. Transfer the Back-Half rinse to a Florence flask. Rinse the original container with 3x10 mL of Toluene, shaking each rinse in the bottle and then adding it to the Florence flask

16. Reduce the Back-Half rinse down to approximately 2 mL on a rotary-evaporator. Refer to BU-WI-3009 Rotary Evaporators for the proper use of the rotary evaporator.

17. Set up for a separatory funnel extraction.

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a. Wash and solvent rinse a 2L separatory funnel as outlined in BU-WI-3000 Organic Glassware/Equipment Cleaning, Proofing, and Maintenance.
b. Sonicate the stopper and stopcock in 80:20 Toluene:Acetone for 15 minutes, followed by a rinse with hexane
c. Assemble the separatory funnel and place it into a ring stand

18. Prepare Allihn Filters with Sodium Sulphate
    a. Wash and solvent rinse an Allihn filter and a 500 mL florence flask, as outlined in BU-WI-3000 Organic Glassware/Equipment Cleaning, Proofing, and Maintenance
    b. Place the Allihn filter on to the 500 mL florence flask
    c. Fill the Allihn filter half full with sodium sulphate
    d. Rinse the Allihn filter containing the sodium sulphate with 1 x 50 mL DCM, discarding the DCM from the florence flask after the rinse
    e. Cover the top of the Allihn filter with clean foil until needed

19. Prepare Method Blank (MB)
    a. Place 1L ± 0.2 L of organic-free water using a 1000mL graduated cylinder to a 2L separatory funnel

20. Pour Impinger contents into prepared separatory funnels.
    Note: Be sure to carefully check client labels to avoid sample cross-contamination.
21. Top up sample with organic free water to approximately 1L.
22. Transfer the reduced Back Half rinse to the soxhlet extractor with 3x1mL DCM rinses.
23. Pour 100mL ± 5 mL of DCM into separatory funnel
24. Cap the separatory funnel with a clean stopper ensuring a good seal
25. Vigorously shake the separatory funnel manually for 2 minutes, with periodic venting
26. If good phase separation cannot be achieved, a small amount of sodium chloride can be added to the separatory funnel to help break the emulsion. If this approach is taken the Method Blank should be treated with the same amount of sodium chloride.
27. Drain the DCM layer (bottom layer) from the separatory funnel, through the Allihn filter containing sodium sulphate, in to a 500 mL florence flask.
28. Using 100 mL ± 5 mL of DCM, measured using a glass graduated beaker, repeat steps 6 to 10 two more times
29. Using 50 mL ± 5 mL DCM using a glass graduated beaker, rinse the sodium sulphate and the Allihn filter, adding it to the previously collected extract
30. Combine Back Half extraction (separatory funnel shake) with Front Half extraction (soxhlet extraction).

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31. Proceed to Section 9.4 Extract Isolation and Purification Procedures

9.3.6.2 Separate Front-Half Extraction (if requested by client)

a) Transfer the Front Half rinse to a Florence flask. Rinse the original container with 3x10 mL of toluene, shaking each rinse in the bottle and then adding it to the Florence flask.

b) Reduce the Front-Half rinse down to approximately 2 mL on a rotary-evaporator. Refer to BU-WI-3009 Rotary Evaporators for the proper use of the rotary evaporator.

c) Place 1cm of clean glass wool into the bottom of a soxhlet body.

d) Place 1cm of sodium sulphate on to the glass wool layer.

e) Method Blank (MB)
   i. Prepare a separate MB for each set of up to 20 client samples.
   ii. MB consists of 30 grams ± 2 grams of the same lot of XAD that the samples contain. Along with one filter from the same lot of filters that the samples contain.

f) Laboratory Control Sample (LCS)
   i. Prepare a separate LCS for each set of up to 20 client samples.
   ii. The LCS consists of 30 grams ± 2 grams of the same lot of XAD that the samples contain. Along with one filter from the same lot of filters that the samples contain.

Note: Be sure to carefully check client labels to avoid sample cross-contamination.

g) Place the Filter on top of the sodium sulphate layer.

Note: Be sure to carefully check client labels to avoid sample cross-contamination.

h) Transfer the reduced Front-Half rinse to the soxhlet extractor with 3x1mL toluene rinses.

i) Spike the appropriate volume of Extraction Standard, as indicated on the batch sheet, on to the sample. Refer to BU-WI-3002 Standards Prep, Spiking, and Witnessing for proper spiking procedures.

j) Soxhlet extract sample with toluene for a minimum time of 16 hours. Heat Setting = High. Refer to BU-WI-3003 Soxhlet Set-up Procedure for the proper setup of the soxhlet extraction apparatus.

k) Turn the soxhlet off and allow it to cool for a minimum of one hour.

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l) Remove the condenser.

m) Reduce the extract down to approximately 2 mL on a rotary-evaporator. Refer to BU-WI-3009 Rotary Evaporators for the proper use.

n) Proceed to Section 9.4 Extract Isolation and Purification Procedures.

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9.3.6.3 Separate Back-Half Extraction

a) Place 1 cm of clean glass wool into the bottom of a soxhlet body.

b) Place 1 cm of sodium sulphate on to the glass wool layer.

c) Method Blank (MB)
   i. Prepare a separate MB for each set of up to 20 client samples.
   ii. MB consists of 30 grams ± 2 grams of the same lot of XAD that the samples contain, along with one filter from the same lot of filters that the samples contain.

d) Laboratory Control Sample (LCS)
   i. Prepare a separate LCS for each set of up to 20 client samples.
   ii. 30 grams ± 2 grams of the same lot of XAD the samples contain is used. Along with one filter from the same lot of filters the samples contain is used.

   Note: Be sure to carefully check client labels to avoid sample cross-contamination.

e) Transfer the XAD from sorbent trap to the soxhlet body.

   Note: Be sure to carefully check client labels to avoid sample cross-contamination.

f) Rinse the trap in to the soxhlet with toluene.

g) Spike the appropriate volume of Extraction Standard, as indicated on the batch sheet, on to the sample. Refer to BU-WI-3002 Standards Prep, Spiking, and Witnessing for proper spiking procedures.

h) Soxhlet extract sample with toluene for a minimum time of 16 hours. Heat Setting = High. Refer to BU-WI-3003 Soxhlet Set-up Procedure for the proper setup of the soxhlet extraction apparatus.

i) Turn the soxhlet off and allow it to cool for a minimum of one hour.

j) Remove the condenser.

k) Reduce the extract down to approximately 10 mL on a rotary-evaporator. Refer to BU-WI-3009 Rotary Evaporators for the proper use of the rotary evaporator.

l) Top the flask up to approximately 350 mL ±10mL with a portion of the Back-Half Rinse.

m) Continue reducing until the entire Back-Half Rinse is consumed.

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n) Rinse the original container with 3 x 10 mL of toluene, shaking each rinse in the bottle and then adding it to the florence flask.

o) Proceed to Section 9.4 Extract Isolation and Purification Procedures.

9.3.7 Extraction of Butter and Edible Oils

a) Method Blank (MB)
   i. Prepare a separate MB for each set up of 20 client samples.
   ii. Subsample 10 grams ±2 grams of Mazola Corn Oil as described below.

b) Laboratory Control Sample (LCS)
   i. Prepare a separate LCS for each set up of 20 client samples.
   ii. Subsample 10 grams ±2 grams of Mazola Corn Oil as described below.

c) Subsampling Procedure
   i. Place the sample jar on to a two-place balance, and tare the balance
   ii. Subsample the amount of sample as required on the batch sheet ± 1.0 g (if sampling less than 10 grams defer to the Supervisor for acceptable subsampling range criteria) into the jar and record the weight of the subsample to two decimal places. Refer to BU-WI-3001 Subsampling and Homogenization of Samples for Organics Analysis for proper sub-sampling and solid homogenization procedures and BU-WI-3005 Food Sample Homogenization for food prep instructions.

d) Subsample the predetermined amount of sample into a 500 mL jar. Record the weight of the subsample to two decimal places.

e) Spike the appropriate volume of Extraction Standard, as indicated on the batch sheet, on to the sample. Refer to 'BU-WI-3002 Standards Prep, Spiking, and Witnessing' for proper spiking procedures

f) Add 50 mL ±5mL of hexane. If there are gel capsules, break them open using pre-cleaned tweezers and pre-cleaned scissors – a cut in half is preferred.

g) Cap tightly and shake (~10 seconds). Carefully loosen the cap to vent, and re-cap.

h) Pour off hexane and oil mixture into another labelled jar. Repeat two more times.

i) Add ~50 grams ±10 grams of acid silica (refer to BU-WI-3016 Acid Silica Column Preparation and Use for preparation of acid silica) to the jar containing the oil and hexane mixture. Swirl the jar to ensure even distribution of acid silica throughout the sample.

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j) Pour the hexane/acid silica mixture through an Allihn filter filled with 3-4cm of acid silica.

k) Rinse this jar 3 times with 50 mL ±5mL of DCM to transfer all acid silica onto the Allihn filter. Provide a final rinse of ~100 mL ±10ml of DCM through the Allihn filter.

l) Proceed to Section 9.4 Extract Isolation and Purification Procedures.

9.3.8 Base Digestion/Extraction of Food Matrices

Refer to BU-TP-2170 Base Digestion.

9.4 Extract Isolation and Purification Procedures

9.4.1 Extract Reduction and Addition of Cleanup Standard

1. If the Extract is still at a large volume reduce the collected extract down to approximately 2 mL on a rotary-evaporator. The bath temperature must not exceed 35˚C for PCB extracts, 30˚C for PBDE or PCN extracts and 45˚C for DX extracts. If the extract is for multiple analyses, use the lowest temperature indicated, Refer to BU-WI-3009 Rotary Evaporators for the proper use of the rotary evaporator.

2. Spike the appropriate volume of Cleanup Standard, as indicated on the preparative batch sheet, into remaining 2 mL of solvent. Refer to BU-WI-3002 Standards Prep, Spiking, and Witnessing for proper spiking procedures.

3. Mix the Cleanup Standard in to extract by lightly swirling.

9.4.2 Copper Treatment

Refer to BU-WI-3023 Copper Treatment.

9.4.3 Acid Columns

Refer to BU-WI-3016 Acid Silica Column Preparation and Use.

9.4.4 Alumina Columns

Refer to BU-WI-3011 Alumina Column.

9.4.5 Florisil Columns

Refer to BU-WI-3017 Florisil Column.

9.4.6 Carbon Columns

Refer to BU-WI-3006 Chromacarb.

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9.4.7 Solvent Exchange to Hexane

1. Place the c-tube containing the extract on to the N-Evap. The bath temperature must not exceed 30˚C for PCBE and PCN, 35˚C for PCB extracts and 45˚C for DX extracts. If the extract is for multiple analyses, use the lowest temperature indicated, Refer to BU-WI-3013 Nitrogen Evaporators for the proper use of the N-Evap.
2. Reduce the extract down to approximately 50 µL for PCN and PCB, 200 µL for PBDE, and dryness for DX.
3. Ensure that extracts are mixed well at least every 30 minutes either by using a pipette to draw solvent from the bottom of the extract and gently dispense at the top or by gently swirling the c-tube. This ensures solvent layers do not stratify and all traces of previous solvents are gone
4. Remove the extract from the N-Evap.
5. Add approximately 1 mL of hexane to the extract using a Pasteur pipette.
6. Vortex for 5 seconds.

9.4.8 Preparing DX[2] Extracts for Instrument Injection

1. Place the c-tube containing the extract on to the N-Evap. Maximum bath temperature of 45˚C. Refer to BU-WI-3013 Nitrogen Evaporators for the proper use of the N-Evap.
2. Reduce the extract down to approximately 500 µL.
3. Transfer the extract to the microvial being careful to transfer every last drop of extract.

NOTE: All transfers are to be done from one labelled container to another labelled container. Reading both labels and ensuring a perfect transfer is crucial in all circumstances to avoid sample exchanges.

4. Place the micro-vial on to the N-Evap.
5. Reduce the extract to just-dry.
6. Spike the appropriate volume of Injection Standard, as indicated on the preparative batch sheet, into the micro-vial, and mark the level with a fine tip marker on the micro-vial. Refer to BU-WI-3002 Standards Prep, Spiking, and Witnessing for proper spiking procedures.
7. Cap the micro-vial.
8. Vortex the micro-vial for 5 seconds, then submit to instruments.
9. The final volume is 20 µL or the amount of injection standard added with a syringe. Refer to the batch sheet to ensure any deviations for projects are followed.

9.4.9 Preparing PCB Extracts for Instrument Injection

1. Add 20 µL ± 0.8 µL of Nonane to a micro-vial, using a verified dedicated syringe, and mark the level with a fine tip marker.
2. Place the c-tube containing the extract on to the N-Evap. Maximum bath temperature of 35˚C. Refer to Refer to BU-WI-3013 Nitrogen Evaporators for the proper use of the N-Evap.
3. Reduce the extract down to approximately 500 µL. Vortex extract to mix well.
4. Transfer the extract to the microvial being careful to transfer every last drop of extract.

[2] DX designates PCDD/F and selected PCB for a combined instrumental analysis

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NOTE: All transfers are to be done from one labelled container to another labelled container. Reading both labels and ensuring a perfect transfer is crucial in all circumstances to avoid sample exchanges.

5. Place the micro-vial on to the N-Evap.
6. Reduce the extract down to the mark on the micro-vial (20 µL).
7. Spike the appropriate volume of Injection Standard, as indicated on the preparative batch sheet, into the micro-vial. Refer to BU-WI-3002 Standards Prep, Spiking, and Witnessing for proper spiking procedures into the micro-vial.
8. Cap the micro-vial.
9. Vortex the micro-vial for 5 seconds, then submit to instruments.
10. The final volume is typically 25 µL (volume of nonane added plus the volume of the spike, both measured with a syringe). Refer to the batch sheet to ensure any deviations for projects are followed. The final volume for edible oils is 45uL.

9.4.10 Preparing PCN Extracts for Instrument Injection

1. Add 40 µL ± 0.8 µL of Toluene to a micro-vial, using a verified dedicated syringe, and mark the level with a fine tip marker.
2. Place the c-tube containing the extract on to the N-Evap. Maximum bath temperature of 30˚C. Refer to Refer to BU-WI-3013 Nitrogen Evaporators for the proper use of the N-Evap.
3. Reduce the extract down to approximately 500 µL. Vortex extract to mix well.
4. Transfer the extract to the micro-vial being careful to transfer every last drop of extract.

NOTE: All transfers are to be done from one labelled container to another labelled container. Reading both labels and ensuring a perfect transfer is crucial in all circumstances to avoid sample exchanges.

5. Place the micro-vial on to the N-Evap.
6. Reduce the extract down to the mark on the micro-vial (40 µL).
7. Spike the appropriate volume of Injection Standard, as indicated on the preparative batch sheet, into the micro-vial. Refer to BU-WI-3002 Standards Prep, Spiking, and Witnessing for proper spiking procedures into the micro-vial.
8. Cap the micro-vial.
9. Vortex the micro-vial for 5 seconds, then submit to instruments.
10. The final volume is typically 45 µL (volume of toluene added plus the volume of the spike, both measured with a syringe). Refer to the batch sheet to ensure any deviations for projects are followed.

10.0 DATA CALCULATION AND REPORTING

See the appropriate instrumental method for sample data calculations.

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11.0 REFERENCES

11.1 Primary References

1. EPA Method 1613B, Analysis of Dioxins and Furans in Wastewater, Revision B, October, 1994.[water samples by 1613B Dioxins/Furans]
2. EPA Method 1668C, Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, Revision C, April, 2010. [water samples by 1668C PCBs]
3. EPA Method 8290A, Polychlorinated Dibenzo-p-dioxins (PCDDs) and Polychlorinated dibenzofurans (PCDFs) by high-resolution gas chromatography/high-resolution mass spectrometry (HRGC/HRMS), Revision A, January, 1998. [water samples by 8290A Dioxins/Furans]
4. EPA Method 0023A, Sampling Method for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofuran emissions from stationary sources, Revision 1, December, 1996.[air samples by 0023A – Stack ambient]
5. EPA TO-9A, Determination of Polychlorinated, Polybrominated and Brominated/Chlorinated Dibenzo-p-Dioxins and Dibenzofurans in ambient air, Edition 2, January, 1999.[air samples by T0-9A]

11.2 Secondary References

1. US FDA Division of Field Science, Bulletin #4084 Dioxins (July 7, 1997) [milk extraction method]
2. Code of the Federal Registry, Volume 40, Chapter I (D), Part 136 App. B; Method 23.(2012) [Procedure for calculating MDLs]
3. Ontario MOE Laboratory Services Branch, Practices for the Collection and Handling of Drinking Water Samples, Version 2 (April 2009)
4. SW846 Method 3540C Soxhlet Extraction (December, 1996)
5. SW846 Method 3510C Separatory Funnel Liquid-Liquid Extraction (December, 1996)
6. CDC Laboratory Procedure Manual, NHANES 2003-2004 (October 2006)

11.3 Supporting ALS Documents and Forms

1. NA-SP-0001 Definitions of Key Terms
2. NA-SP-0100 Initial Method Validation – Chemistry
3. NA-SP-0102 Method Revalidation and MU - Chemistry
4. BU-TP-1110 Overall HR Prep for Serum
5. BU-TP-2170 Base Digestion
6. BU-TM-1107 PCDD_F
7. BU-TM-1102 PCN
8. BU-TM-1105 PCB Congeners
9. BU-TM-1120 OCP & PCB via LRMS
10. BU-TM-1200 Moisture Content by Gravimetry
11. BU-TM-1700 Lipids
12. BU-WI-0325 Water Preservation
13. BU-SP-0261 Sample Control, Login and Receipt

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14. BU-SP-0253 Laboratory Water
15. BU-SP-0018 International Soil
16. BU-SP-0250 Control of Reagents and Reference Materials
17. BU-SP-0020 MSDS and Chemical Handling
18. BU-SP-0021 Waste Handling and Sample Disposal
19. BU-WI-3002 Standards Prep, Spiking and Witnessing
20. BU-WI-3004 Nonane Distillation
21. BU-WI-3001 Subsampling and Homogenization of Samples for Organic Analysis
22. BU-WI-3005 Food Sample Homogenization
23. BU-WI-3009 Rotary Evaporators
24. BU-WI-3003 Soxhlet Set-up Procedure
25. BU-WI-3016 Acid Silica Column Preparation and Use
26. BU-WI-3011 Alumina Column
27. BU-WI-3017 Gel Permeation Chromatography
28. BU-WI-3018 Florisil Column
29. BU-WI-3006 Chromacarb
30. BU-WI-3013 Nitrogen Evaporators
31. BU-WI-3000 Organic Glassware/Equipment Cleaning Proofing and Maintenance
32. BU-WI-3023 Copper Treatment
33. BU-FM-xxxx "Batch Sheet Templates"

12.0 DEVIATIONS FROM THE PRIMARY REFERENCE

The reference methods applicable to this document are:
US EPA Office of Water, Methods 1613B and 1668C
US EPA Office of Solid Waste, SW846 Methods 8290A and 0023/8290A
US EPA Office of Research & Development Method TO-9A.
US EPA Office of Air Quality Planning & Standards Method 23.

The following changes and clarifications apply:

1) Although categorically stated in many associated PCDD/F methods, but not all, and also stated in in the PCB method 1668C, we maintain that each and every individual clean-up procedure is, by definition, performance-based and optional. There is not an expectation within the industry to follow exactly the descriptions of clean-ups in reference methods. Adaptations which meet or exceed the required performance criteria are therefore acceptable within the scope of each reference method. The reference method descriptions are intended as guidelines or templates available to help the laboratory to define effective in-house clean-up methods. The objective within the laboratory is to provide quality clean extracts to the instrument for analysis. Each individual clean-up is part of the laboratory's 'arsenal' in order to achieve this objective.

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2) There are differences within the individual reference methods as to the precise spiking protocols for adding extraction standards and native spikes (for LCS, MS and MSD). This prep method requires solids (including stack and ambient sorbents/filters) to be spiked in the soxhlet extractor from a nonane solution and waters are spiked before filtering from an acetone solution. This is consistent with the 8290A approach.

3) Sub-sampling of solids and pre-extraction processing is done in a manner that minimizes potential for cross-contamination. These processes are designed around SW846 protocols rather than 1613B or 1668C protocols. Solids are sub-sampled directly from the bottle as submitted to the laboratory wherever practical. If the sample is submitted such that homogenization in the bottle is impractical (e.g. the bottle is too full or lumps cannot be broken down), then transferring the sample to a tray or another bottle maybe in order.

4) The concentrations of labelled and native spiking solutions are not consistent with those listed in all of the reference methods. These concentrations are prepared at levels convenient and expedient for accurate laboratory processing.

5) The method 1613B (see Sections 6.5.6/11.4.3) protocol for the filtration of water samples with visible suspended solids is via a suction filtration device through a glass fibre filter of unspecified pore size. ALS BU has modified this procedure (Section 9.3.4), to employ gravity filtration through a bed of glass wool. This modification avoids the potential for sample cross contamination during the use of glass frits or Buchner funnels employed in suction filtration devices.

6) Methods 1613B and 1668AC discuss water and solid samples equilibrating for 1-2 hours after standards are added, and tissue samples equilibrating for 12-24 hours after addition of sodium sulphate. Method 8290A does not list any equilibrium requirements. Samples are allowed to warm up to room temperature before further processing but are not given specific equilibrium time after standards are added.

7) For PCB extracts, 5uL of injection standard for PCB is employed rather than 2uL as per method 1668A/C. This ensures a quantitative addition of the recovery standards but increases the actual extract final volume from 22uL to 25uL. (See footnote in Section 7.14 of methods 1614A, 1668A and 1668C).

8) Solids destined for 8290A analysis may be stored frozen at <-10˚C.

13.0 REVISION SUMMARY

Version #	Date	Summary of Major Changes Made / By
01	09 Jan 2007	Created from the now retired method 15SVSM_PD_MM_01 to include PCB prep and to add food matrices. Ron McLeod/Alastair Blythe/Steve Kennedy
02	23 Jan 2007	Refer to v02
05	25 May 2009	Refer to v03
06	27 May 2009	Refer to v04
07	14 Jun 2009	Refer to v07
08	17 Sep 2010	Refer to v08
09	10 Nov 2011	Refer to v09
10	29 Nov 2011	Refer to v10

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Version #	Date	Summary of Major Changes Made / By
11	20 June 2012	Refer to v11
12	15 Nov 2012	Refer to v12
13	12 Mar 2013	Refer to v13
14	18 July 2013	Refer to v14
15	08-May-2014	Refer to v15
16	11-July-2014	Refer to v16
17	31-Jul-2014	Refer to v17
18	20-Feb-2015	Refer to v18
19	4-Mar-2016	Refer to v19
20	2-Feb-2017	Refer to v20
21	10-Sep-2018	Refer to v21
22	14-May-2020	Addition of Quebec DX water LCSD requirement. Updated water extraction method time for second and third shake. Added deviation #6. / R.Saxon Minor edits. Added Ontario drinking water hold times to Section 5.0. / T.Jackson
23	17-Feb-2021	Addition of Job task to Hazard Assessment table, Section 15.0: Venting separatory funnels. Updated Hazard Assessment table, section 15.0 to newer safety table format. / R/Menon

14.0 WASTE MANAGEMENT

Refer to BU-SP-0021 Waste Handling and Sample Disposal.

Waste solvent and waters contaminated with solvent must be disposed into the liquid halogenated waste stream. Spent solid sorbents must be left in an open container within a fume hood to allow dissipation of solvent vapours before disposal into the municipal waste stream. Unless handling tissues requiring biosafety protocols (e.g. blood products or vole tissues), disposable glassware, gloves, aluminium foil etc. can all be disposed into the municipal waste stream.

Waste information related to this activity, which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

15.0 SAFETY

This task may include CHEMICAL, BIOLOGICAL, OPERATIONAL and/or EQUIPMENT hazards. Staff must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

Refer to Material Safety Data Sheets (MSDS) for detailed information about safe handling guidelines and hazards associated with chemicals used in this method.

HAZARD ASSESSMENT		
Job Task #1:	**Hazards**	**Preventative Measures**
Sample Preparation	Exposure to solvents, exposure to contaminants in samples.	Wear gloves, safety glasses and lab coat. Work in fume hood. Avoid skin contact with solvents, samples, and sample extracts.
Job Task #2: Handling Reagents and	Hazards (See hazard code)	Preventative Measures

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HAZARD ASSESSMENT			
Solvents			
DCM	Warning: Causes skin and eye irritation, May cause respiratory irritation, drowsiness or dizziness. Suspected of causing cancer		Wear DCM gloves, safety goggles, and lab coat. Work in fume hood. Avoid Skin Contact with solvents, samples and sample extracts.
Acetone	Danger: Highly flammable liquid and vapor, causes serious eye irritation, may cause drowsiness or dizziness.		Wear gloves, safety glasses and lab coat. Work in fume hood. Avoid skin contact with solvents, samples and sample extracts. Keep away from ignition sources,
Toluene	Danger: Flammable, irritant, health hazard		Wear gloves, safety glasses and lab coat. Work in fume hood. Avoid skin contact with solvents, samples and sample extracts. Keep away from ignition sources,
Hexanes	Danger: Highly flammable liquid and vapor, causes serious eye irritation, may cause drowsiness or dizziness. Toxic to aquatic life		Wear gloves, safety glasses and lab coat. Work in fume hood. Avoid skin contact with solvents, samples and sample extracts. Keep away from ignition sources,
Job Task #3:	**Hazards**		**Preventative Measures**
Standard preparation	Exposure to solvents, exposure to toxic standard components.		Wear gloves, safety glasses and lab coat. Work in fume hood. Avoid skin contact with solvents, samples and sample extracts.
Job Task #4:	**Hazards**		**Preventative Measures**
Extended computer use	Eye strain and fatigue		Take regular short breaks to work on different tasks. Focus on objects at varying distances and/or rest your eyes for a few moments throughout the day.
Roto-vaps and N-evaps	Exposure to solvents, fire or electrocution, cuts or pinches		Wear PPE. Receive appropriate training before operation. Perform a visual inspection before operation

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HAZARD ASSESSMENT		
	from moving parts.	to ensure all parts are in correct working condition. Check for exposed wires, cracks on glass vessels, leaks on lines (hoses), solvent or water near electrical connections. Operate with caution. To be operated in a functional fume hood only.
Burners	High voltage, burns.	Wear PPE. Receive appropriate training before operation. Perform a visual inspection before operation to ensure all parts are in correct working condition. Do not touch the heating plates or compartments. Check for leaks on lines (hoses), solvent or water near electrical connections. Operate with caution. Do not use if condensers or fume hoods are not functioning.
Job Task #5:	**Hazards**	**Preventative Measures**
High Level Sample Preparation	Contamination of employee and environment	Wear gloves, safety glasses and lab coat. Work in fume hood. Avoid skin contact with solvents, samples, and sample extracts. Ensure all disposal techniques are followed as in BU-SP-0021 Waste Handling and Sample Disposal.
Glassware Handling	Cuts	No broken or cracked glassware permitted in the lab. Wear proper PPE (including cut proof gloves) when handling any broken glassware.
Job Task #6:	**Hazards**	**Preventative Measures**
Venting Separatory Funnels	Pressure build up in funnel- spills, solvent exposure	Shaking/Venting must begin immediately after cap is placed on funnel. Vent samples prior to shaking.

Although only dilute solutions of standards are used in the laboratory to minimize handling risks, the intrinsic risks in handling PCDD/F and PCB standards and extracts must be recognized. The 2,3,7,8-TCDD isomer has been found to be carcinogenic and teratogenic in laboratory animal studies. On the basis of available toxicological information, all tetra- to octachlorinated dibenzo(p)dioxins/furans should be handled only by highly trained personnel thoroughly familiar with the handling and cautionary procedures and the associated risks.

Hazard information related to this activity, which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

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16.0 APPENDICES

16.1 Appendix A

Table 1: Combined Extraction Compatibility Guide

		Target Groups						
		PCDD/F	209 PCB	PCB-77,81,126,169	WHO Toxic PCB	Non-Ortho-Substituted PCB	BDPE	PCN
Clean-Up Options								
Silica Gel		C	C	C	C	C	C	C
Acid/Silica		C	C	C	C	C	C	C
AgNO3/Silica		C	C	C	C	C	NC	C
Alumina		C	C	C	C	C	C	C
Florisil		C	C	C	C	C	C	C
Carbon		C, R	NC, F&R	C, R	CS, R	CS, F	C, F	C, R
Sulphur		C	C	C	C	C	C	C
Extract Dryness		C	NC	C	C	C	NC	NC
Final Solvent	Default	nonane	nonane				toluene	nonane
	Option	toluene	toluene				n/a	toluene

C = Compatible
NC = Non-Compatible
CS = Compatible under selected conditions and/or for selected targets
R = Reverse Elution
F = Forward Elution

1) All four target groups can be co-extracted and analyzed without significant interferences when handled appropriately. When sample size is limited and to avoid compromise to detection limits, all analyses can be done from a single extract without splitting for separate analyses.
   To accomplish this, an extract that contains all targets in a single extract can be analyzed for PCB and/or BDPE before carbon column cleanup. PCDD/F and PCN and coplanar PCB can be analyzed after carbon cleanup.
2) Co-planar PCBs fractionate with the PCDD/F targets on a carbon column and reverse elute.

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3) Dependent upon levels, PCB can cause interferences on the PCDD/F and PCN analyses. The PCB interferences can be removed by florisil (re PCDD/F) or carbon (re PCDD/F and PCN) column cleanup

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OCP & Toxaphene Prep

Document Authorization

Authorized By: _____ Date: _____
Quality Authorization

Authorized By: _____ Date: _____
Technical Authorization

Implementation Authorization

Authorized By: _____ Effective Date: _____
Laboratory Manager

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1.0 SCOPE AND APPLICATION

1.1 Scope

Organochlorine pesticides are insecticides composed primarily of carbon, hydrogen, and chlorine. Most of them break down slowly and can remain in the environment long after application and in organisms long after exposure. Several commonly known organochlorines have been banned for use in the U.S. including DDT, aldrin, dieldrin, toxaphene, chlordane and heptachlor. Those that still remain in use include lindane, endosulfan, dicofol, methoxychlor and pentachlorophenol.

Organochlorine pesticides are used mostly as insecticides. Specific uses take a wide range of forms, from pellet application in field crops to sprays for seed coating and grain storage. Some organochlorines are applied to surfaces to kill insects that land there. This is how DDT is used in mosquito control.

1.2 Parameters and Reporting Limits

Refer to BU-TM-1103 OC Pesticides & BU-TM-1121 8276 Toxaphene.

1.3 Method Performance Data and Measurement Uncertainty

Refer to BU-TM-1103 OC Pesticides & BU-TM-1121 8276 Toxaphene.

2.0 PRINCIPLE OF METHOD

This procedure provides the preparation details of samples destined for instrumental analysis of organochlorine pesticides (BU-TM-1103 OC Pesticides) & Toxaphenes (BU-TM-1121 8276 Toxaphene). Solid samples are extracted via Soxhlet technique using dichloromethane as the extracting solvent; refer to 'BU-WI-3003 Soxhlet Setup Procedure'. Aqueous samples are extracted using a liquid-liquid separatory funnel extraction technique. Serum samples are extracted using a subsampling procedure including methanol and sodium sulphate. Optionally, extracts from tissues or other matrices containing high molecular weight contaminants may be cleaned-up by gel permeation chromatography, refer to 'BU-WI-3017 Gel Permeation Chromatography'.

3.0 DEFINITIONS

Refer to 'NA-SP-0001 Definitions of Key Terms' for definitions of common terms.

4.0 INTERFERENCES

Refer to BU-TM-1103 OC Pesticides & BU-TM-1121 8276 Toxaphene.

5.0 SAMPLE HANDLING AND STORAGE

Parameter(s) and Matrix: All Parameters - Water	
Container	1 litre amber bottle with Teflon lined lid pre-cleaned, Reference: EPA 1699 Section 6.1.1.
Preservative	If residual chlorine is present, add 80 mg sodium thiosulfate per liter of

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Parameter(s) and Matrix: All Parameters - Water	
	water. EPA 1699 Section 8.0
Min. Sample Amount	1 liter
Sample Pre-treatment	N/A
Lab Storage Conditions	>0 - 6˚C Ref: US EPA 40CFR Part 136 2012 If sample will be frozen allow room for expansion. Ref: EPA 1699 Section 8.0
Maximum Sample Holding Time (Provinces / Reference)	7 days from collection EPA 1699 Section 8.5.1
Maximum Extract / Digest Holding Time (Provinces / References)	40 days Reference: EPA 1699 Section 8.5.1

Parameter(s) and Matrix: All Parameters - Tissue	
Container	250 mL amber jar with Teflon lined lid Reference: EPA1699 Section 6.1.1
Preservative	N/A
Min. Sample Amount	20g
Sample Pre-treatment	N/A
Lab Storage Conditions	Freeze tissue samples upon receipt at the laboratory and maintain in the dark at less than -10˚C until prepared. Maintain unused sample in the dark at less than -10˚C. Reference EPA 1699 Section 8.4.3 Extracts are kept in fridge at less than 10˚C this is a variance from EPA 1699 Section 8.4.4 backed by historical data.
Maximum Sample Holding Time (Provinces / Reference)	1 year Reference EPA 1699 Section 8.5.2
Maximum Extract / Digest Holding Time (Provinces / References)	1 year Reference: EPA 1699 Section 8.5.2

Parameter(s) and Matrix: All Parameters – Soil	
Container	250 mL amber jar with Teflon lined lid Reference: EPA1699 Section 6.1.1
Preservative	N/A
Min. Sample Amount	20g
Sample Pre-treatment	N/A
Lab Storage Conditions	Store solid, semi-solid, oily, and mixed-phase samples in the dark at less than -10˚C Reference EPA 1699 Section 8.3.2 Extracts are kept in fridge at less than 10˚C this is a variance from EPA 1699 Section 8.4.4 backed by historical data.
Maximum Sample Holding Time (Provinces / References)	1 year Reference EPA 1699 Section 8.5.2
Maximum Extract / Digest	1 year

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Parameter(s) and Matrix: All Parameters – Soil	
Holding Time (Provinces / References)	Reference: EPA 1699 Section 8.5.2

Parameter(s) and Matrix: All parameters - Serum	
Container	Sample containers must be able to withstand freeze/thaw cycles and temperatures down to -80˚C without compromise to the samples. Options include: (a) amber glass with caps or lids which are lined with polytetrafluoroethene (PTFE) or (b) polypropylene vials.
Preservative	None
Min. Sample Amount	Minimum sample amount required to meet the contract required RL
Sample Pre-treatment	None
Lab Storage Conditions	In the dark at <-70˚C for long term storage In the dark at <-10˚C for storage of up to 1 year Reference: Stats/Health Canada Standing Offer 4600000355
Maximum Sample Holding Time	Indefinite at <-70˚C [likely several years]; 1 year at <-10˚C. Reference: Stats/Health Canada Standing Offer 4600000355
Storage of Leftover Sample	In the dark at <-70˚C for long term storage In the dark at <-10˚C for storage of up to 1 year Reference: Stats/Health Canada Standing Offer 4600000355
Sample Disposal	Treatment with bleach (hypochlorite) and then rinsed down the sink

5.1 Sampling Instructions

Sample collection is not conducted by the laboratory.

5.2 Sampling Handling and Transportation

Refer to ALS Sample Handling and Preservation Guidelines for further details on the sample handling procedures recommended by ALS.

If holding time violations occur, Account Manager(s) must be notified.

6.0 EQUIPMENT AND SUPPLIES

6.1 Primary Equipment

1. Soxhlet heating mantle.
2. Rotary evaporator
3. N-Evap
4. Analytical balance capable of accurately weighing to four decimal places.
5. Analytical balance capable of accurately weighing to two decimal places.

6.2 Support Equipment

1. Muffle furnace capable of maintaining 450˚C ±5˚C for a minimum of 8 hours
2. Vortex Mixer

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6.3 Supplies

1. Support racks and various clamps
2. Teflon squeeze bottles for solvent rinsing MEOL#4030011
3. Stainless steel spatulas MEOL#4554571
4. Scissors MEOL#4020036
5. 1 mL pipette bulbs MEOL#4430106
6. Boiling chips, MEOL#6040942
7. pH paper MEOL#4350113
8. Aluminium foil MEOL#4550248/4550376
9. Nitrogen, ultra high purity. Expiry date: indefinite. Supplier: Praxair, Supplier # (fill in); MEOL # n/a (ordered on the Praxair website)
10. Teflon tape. MEOL#4030046/4030089
11. Adhesive labels, MEOL# 8020034
12. Permanent fine-tip marker
13. 500 mL florence flasks with 24/40 ground glass joint. MEOL#4001540
14. Calibrated Bottle-Top Dispensers, for solvents (10.0 mL delivery volume required). MEOL#4120149 **Note**: Some bottle-top dispensers are incompatible with acetone.
15. Soxhlet extraction apparatuses, Jon Vandenhoff, ordered directly
16. Dean-Stark adapters, Jon Vandenhoff, ordered directly.
17. Disposable Glass Column, Jon Vandenhoff, ordered directly.
18. 1000 mL 'Class A' glass graduated cylinder, VWR, MEOL# n/a
19. 100 mL 'Class A' glass graduated cylinder, VWR, MEOL# 4001645
20. 10 mL 'Class A' glass graduated cylinder, VWR, MEOL# 4001647
21. 1000 mL Erlenmeyer flask, Fisher, MEOL# n/a
22. 250 mL Erlenmeyer flasks. MEOL#4001559, Fisher, MEOL# 4000090
23. 600 mL glass beakers. MEOL#4001558
24. 600 mL quartz beakers MEOL#4001474
25. Allihn filters, column length = 11cm, internal diameter = 4cm, contain a coarse fritted disk (40-60 microns), Jon Vandenhoff, ordered directly
26. 2 L separatory funnels
27. 5 ¾" disposable Pasteur pipettes borosilicate glass/non sterile. MEOL#4430061
28. 9" disposable Pasteur pipettes borosilicate glass/non sterile. MEOL#4430062
29. 10 µL-1000 µL syringes, Hamilton, MEOL # for various syringes can be found in G:\Enviro\Purchasing\Organic Prep\Approved Supplies.xls (Syringes Tab)
30. C-tubes MEOL#4000187/4000202
31. Robovials MEOL# 4320490, lids MEOL# 4320176
32. Glass wool, borosilicate from Fisher, Cat# 11-388, MEOL# 4340169

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7.0 REAGENTS AND STANDARDS

7.1 Reagents

Refer to 'BU-SP-0250 Control of Reagents and Reference Materials' for ordering, labeling, preparation guidelines, and record-keeping requirements.

1. Toluene distilled in glass, purchased from Caledon. Cat # 9201-2-40. MEOL#6010007
2. Dichloromethane (DCM) distilled in glass, purchase from Caledon. Cat # 3601-2-40. MEOL#6010024
3. Hexane distilled in glass, purchased from Caledon. Cat # 5500-2-40.MEOL#6010005
4. Acetone distilled in glass, purchased from Caledon. Cat # 1201-2-40. MEOL#6010003
5. Type 1 Laboratory Water (Ultra-Pure DI) – In-house
6. Sodium Chloride, supplied by VWR, crystals. MEOL# 6030479.
7. Silica Gel 60, 100-200 mesh, purchased from Caledon Part#: 6985-8 MEOL#6040115/6030881
    i. Bake the desired amount of Silica in a quartz beaker at 450˚C for a minimum of 8 hours.
    ii. Remove the Silica from the muffle furnace, cover loosely with foil, and allow it to cool to the point of being able to be handled.
    iii. While still warm, place the Silica in to a clean, nitrogen-purged, flask.
    iv. Purge the Silica with nitrogen for approximately two minutes.
    v. Stopper the flask, and seal stopper with teflon tape. Store at room temperature.
8. Sodium Sulphate (A.C.S grade) andhydrous powder. Purchased from Caledon.Part#8220-1 MEOL#6030433
    i. Place the desired amount of sodium sulphate in to a quartz beaker.
    ii. Bake at 450˚C for a minimum of 6 hours.
    iii. Store in an air-tight glass jar. Label and store at room temperature for up to 3 months.
9. Chromacarb
    i. 'BU-WI-3006 Chromacarb' for preparation and use.
10. 80:20 Toluene:Acetone
    i. Prepare a 5:1 solution of Toluene/Acetone in 1 L clear jar. This for cleaning only and does not need to be prepared quantitatively.
    ii. Do not re-use/store. Dispose of after use (refer to section 14.0 "Waste Management").
11. 1:1 DCM:Hexane
    i. 50:50 is made using a Class A glass graduated cylinder capable of measuring 2000 mL.
    ii. 2000 mL of DCM is measured out and poured into the Amber jug.
    iii. 2000 mL of Hexane is added to the jug.
    iv. Contents are mixing thoroughly by inverting the jug several times.

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v. The reagent is logged into the organic reagent prep binder with an expiry of 3 months.
vi. Label the jug and store at room temperature.

7.2 Standards

The following standards are prepared by the instrumentation lab (Refer to BU-TM-1103 OC Pesticides & BU-TM-1121 8276 Toxaphene) and used for extraction procedure for all matrices:

OCP Standard Type	Volume per split (µL)	Volume per Portion (µL)	Name
Extraction	20		HROCP-ES#1-xxx
Native	20		HROCP-NS#1-xxx
Injection		20	HROCP-IS#1-xxx

Toxaphene Standard Type	Volume per split (µL)	Volume per Portion (µL)	Name
Extraction	20		TXN-ES#2-xxx
Native	120		TXN-NS#1-xxx
Injection		20	TXN-IS#1-xxx

NOTES:
i. Refer to BU-WI-3002 Standards Prep, Spiking and Witnessing for proper spiking procedures.
ii. Refer to the preparative batch sheet for standard to be used in spiking.
iii. Refer to BU-FM-3002c Syringe List in the "Syringe Control – Organic Extraction" binder (located at the bench), every time a syringe is specified to be used in this document, for proper syringe selection.

See BU-TM-1103 OC Pesticides & BU-TM-1121 8276 Toxaphene for standard information.

8.0 QUALITY CONTROL

8.1 Method QC

Maximum (Sample Preparation) Batch Size = 20 Client Samples

Method QC	Min. Frequency	Data Quality Objective
Method Blank (MB)	1 per batch	Refer to BU-TM-1103 OC Pesticides & BU-TM-1121 8276 Toxaphene
Laboratory Control Sample (LCS)	1 per batch	Refer to BU-TM-1103 OC Pesticides & BU-TM-1121 8276 Toxaphene
Reference Material (RM)	Upon Request	Refer to BU-TM-1103 OC Pesticides & BU-TM-1121 8276 Toxaphene
Matrix Spike (MS)	Upon Request	Refer to BU-TM-1103 OC Pesticides & BU-TM-1121 8276 Toxaphene

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Method QC	Min. Frequency	Data Quality Objective
Surrogate(s) (SURR)	All samples and QC samples	Refer to BU-TM-1103 OC Pesticides & BU-TM-1121 8276 Toxaphene
Sample Duplicate	1 per batch, where sufficient sample is available	Refer to BU-TM-1103 OC Pesticides & BU-TM-1121 8276 Toxaphene

For Method QC preparation information see the relevant pre-extraction procedure below.

8.2 Instrument QC

Refer to BU-TM-1103 OC Pesticides & BU-TM-1121 8276 Toxaphene.

9.0 TEST PROCEDURE

Note: Not all of the steps in the Extract Purification Procedures are required for all matrices. The decision on which purification steps are required will be made based on matrix type, matrix history, and sample assessment. This decision will be made by Management Staff and detailed on each preparative batch sheet.

9.1 Extraction of Water Samples (<1% Suspended Solids)

Note: If the sample contains visible solids, notify appropriate Supervisor.

9.1.1 Pre-Extraction Procedures

1. Temperature Adjustment of Sample

a) Place the samples on the bench, away from direct heat sources or sunlight.
b) Allow the bottles to come up to ambient room temperature before proceeding with extraction.

2. Check the pH of the Sample

a) Mix the sample by vigorously shaking for 20 seconds.
b) Using a disposable pipette, deliver a few drops of the sample onto pH paper.
c) Record the pH of the sample on the batch sheet.
d) If the pH is above 9 or less than 6 report this information to the Supervisor. Do not proceed with the extraction procedure until given further instructions on this matter.

3. Prepare Separatory Funnels

a) Wash and solvent rinse a 2 L separatory funnel as outlined in 'BU-WI-3000 Glassware Cleaning, Proofing, and Maintenance.'

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b) Sonicate the stopper and stopcock in enough 80:20 Toluene:Acetone to cover the contents in a 1 L clear jar for 15 minutes, followed by a rinse with hexanes by emptying the jar of solvent and refilling with Hexane.
c) Assemble the separatory funnel and place it in to a ring stand.
d) Rinse the sep funnel with 1x exactly 100 mL DCM, measured using a class A, to deliver, glass graduated cylinder, shaking the sep funnel for 15 seconds. Discard the DCM rinse.

4. Prepare Allihn Filters with Sodium Sulphate

a) Wash and solvent rinse an Allihn filter and a 500 mL florence flask, as outlined in 'BU-WI-3000 Glassware Cleaning, Proofing, and Maintenance.'
b) Place the Allihn filter on to the 500 mL florence flask.
c) Fill the Allihn filter half full with sodium sulphate.
d) Rinse the Allihn filter containing the sodium sulphate with 50 mL ± 5 mL DCM, measured using a graduated beaker, discarding the DCM from the Florence flask after each rinse.
e) Cover the top of the Allihn filter with clean foil until needed.

5. Mark the Sample Level on the Original Bottle

a) Do not perform this step until the sample has come up to room temperature.
b) Using a fine-point permanent marker, mark the line of the sample meniscus on the bottle.
c) Once the sample has been removed from the bottle and the residual DCM rinse has evaporated, fill the bottle to the line with tap water.
d) Transfer the tap water to a class A, to contain, graduated cylinder. Record the volume on the sample worksheet.

6. Method Blank (MB)

a) Prepare a separate MB for each set of up to 20 client samples.
b) Measure 1.0 L ± 0.2 L of Type 1 (Ultra-Pure DI) Laboratory Water using a graduated beaker and place into a 2 L separatory funnel. Record volume on batch sheet.
c) Proceed to Step 1b in Section 9.1.2.

7. Laboratory Control Sample (LCS)

a) Prepare a separate LCS for each set of up to 20 client samples.
b) Measure 1.0 L ± 0.2 L of Type 1 (Ultra-Pure DI) Laboratory Water using a graduated beaker and place into a 2 L separatory funnel. Record volume on batch sheet.
c) Proceed to Step 1b in Section 9.1.2.

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8. Spike Extraction/Native Standard in to 1 mL Acetone

a) Pipette, using a disposable glass pipette, ~ 1 mL of Acetone into a 15 mL disposable C-tube, this is not quantitative.
b) For every sample and QC sample, spike using the appropriate syringe and volume of Extraction Standard in to a C-Tube as per Section 7.2 Standards.
c) For the LCS and MS (if required), spike using the appropriate syringe and volume of Native Standard in to the corresponding C-Tube as per Section 7.2 Standards.
d) Vortex the C-tube for 5 seconds

9.1.2 Extraction Procedures

Note: The default sample size is 1 L. For sample sizes larger than 1 L the volume of the extraction solvent portions must be adjusted (100 mL portions per 1 L of sample). If sample size is less than 1 L defer to the Supervisor for further instruction.

1. Liquid-Liquid Extraction

a) Quantitatively transfer the 1 mL Acetone spiked with Extraction Standard (and Native Standard – if required) to the corresponding sample bottle with 2x 1 mL ±0.5 mL acetone rinses using a disposable pipette . Cap and gently invert to mix
b) Pour the entire contents of the sample bottle in to a 2 L separatory funnel. Be sure that the bottle has been marked as per Step 5 in Section 9.1.1 before the sample is transferred to the separatory funnel.

c) Allow the sample to equilibrate for 10 minutes before proceeding to the next step.
d) Shake 100 mL ± 5 mL of DCM, measured using a glass graduated beaker, in the original sample bottle for 10 seconds, then add this to the separatory funnel. Place the empty original sample container in to the fume hood. This will be used to determine the original volume of the sample (Refer to Step 5, "Mark the Sample Level on the Original Bottle", in Section 9.1.1).
e) Vigorously shake the separatory funnel for 2 minutes, with periodic venting.
f) Allow the sample to sit for a minimum of 5 minutes to allow for good phase separation.
g) If good phase separation cannot be achieved after 5 minutes, a small amount of sodium chloride can be added to help break the emulsion.
h) Drain the DCM layer (bottom layer) from the separatory funnel, through the Allihn filter containing sodium sulphate, in to a 500 mL Florence flask.
i) Repeat steps 4 to 8 two more times.
j) Collect a rinse of 50 mL ± 5 mL DCM through the Allihn filter using a graduated beaker, adding it to the previously collected extract.
k) Proceed to Section 9.5 "Clean-up and Final Reductions of Extract" for volume reduction and cleanup steps.

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9.2 Extraction of Solids and Biota Samples

Conventional Soxhlet procedures can handle sample sizes up to 30 g of tissue (or even 50 g of 'dry' solids) but is limited to approximately 1 g of lipid based upon the capacity of the Gel-Permeation Chromatography cleanup column.

Refer to 'BU-WI-3005 Food Sample Homogenization' for food prep instructions and 'BU-WI-3001 Subsampling and Homogenization of Samples for Organic Analysis' for solids homogenization procedures.

9.2.1 Pre-Extraction Procedure

1. Method Blank (MB)

a) Prepare a separate MB for each set up of 20 client samples.
b) Place the sample jar on to a two place balance, and tare the balance
c) Subsample 30-40g of baked sodium sulphate.

2. Laboratory Control Sample (LCS)

a) Prepare a separate LCS for each set up of 20 client samples.
b) Place the sample jar on to a two place balance, and tare the balance
c) Subsample 30-40g of baked sodium sulphate.

3. Duplicate Sample (DUP)

a) Prepare a duplicate for each batch consisting of 4 or more samples or one every 5th batch when these batches contain less than 4 samples.
b) Subsample duplicate as described in Step 6 "Percent Moisture Determination" and 7 "Drying Samples with Sodium Sulphate".

4. Reference Material (RM) – If required

a) Prepare a separate RM for each set up of 20 client samples if required (See Section 8.0 "Quality Control").
b) Matching the sample matrix, Subsample as described in Step 7 "Drying Samples with Sodium Sulphate".

5. Matrix Spike (MS) – If required

a) Prepare a separate MS for each set up of 20 client samples if required (See Section 8.0 "Quality Control").

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b) Subsample a duplicate for matrix spiking as described in Step 6 "Percent Moisture Determination" and 7 "Drying Samples with Sodium Sulphate".

6. Percent (%) Moisture Determination

Refer to BU-TM-1200 Moisture Content by Gravimetry.

7. Drying Samples with Sodium Sulphate

a) Place the sample jar on to a two place balance, and tare the balance.
b) Subsample the amount of sample as required on the batch sheet ± 1.0 g (if sampling less than 10 grams defer to the Supervisor for acceptable subsampling range criteria) in to the jar and record the weight of the subsample to two decimal places. Refer to 'BU-WI-3001 Subsampling and Homogenization of Sample for Organic Analysis' for proper sub-sampling and solid homogenization procedures and 'BU-WI-3005 Food Sample Homogenization' for food prep instructions.
c) Thoroughly mix the sample and sodium sulphate mixture with a spatula.
d) Add additional sodium sulphate and mixing until a dry, free-flowing mixture is obtained.

9.2.2 Extraction Procedures

1. Soxhlet Extraction

Note: Routine soxhlet extraction is appropriate for dry solids, or for wet solids pre-treated according to Step 7 (Drying Samples with Sodium Sulphate) of Section 9.2.1.

a) Refer to 'BU-WI-3003 Soxhlet Setup Procedure' for the proper setup of the soxhlet extraction apparatus.
b) Transfer sample to pre-soxhleted cellulose thimble.
c) Spike, using the appropriate syringe and volume of Extraction Standard (refer to Section 7.2 of BU-TM-1103 OC Pesticides & BU-TM-1121 8276 Toxaphene), on to the samples and QC samples. Also, spike the Native Standard into LCS and MS (if required) as per Section 7.2 of BU-TM-1103 OC Pesticides & BU-TM-1121 8276 Toxaphene.
d) Soxhlet extract sample with 400 mL ± 10 mL of dichloromethane (DCM), for a minimum time of 16 hours. Heat Setting = Medium or less.
e) Turn the soxhlet off and allow it to cool to room temperature.
f) Remove the condenser, and tip the soxhlet so that any solvent remaining in the soxhlet body will siphon in to the boiling flask.
g) Proceed to Section 9.5 "Clean-up and Final Reductions of Extract" for cleanup and volume reduction steps.

9.3 Extraction of Oil Samples

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9.3.1 Pre-Extraction

1. Method Blank (MB) - Reagent

a) Prepare a separate MB for each set up of 20 client samples.
b) This will be 5mL of 1:1 DCM:Hexane marked in a GPC autosampler c-tube.

2. Method Blank (MB) – Corn Oil

a) Prepare a separate MB for each set up of 20 client samples.
b) Take a GPC autosampler c-tube and tare on balance.
c) Add 1 gram of Corn Oil and record on batch sheet.
d) Dilute the Corn oil up to 5mL with 1:1 DCM:Hexane marked in a GPC autosampler c-tube.

3. Laboratory Control Sample (LCS)

a) Prepare a separate LCS for each set up of 20 client samples.
b) Take a GPC autosampler c-tube and tare on balance.
c) Add 1 gram of Corn Oil and record on batch sheet.
d) Dilute the Corn oil up to 5mL with 1:1 DCM:Hexane marked in a GPC autosampler c-tube.

4. Duplicate Sample (DUP)

a) Prepare a duplicate for each batch consisting of 4 or more samples or one every 5th batch when these batches contain less than 4 samples.
b) Subsample duplicate as described in Section 9.3.2 Step 1 “Subsampling”.

5. Reference Material (RM) – If required

a) Prepare a separate RM for each set up of 20 client samples if required (See Section 8.0 “Quality Control”).
b) Matching the sample matrix, Subsample as described in Section 9.3.2 Step 1 “Subsampling”.

6. Matrix Spike (MS) – If required

a) Prepare a separate MS for each set up of 20 client samples if required (See Section 8.0 “Quality Control”).
b) Subsample a duplicate for matrix spiking as described in Section 9.3.2 Step 1 “Subsampling”

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9.3.2 Extraction Procedure

1. **Subsampling**
   a) Using a GPC auto-sampler tube, tare the vial on the two-place balance.
   b) Subsample 1 gram of the sample into the vial following BU-WI-3001 Subsampling and Homogenization of Sample for Organic Analysis.

2. **Gel-Permeation Chromatography**
   a) See BU-WI-3017 Gel Permeation Chromatography for operation procedures.
   b) Proceed to Section 9.5 "Clean-up and Final Reductions of Extract" for cleanup and volume reduction steps.

9.4 Extraction of Serum Samples

9.4.1 Pre-Extraction Procedures

a) Place a 250 mL pre-cleaned jar onto a two-place balance. Tare the balance.
b) Subsample the required amount of sample, as indicated on the batch sheet, into the jar. Record the weight of the subsample. **Note**: This procedure will work for 2g – 20g of serum sample.
c) Spike the appropriate volume of Extraction Standard, as indicated on the batch sheet, on to the sample. Refer to 'BU-WI-3002 Standards Prep, Spiking, and Witnessing' for proper spiking procedures.
d) Add a 1:1 ratio of Methanol to serum weight to the sample.
e) Tare the balance.
f) Weigh 60g to 70 g ± 5g of sodium sulphate into the jar for routine extraction.
g) Thoroughly mix the sample and sodium sulphate mixture with a spatula.
h) Add additional sodium sulphate and mix until a dry, free-flowing mixture is obtained.

9.4.2 Extraction Procedures

9.4.2.1 Soxhlet Extraction

a) Transfer sample to pre-cleaned thimble and place in pre-cleaned soxhlet body. Refer to 'BU-WI-3003 Soxhlet Setup Procedure' for thimble cleaning procedure.
b) Continue following 'BU-WI-3003 Soxhlet Setup Procedure' using DCM to extract samples.
c) Proceed to Section 9.5 "Clean-up and Final Reductions of Extract" for cleanup and volume reduction steps.

9.5 Clean-up and Final Reductions of Extract

9.5.1 Solvent Exchange to Hexane

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a) Add 1mL of Iso-octane to the sample flask.
b) Using a roto-vap (max bath temp. = 30˚C) reduce the extract to approximately 3mL. Refer to 'BU-WI-3009 Rotary Evaporators'.
c) Transfer the extract to a disposable c-tube with 3x 2mL Hexane rinses.
d) Using an N-Evap (max bath temp. = 30˚C) reduce the extract down to approximately 1mL. Refer to 'BU-WI-3013 Nitrogen Evaporators'.

Refer to sample batchsheet as to which Column Clean-up to proceed with, if unsure consult supervisor.

9.5.2 Silicarb Column Clean-up

a) Place approximately 1.5 cm of glass wool in to the bottom of a disposable glass column having dimensions L = 40cm, I.D. = 15mm.
b) Using a permanent fine-tip marker, mark the levels of the various column packing materials as shown in **Figure 1** in the Appendix**.**
    i. Chroma-Carb is prepared using 24 grams of sodium sulphate and 1 gram of Carbon Norit placed into a 60mL vial. The tube is then placed on the paint shaker for 3.5 hours. The mixture is then sifted to remove large carbon pieces.
    ii. 2% deactivated silica is prepared on a weight by weight basis of 98 grams of silica and 2 grams of Type 1 (Ultra-Pure DI) Laboratory Water placed in a jar. The jar is then shaken very well to remove all clumps and let sit for one hour.
c) Place the column on to a support rack and rinse with 2x4mL Toluene, then 2x4mL Hexane. Allow column to air dry for 15 minutes before proceeding to next step.
d) Add each packing material to the column in the order and amount outlined in **Figure 1**, tapping after each addition to ensure the material is properly packed.
e) Elute the column with a 25mL Hexane rinse.
f) When the column has just stopped dripping, place a disposable 50mL C-tube under the column. This fraction is F1, and will be archived for three months.
Note: Check samples for presence of water. See BU-WI-3027 Water Troubleshooting.
g) Transfer the extract to the column, followed by 3x1mL Hexane rinses of the previous vessel. Add each rinse once the solvent level in the column has dropped to the top surface of packing.
h) Add 25mL of Hexane to the column.
i) Once the column has stopped dripping exchange the c-tube with a clean flask. This fraction is F2, and will be analyzed.
j) Add 250mL of 1:1 DCM:Hexane to the column.
k) Once the column has stopped dripping remove the c-tube and save the column.
l) Proceed to solvent reduction and robo-vialing steps.

9.5.3 Gel Permeation Chromatography

a) Refer to 'BU-WI-3017 Gel Permeation Chromatography'.
b) Once the run sequence is completed remove the c-tubes from the rack and proceed to Solvent Reduction and Robo-Vialing in Section 9.5.5.

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9.5.4 Cleanup for Toxaphene

a) See section 9.5.2 for Silicarb Cleanup.
   i. Note: the F2 elution for toxaphene is 50mL of 1:1 DCM:Hexane
b) Using an N-Evap (max bath temp. = 30˚C) reduce the extract down to approximately 1mL. Refer to 'BU-WI-3013 Nitrogen Evaporators'.
c) Build a Mini Acid Silica Column
   i. Place approximately 1cm of glass wool in the tip of the column attached to reservoir as per **Figure 2** in the Appendix. Using a permanent fine-tip marker, mark the levels of the various column packing materials as shown in **Figure 2**.
   ii. Place the column on to a support rack and, using a squeeze bottle, rinse one time with approximately 4 mL Toluene, then one time with approximately 4 mL Hexane. Allow column to air dry before proceeding to next step.
   iii. Add 1 cm of Sodium sulphate to the column. Tap the column to ensure the material is properly packed.
   iv. Add 1 cm of Neutral Silica to the column. Tap the column to ensure the material is properly packed. See BU-WI-3016 Acid Silica Column Preparation and Use for reagent prep instructions.
   v. Add 4cm of Acid Silica to the column. Tap the column to ensure the material is properly packed. See BU-WI-3016 Acid Silica Column Preparation and Use for reagent prep instructions.
   vi. Add another 1mL Neutral Silica followed by another 1mL of Sodium Sulphate to the column. Tap the column to ensure the material is properly packed.
d) Transfer the extract to the column followed by 3x1mL hexane rinses of the previous vessel. Add each rinse once the solvent level in the column has dropped to the top surface of packing.
e) Add 15mL DCM to the column.
f) Once the column has stopped dripping remove the c-tube and save the column.
g) Proceed to solvent reduction and robo-vialing steps.

9.5.5 Solvent Reduction and Robo-Vialing

a) Using an N-Evap (max temp=30˚C) to reduce the extract to combine the two c-tubes together using 3x1mL DCM rinses. A final volume of 1mL for each sample should be reached.

NOTE: For some projections a smaller final volume may be needed to achieve appropriate detection limits. Please consult AM for project requirements and ensure it is adhered to as described on the sample extraction work up sheet.

b) Mix sample with a vortex mixer, then transfer to a 2mL robo-vial without solvent rinses.

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c) Spike the c-tube with injection standard. Cap the vial and mix the samples with the vortex mixer.
d) Mark the extract level on the side of the vial.
e) Submit vials to instrumentation for analysis.

10.0 DATA CALCULATION AND REPORTING

10.1 Percent Moisture Calculation

Refer to BU-TM-1200 Moisture Content by Gravimetry.

11.0 REFERENCES

11.1 Primary References

US EPA Office of Water, Method 1699 (December, 2007).

11.2 Instrument Manuals

1. Mettler Toledo ML Model Manual
2. Mettler Toledo PB Model Manual
3. Sartorius Practum Model Manual

All above manuals can be found electronically in G:\Enviro\Library\Equipment diagrams and manuals\Balances

11.3 Supporting ALS Documents

1. NA-SP-0001 Definitions of Key Terms
2. BU-TM-1103 OC Pesticides
3. BU-TM-1121 8276 Toxaphene
4. BU-TM-1200 Moisture Content by Gravimetry
5. BU-WI-3000 Glassware Cleaning Proofing and Maintenance
6. BU-WI-3001 Subsampling and Homogenization of Samples for Organic Analysis
7. BU-WI-3002 Standards Prep Spiking and Witnessing
8. BU-WI-3003 Soxhlet Setup Procedure
9. BU-WI-3005 Food Sample Homogenization
10. BU-WI-3006 Chromacarb
11. BU-WI-3009 Rotary Evaporators
12. BU-WI-3013 Nitrogen Evaporators
13. BU-WI-3017 Gel Permeation Chromatography
14. BU-SP-0021 Waste Handling and Sample Disposal
15. BU-SP-0250 Control of Reagents and Reference Materials
16. BU-WI-3027 Water Troubleshooting
17. BU-WI-3016 Acid Silica Column Preparation and Use

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12.0 DEVIATIONS FROM THE PRIMARY REFERENCE

1. Method 1699 states to verify calibration of the GPC with a calibration solution after every 20 extracts. The samples have a d6-Methoxychlor as an extraction standard so this is tested on a per sample basis. When the GPC is re-packed or has maintenance, a GPC cutpoint proof is run to ensure all targets are eluting fully.

13.0 REVISION HISTORY

Version #	Date	Summary of Major Changes Made / By
01	14-June-10	Initial method creation and definition. KJames
02	12-Apr-12	Update to national template. TBabel
03	26-Feb-2014	Add all OCP prep instructions. / SEagle, KZwambag
04	27-Jan-2017	Sample handling & storage tables updated, MEOL #'s added, sodium chloride purchasing information added, standards section updated, QC prep instructions added, % moisture instructions removed, reference to BU-TM-1200 added, instructions for extraction of oil samples added, other minor updates throughout. / M.McPherson.
05	17-Jul-2017	Toxaphene prep instructions added. / J.Porter
06	5-Oct-2018	Corrected name of BU-TM-1121 throughout, expanded references in section 5, added MEOL and/or supplier information to section 6, updated section 11 to national format, added deviation #1. Minor corrections and formatting throughout. / R.Saxon

14.0 WASTE MANAGEMENT

Refer to 'BU-SP-0021 Waste Handling and Sample Disposal'. Waste information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

15.0 SAFETY

HAZARD ASSESSMENT		
Job Task	**Hazards**	**Preventive Measures**
Handling Organic Solvents	Eye damage	Wear appropriate safety glasses (with side shields) when working with solvents.
	Solvent adsorption through skin contact	Use appropriate personal protective equipment. Avoid skin contact with solvents. Wear a labcoat when handling solvents or working near solvents. Wear appropriate shoes (e.g. no open toes).
	Vapour inhalation	Handle solvents in a fumehood, except when they are contained in sealed vessels
Handling Glassware	Cuts	Wear gloves. Use appropriate care when handling glassware, especially pipettes.

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HAZARD ASSESSMENT		
Attaching glass pipettes to the ports in the nitrogen evaporator	This can be done in a way to jam the pipette into the port, therefore making it difficult to remove the pipette.	Do not apply force to try to remove the pipette as it may break and the analyst may sustain a laceration. Instead follow the TUBECON SOP and if necessary cut the silicon tubing on the port to release the pipette.
Moving Parts on roto-vap	Cuts, pinches, etc.	Exercise care. Lock-out before servicing.

16.0 APPENDICES

16.1 Appendix A: Figure 1 - SiliCarb Column
16.2 Appendix B: Figure 2 - Mini Acid Silica Column

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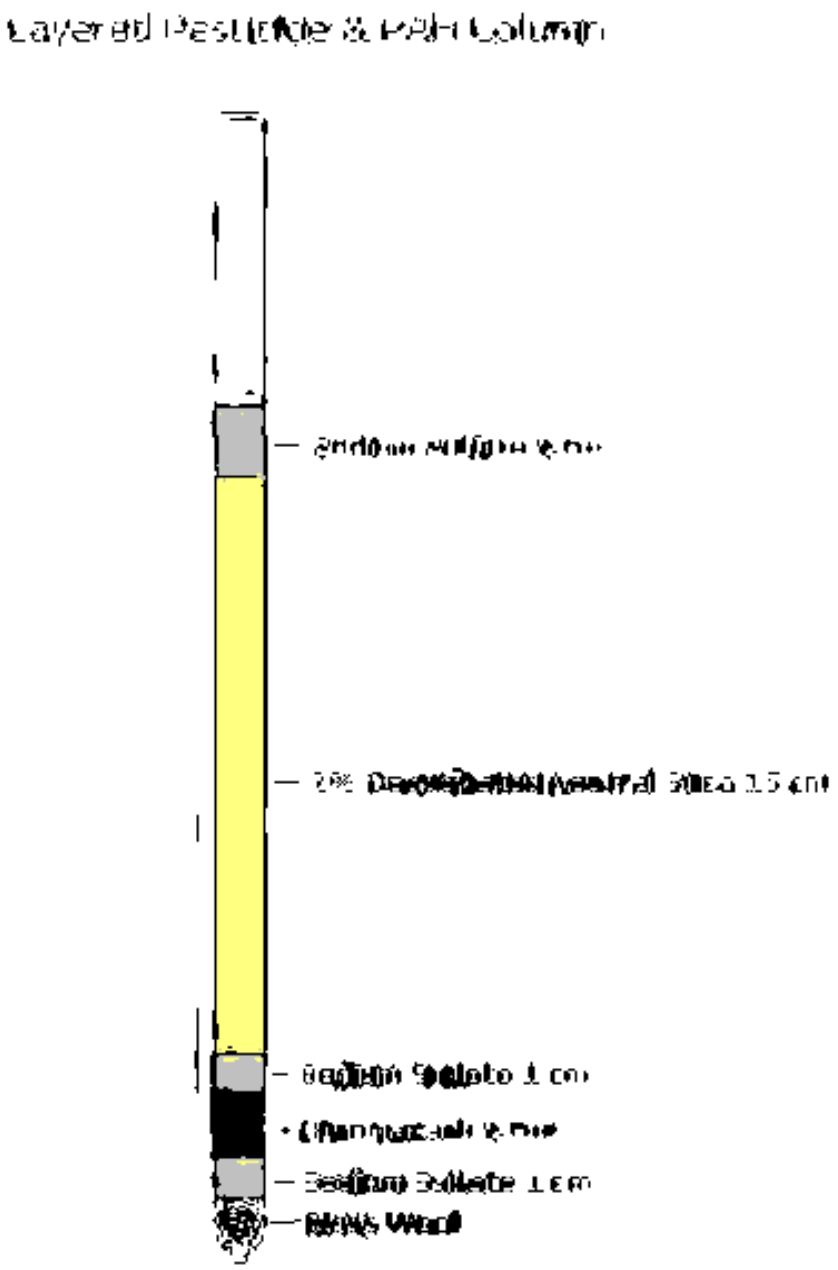
16.1 Appendix A: Figure 1 - SiliCarb Column



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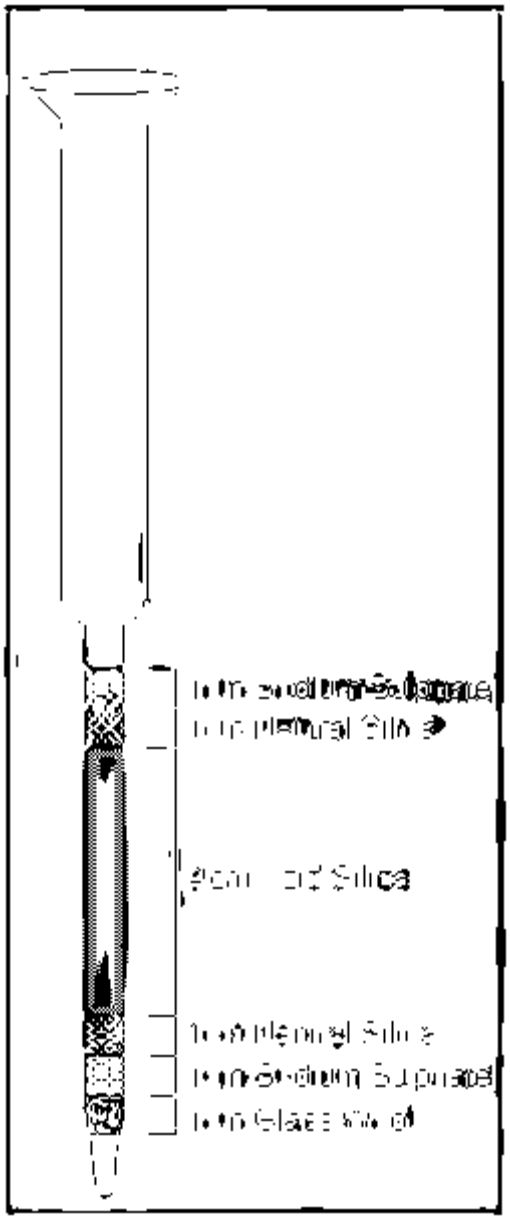
16.2 Appendix B: Figure 2 - Mini Acid Silica Column



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TOTAL AND DISSOLVED ORGANIC CARBON (TOC, DOC), TOTAL INORGANIC CARBON (TIC), AND TOTAL CARBON (TC) IN WATER

SM 5310C-2011; EPA 9060A; EPA 415.1

DOCUMENT I.D. GEN-TOC

Prepared By: _____ Date: 1/4/19
Department Manager, Jeff Coronado

Prepared By: _____ Date: 1/4/19
Quality Assurance Manager, Carl Degner

Approved By: _____ Date: 1/4/19
General Manager, Ambrose Hughey

Doc Control ID:	_____	Archived Date:	_____

ALS-Kelso SOP Annual Review Statement

SOP Code: GEN-TOC
Revision: 15

An annual review of the SOP listed was completed on (date): **02/26/20**

☒ The SOP reflects current practices and requires no procedural changes.

Supervisor: TH Date: 02/26/20

☐ Revision of the SOP is needed to reflect current practices. Draft revisions are listed below.

SOP Section Number	**Description of Revision Needed**	**Date Procedure Change Implemented**	**Supervisor Initials** Indicating Approval of Revision

ALS-Kelso SOP Annual Review Statement

SOP Code: GEN-TOC
Revision: 15

An annual review of the SOP listed was completed on (date): _____

☐ The SOP reflects current practices and requires no procedural changes.

Supervisor: Date:

☒ Revision of the SOP is needed to reflect current practices. Draft revisions are listed below.

SOP Section Number	**Description of Revision Needed**	**Date Procedure Change Implemented**	**Supervisor Initials** Indicating Approval of Revision
7.6	Add: Samples must be filtered and preserved within 48 hours of collection.	08/23/19	JC

1) Scope & Applicability

1.1 This procedure is applicable to the determination of Total Organic Carbon (TOC) in drinking, surface and saline waters, domestic and industrial wastewater using methods EPA 9060A, EPA 415.1,and Standard Methods 5310C-2011. The procedure may also be extended to certain domestic or industrial wastes.

1.2 This procedure may be modified for quantification of Dissolved Organic Carbon (DOC) where

1.3 DOC is determined from a filtered sample.

1.4 Normal operating parameters (i.e. 1 ml sample loop) yield a Method Reporting Limit (MRL) of 0.5 mg/L C. A 5 ml sample loop may be used to lower the MRL to 0.1 mg/L C. The data quality objectives for target analytes in water are presented in Table 2 and in the ALS Kelso DQO Table.

1.5 In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DOD ELAP. QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management (ADM-DOD)* may supersede the requirements defined in this SOP.

2) Summary of Procedure

2.1 Total Organic Carbon (TOC) is determined by measuring carbon dioxide released by chemical oxidation of the non-purgeable organic carbon in the sample. After the sample has been acidified and purged of inorganic carbon, sodium persulfate, a strong oxidizer, is added. This oxidant quickly reacts with non-purgeable organic carbon in the sample at 100°C to form carbon dioxide. When the reaction is complete, the carbon dioxide is purged from the solution, concentrated by trapping then thermally desorbed (200°C) and carried into a non-dispersive infrared detector that has been calibrated to directly display the mass of carbon dioxide detected. The resulting carbon mass in the form of carbon dioxide is the equivalent to the mass of organic carbon originally in the sample.

2.2 Total Inorganic Carbon is determined by carbon dioxide released by acidification of a sample. The pH of the sample is lowered; the carbonate and bicarbonate ions are then converted to carbon dioxide. This carbon dioxide is purged from the solution, concentrated by trapping, and detected as described for TOC.

3) Definitions

3.1 Batch - A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.1.1 Preparation Batch - A preparation batch is composed of one to twenty field samples, all of the same matrix, and with a maximum time between the start

of processing of the first and last samples in the batch to be 24 hours.

3.1.2 Analysis Batch - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration (initial or continuing verification) followed by sample extracts interspersed with calibration standards (CCBs, CCVs, etc.) The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria indicate an out-of-control situation.

3.2 Sample

3.2.1 Field Sample - An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.2.2 Laboratory Sample - A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.3 Quality System Matrix - The *matrix* of an environmental sample is distinguished by its physical and/or chemical state and by the program for which the results are intended. The following sections describe the matrix distinctions. These matrices shall be used for purpose of batch and quality control requirements.

3.3.1 Aqueous - Any groundwater sample, surface water sample, effluent sample, and TCLP or other extract. Specifically excluded are samples of the drinking water matrix and the saline/estuarine water matrix.

3.3.2 Drinking water - Any aqueous sample that has been designated a potable or potential potable water source.

3.3.3 Saline/Estuarine water - Any aqueous sample from an ocean or estuary or other salt-water source.

3.4 Method Blank (MB) - a solution of the laboratory prepared deionized water that is carried through analysis like a sample, to serve as a measure of contamination associated with laboratory storage, preparation, or instrumentation.

3.5 Filtration Blank - The filtration blank is an artificial sample composed of analyte-free water and is designed to monitor the introduction of artifacts into the filtration process. The filtration blank is carried through the entire filtration procedure.

3.6 Laboratory Control Samples (LCS) – The LCS is an aliquot of analyte free water or analyte free solid to which known amounts target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.7 Sample Duplicate - a second aliquot of a sample that is treated exactly the same throughout laboratory analytical procedures. The purpose is to verify the precision associated with the laboratory procedures. Matrix Spike - aliquots of sample to which known amounts of an analyte of interest has been added. These are treated exactly the same throughout laboratory analytical procedures. The purpose of a matrix spike is to determine whether the sample matrix contributes bias to the analytical results.

3.8 Calibration Standards - a solution of analytes prepared in the laboratory from stock standard solutions, diluted as needed, and used to calibrate the instrument response with respect to analytical concentration.

3.9 Independent Verification Standard (ICV) - A mid-level standard injected into the instrument after the calibration curve and prepared from a different source than the initial calibration standards. This is used to verify the validity of the initial calibration standards. The LCS when prepared from a different source will also serve as the ICV.

3.10 Laboratory Control Standards (LCS) - a solution of analytes prepared in the laboratory from stock standard solutions purchased or prepared independently from calibration standards.

3.11 Continuing calibration blank (CCB) - a blank solution of deionized water. CCB's are analyzed to verify that the instrument has not become contaminated during the course of the analytical run.

3.12 Continuing calibration verification standard (CCV) - a solution of prepared in the laboratory at approximately the midpoint of calibration curves. CCV's are analyzed to verify that the instrument performance has not changed during the course of the analytical run.

3.13 Rinse Blank (RB) - a solution of the laboratory prepared deionized water that is carried through analysis like a sample, to serve as a measure of carry-over from the previous sampling and analytical run.

4) Responsibilities

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

4.2 It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency is also the responsibility of the department supervisor/manager or designee.

5) Interferences

5.1 Carbonate and bicarbonate carbon are interferences under the terms of this test and must be removed or accounted for in the final calculations

5.2 This procedure is applicable only to homogenous samples that can be injected reproducibly by microliter type syringe or pipette. The opening of the syringe or pipette limits the size of particles which may be included in the samples. The Teledyne TOC Fusion™ analyzer can analyze samples with suspended solids up to 500 microns diameter.

5.3 Positive bias may be caused by contaminants in the gas, dilution water, reagents, glassware, or other sample processing hardware. The use of high purity reagents and gases help minimize interference problems. Materials may be demonstrated to be free from interference by running reagent blanks

5.4 Interference by non-CO2 gases: The infrared detector is sensitized to carbon dioxide and accomplishes virtually complete rejection of response from other gases which absorb energy in the infrared region. Trapping and desorption of carbon dioxide on

the molecular sieve trap isolates the component of interest and allows the complete absence of interference in the system from gases other than carbon dioxide.

6) Safety

6.1 All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personal protective equipment, such as, safety glasses, lab coat and the correct gloves.

6.2 Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in SDSs where available. Refer to the ALS Chemical Hygiene Plan and the appropriate SDSs prior to beginning this method.

6.3 Sodium Persulfate is a strong oxidizer and should be handled with extreme care.

6.4 Phosphoric Acid is a corrosive material should be handled with extreme care.

6.5 Potassium Biphthalate and Sodium Carbonate are chemical irritants and may cause eye burns.

7) Sample Collection, Containers, Preservation, and Storage

7.1 For most accurate analyses, sampling containers should be free of organic contaminants.

7.2 Sampling and storage of samples in glass bottles is preferable. If this is not feasible, sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples.

7.2.1 **Note:** A brief study performed at the EPA Laboratory indicated that distilled water stored in new, one quart cubitainers did not show any increase in organic carbon after two weeks exposure.

7.3 For samples requiring very low-level TOC analysis (below about 500 ppb C) attention to limiting contamination may be required. If possible, rinse bottles with sample before filling and carry field blanks through sampling procedure to check for any contamination that may occur. Collect and store samples in glass bottles protected from sunlight and seal with TFE-backed septa. Use certified clean sample vials for sampling and analysis. However if certified clean containers are not available or are found to be cleaned insufficiently further cleaning may be required. If necessary before use, wash bottles with acid, seal with Aluminum foil, and bake at 400˚C for at least one hour. Wash un-cleaned TFE septa with detergent, rinse repeatedly with organic free water, and wrap in aluminum foil and bake at 100˚C for one hour. Check performance of new or cleaned septa by running appropriate blanks. Preferably use thick silicone rubber-backed TFE septa with open ring caps to produce a positive seal. Less rigorous cleaning may be acceptable if the concentration range is relatively high. Check bottle blanks to determine effectiveness or necessity of cleaning.

7.4 Because of the possibility of oxidation or bacterial decomposition of certain components in aqueous samples, the time between sample collection and analysis should be minimized. In addition, the samples should be kept cool (4°C) and protected from sunlight and atmospheric oxygen.

7.5 In situations where analysis cannot be performed within two hours (2 hours) of sampling, the sample must be acidified (pH < 2) with Phosphoric or Sulfuric acid. Once preserved, samples must be analyzed within 28 days. Note that acid preservation invalidates any inorganic carbon determination on the samples.

7.6 Samples requiring DOC analyses should be filtered through a prewashed 0.45 micron glass microfiber membrane filter prior to acid preservation. A DI water filter blank should also be included with the filtration batch to determine potential for sample contamination from filter or filtration apparatus.

8) Apparatus and Equipment

8.1 TOC analyzer: Teledyne -Tekmar, Model TOC Fusion, S/N: US10165001.

8.2 Whatman 0.45µm glass microfiber membrane filter, or equivalent.

9) Standards, Reagents, and Consumable Materials

9.1 Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Standards, reagents and consumable material documentation shall indicate traceability to purchased neats or compounds. Refer to the SOP *Reagent/Standards Login and Tracking* (ADM-RTL) for the complete procedure and documentation requirements.

9.2 All stocks, working solutions and sample dilutions should be prepared using deionized water (DI) conforming to ASTM Type I or ASTM Type II reagent water. For more information on reagent water generation, refer to the related SOP, Operation and Maintenance of Laboratory Reagent Water Systems.

9.3 Potassium Biphthalate (KHP) stock solutions:

9.3.1 1000 ppm C stock solution is prepared by adding 2.128 g of KHP (previously dried to a constant weight at 105°C) into a 1000 ml volumetric flask. Dilute to volume with reagent water. Solution contains 1.0 ug C per ul.

9.3.2 5000 ppm C stock solution is prepared by adding 10.64 g of KHP (previously dried to a constant weight at 105°C) into a 1000 ml volumetric flask. Dilute to volume with reagent water. Solution contains 5.0 ug C per ul.

Note: Stock solution has a shelf life of six months after preparation. Sodium oxalate and acetic acid are not recommended as stock solutions.

9.3.3 Calibration standards used are 5 ppm and 50 ppm. The instrument is configured to make Standard dilutions.

9.3.4 Sodium Carbonate Stock solution (1000 ppm C) - Prepare stock solution by adding 8.826 g of Na_2CO_3 (previously dried to a constant mass at 105°C) to a

1000 ml volumetric flask. Dilute to volume with reagent water. Solution contains 1.0 ug C per ul.

9.3.5 Sodium Persulfate - Prepare solution of sodium persulfate by dissolving 100g $Na_2S_20_{8,}$ into (852 mL DI H_2O plus 36 mL H_3PO4), then purge with N_2 for 30 minutes before use. Reagent has a shelf life of one month.

9.3.6 Phosphoric Acid (21%) - Prepare 21% by volume solution of phosphoric acid by adding 150 mL of ACS reagent grade 85% H_3PO_4 to 450 mL reagent water. Reagent has a shelf life of one month.

9.3.7 Continuing Calibration Verification (CCV) - The CCV is prepared by diluting 10.0 mLs of 5000 ppm TOC stock solution 1000 mLs of deionized water in a Class "A" volumetric flask. Resulting concentration is 50.0 ppm. The instrument runs the CCV check standard at a 1:2 dilution (25 ppm).

9.3.8 Laboratory Control Sample (LCS) - The LCS is prepared from an ERA QC - Plus Demand solution. The true value is determined based on the lot number of the standard.

9.3.9 Gas Service: Nitrogen.

10) Preventive Maintenance

10.1 Daily Maintenance Checks

10.1.1 Verify the gas source is supplying an input pressure of 50 psi.

10.1.2 Verify that there is ample persulfate available for sample analysis. Verify that the persulfate has not expired.

10.1.3 Verify that there is ample acid available for sample analysis. Verify that the acid has not expired. Make sure the DI water supply is sufficient for sample analysis.

10.1.4 After the UV lamp has warmed up for 15 minutes, verify that the detector baseline is within the range of 0-5 Absorbance units (Abs). Perform the Detector Offset function if necessary.

10.1.5 Verify that the waste container has sufficient volume to contain the waste generated.

10.2 Weekly Maintenance Checks

10.2.1 Check the copper side of the halogen scrubber. When copper is discolored completely, replace both the copper and tin in the scrubber.

10.2.1 Make sure the two screws that attach the 7-port Valve to the Syringe Pumper are tight.

10.3 Monthly Maintenance Checks

10.3.1 Inspect and clean the reactor and sparger if necessary.

10.3.2 Flush sample transfer line with generous amounts of DI water. Inspect the

permeation dryer for damage and water accumulation.

10.4 Semi-Annually Maintenance Checks

10.4.1 Replace the O-rings in the UV reactor vessel.

10.5 For additional information refer to the Fusion™ Preventative Maintenance section of the User Manual, Page 8-2.

11) Procedure

11.1 TOC analyzer: Teledyne -Tekmar, Model TOC Fusion - Preparation and Analysis

11.1.1 Perform the required daily maintenance checks.

11.1.2 If the TOC TekLink™ software is not already in operation, launch the TOC TekLink™ software.

11.1.3 Login with the User Name: (Fusion 1) and Password: (Fusion1), and connect into the Fusion program.

11.1.4 Open the daily startup schedule, save the schedule to reflect the current date (m/d/y/, make any necessary adjustments, and click "Ready".

- TOC/DOC daily startup schedule: CAS_SALT_010711.
- Extended Reaction (for salt water) daily startup schedule, Extended Reaction 021711.
- TIC/DIC daily startup schedule: IC 030411.
- TOC Low Level daily startup schedule: CAS_High_Sensitivity.

11.1.5 After the UV lamp has warmed up for 15 minutes, verify that the detector baseline is within the range of the 0-5 Absorbance units (Abs).Perform the Detector Offset function if necessary.

Note: *If the instrument is allowed to sit idle for 20 minutes, it will automatically switch to standby mode*.

11.1.6 Click the start button to start the sequence.

11.1.7 The schedule should contain three Cleans, one Reagent/Acid Blank, and one rinse blank before the first CCV.

11.1.8 Scan the samples barcode into the Run Sequence and load the samples into the carousel. Ensure that the sample's positon on the schedule matches the number on the carousel.

11.1.9 An initial CCV is run after the Rinse Blank and must be analyzed following every tenth injection and at the end of the run. The CCV is a 25.0 ppm TOC Standard made from stock KHP solution. Recovery must be **90-110%** of the value (**91-106% for Arizona** samples). For low level analyses (i.e. 0.1ppm MRL), the CCV is a 5.0 ppm standard. Calculate the CCV recovery as follows:

%R = X/TV x 100

Where X = Measured concentration of the CCV
TV = True value of CCV

11.1.10 A Continuing Calibration Blanks (CCB) must be analyzed every 10 injections. CCB measured concentrations must be less than the MRL.

11.1.11 Sample Analysis

11.1.11.1 Once the UV lamp has warmed up for 15 minutes, and the detector baseline is within the range of 0.5 Absorbance units (Abs), the instrument is ready for analysis.

11.1.11.2 Load samples vials into the autosampler carousel according to the analytical run sequence shown below. Thoroughly shake TOC samples before loading. Click the start button on the schedule to begin analysis.

11.1.12 When performing method 5310C and EPA 415.1, analyze all environmental samples in duplicate. .

11.1.13 When performing method 9060A, analyze all samples in quadruplicate.

12) Quality Assurance/Quality Control Requirements

12.1 Initial Precision and Recovery Validation

12.1.1 The ability of each analyst/instrument to generate acceptable accuracy and precision must validated and documented before analysis of samples begins, or whenever significant changes to the procedures have been made To do this, four water samples are spiked with the LCS spike solution, then prepared and analyzed. Method criteria must be met for these results.

12.2 Method Detection Limits and Method Reporting Limits

12.2.1 A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike seven blank matrix (water or soil) samples with MDL spiking solution at a level below the MRL. Follow the analysis procedures to analyze the samples.

12.2.2 Calculate the average concentration found (x) in µg/mL, and the standard deviation of the concentrations (s) in µg/mL for each analyte. Calculate the MDL for each analyte. Refer to the ALS *SOP Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification (CE-QA011).* The MDL study must be verified annually.

12.3 Limits of Quantification (LOQ)

12.3.1 The laboratory must establish a LOQ for each analyte as the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements. Analysis of a standard or extract prepared at the lowest point calibration standard provides confirmation of the established sensitivity of the method. Refer to the ALS SOP *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification (*CE-QA011*).*

12.3.2 The Method Reporting Limits (MRLs) used at ALS are the routinely reported lower limits of quantitation which take into account day-to-day fluctuations in instrument sensitivity as well as other factors. These MRLs are the levels to which ALS routinely reports results in order to minimize false positive or false negative results. The MRL is normally two to ten times the method detection limit.

12.4 Ongoing QC Samples each sample batch (20 or fewer samples) required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project specific quality assurance plans (QAPP). General QC Samples are:

12.4.1 Method Blank (MB)

12.4.1.1 A method blank is extracted and analyzed daily with every batch of 20 (or fewer) samples to demonstrate that there are no method interferences. If the method blank shows any hits above the reporting limit, corrective action must be taken. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and reanalysis. For some project specific needs, exceptions may be noted and method blank results above the MRL may be reported for common lab contaminants.

12.4.2 Laboratory Control Sample (LCS)

12.4.2.1 A Laboratory Control Sample (LCS) for SM 5310C and EPA 415.1 must be analyzed with each batch of 20 or fewer samples. The LCS is prepared from a standard which is an independent source from the calibration standards. Acceptance criteria are given in Table 2. This statistically derived acceptance limit is subject to change as limits are updated.

12.4.2.2 When performing Method 9060 analysis, the second source LCS must be analyzed every 15 samples rather than every 20 samples.

12.4.2.3 Calculate the LCS recovery as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

12.4.3 Sample Duplicates (DUP)

12.4.3.1 A sample duplicate or matrix spike duplicate (MSD) must be analyzed with every analytical batch.

12.4.3.2 Calculate Relative Percent Difference (RPD) as:

$$\%\,RPD = \frac{|R1 - R2|}{(R1 + R2)/2} \times 100$$ Where R = Result

12.4.3.3 The RPD is calculated as follows:

$$\frac{Hi - Lo}{Avg.} \times 100$$

12.4.4 The percent RPD for EPA 9060A and EPA 415.1 must be $\leq$**20%**. This statistically derived acceptance limit is subject to change as limits are updated. For SM 5310C, all duplicates must be within **10%** RPD.

Relative Percent Difference calculation:

$$\%\ RPD = \frac{(S - D)}{((S + D)/2)}$$

where: S = Initial sample result
D = Duplicate sample result

12.4.5 Matrix Spikes

12.4.5.1 For SM 5310C and EPA 415.1, analyze one matrix spike sample (MS) for every analytical batch of twenty samples.

12.4.5.2 Method 9060A analyze one matrix spike sample (MS) for every analytical batch of ten samples.

12.4.5.3 Spike 50 ul of 5000 ppm KHP stock solution to 10.0 mLs of sample. For low level analysis, spike 50 ul of 1000 ppm KHP stock solution to 10.0 mLs of sample. Acceptance criteria are given in Table 2. This statistically derived acceptance limit is subject to change as limits are updated.

Calculate percent recovery as follows:

$$Matrix\ Spike\ Recovery = \frac{Spiked\ Sample - Sample}{Spike\ Added} \times 100$$

13) Data Reduction and Reporting (or Documentation and Records)

13.1 Refer to the SOP for Data Reporting and Report Generation for reporting guidelines.

13.2 Preliminary results are reviewed to determine if dilutions are required. Sample information is transferred to an Excel spreadsheet for calculations (see R:\WET\ANALYSES\TOC\DATA). Instrument baseline is determined by taking the average of all Method Blanks, CCB's, (see R:\WET\ANALYSES\TOC\TOC_CBA1.SPD). Sample concentration is corrected by subtracting calculated blank average (CBA) from instrument response. Concentration and sample identification number are highlighted for reporting purposes.

13.3 For 5310C, report the result from a single analysis. For 9060A, report both the average and the range from the quadruplicate analyses.

13.4 It is the operators' responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified in procedures section of the SOP. Average, RPD, spike level and spike recovery are entered on spreadsheet (see append. B) for corresponding samples. All data will be initialed, dated and attached to required data quality worksheet.

13.5 Reports are generated in the ALS LIMS by compiling the SMO login, sample prep database, instrument date, and client-specified report requirements (when specified). This compilation is then transferred to a file which Excel© uses to generate a report. The forms generated may be ALS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.6 As an alternative, reports are generated using Excel© templates located in R:\WET\FORMS. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The results are then transferred, by hand or electronically, to the templates the saved to R:\WET\WIP.

13.7 Data Review and Assessment

13.7.1 Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the *SOP for Laboratory Data Review Process* for details. The person responsible for final review of the data report and/or data package should assess the overall validity and quality of the results and provide any appropriate comments and information to the Project Chemist to inclusion in the report narrative.

14) Method Performance

14.1 The accuracy and precision of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four LCS aliquots are prepared and analyzed. The average percent recovery must meet the laboratory control sample acceptance limits.

14.2 The method detection limit (MDL) is established using the procedure described in the SOP CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of*

Detection and Quantification. Method Reporting Limits are established for this method based on MDL studies and as specified in the ALS Quality Assurance Manual.

15) Pollution Prevention and Waste Management

15.1 It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

15.2 The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Lab Waste Management Plan.

16) Corrective Actions for Out-of-Control Data or Unacceptable Data

16.1 Refer to the SOP for *Non Conformance and Corrective Action* (CE-QA008) for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

16.2 Handling out-of-control or unacceptable data

16.2.1 On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, run logs, for example.

16.2.2 Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision.
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels.
- Sample holding time missed due to laboratory error or operations.
- Deviations from SOPs or project requirements.
- Laboratory analysis errors impacting sample or QC results.
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc.).
- Sample preservation or handling discrepancies due to laboratory or operations error.
- Customer inquiries concerning data quality or services (when applicable). NCAR not required for simple corrections with no impact to the client.
- Data errors reported to clients, non-conforming re-checks.
- Deficiencies found during internal or external audits.
- Login errors or shipping errors.
- IT issues if there is a significant impact to a client.
- Turnaround time complaints.

17) Training

17.1 Review literature Review this SOP. Also review the applicable SDS for all reagents and standards used. Following these reviews, observe the procedure performed by an experienced analyst.

17.2 The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.3 Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

17.4 Training is documented following the *ALS-Kelso Training Procedure* (ADM-TRAIN). When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor acknowledges that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18) Method Modifications

18.1 There are no known modifications in this laboratory standard operating procedure from the reference method.

19) Summary of Changes

19.1 Reformatted SOP to current ALS format.

19.2 Updated safety references and definitions.

19.3 Miscellaneous format revisions and typographical/grammatical corrections.

19.4 Section 8: Model 1010 Total Organic Carbon Analyzer: removed from the equipment list and when referenced in the SOP.

TOC analyzer: Teledyne -Tekmar, Model TOC Fusion, - was added into numerous sections throughout the SOP.

19.5 Section 9: Updated calibration standards; sodium persulfate preparation; removed the ICV from the analysis.

19.6 Section 11: Numerous edits throughout the section.

19.7 Added EPA 415.1 into the SOP, which had previously been previously removed.

20) References and Related Documents

20.1 U.S. Environmental Protection Agency, Total Organic Carbon, Method 9060A, Revision 1 November 2004.

20.2 Total Organic Carbon, Combustion-Infrared Method, and 5310C. Standard Methods for the Examination of Water and Wastewater, 20th ed., 1998.

20.3 Organic Carbon, Total (Combustion Or Oxidation), Method 415.1.G:\QA\Methods\EPA\415.1.pdf.

20.4 TNI Standard, Volume 1- 2009.

20.5 DoD Quality Systems Manual for Environmental Laboratories. Current version.

20.6 Updated Table 1.

TABLE 1: Summary of Corrective Actions

Method Reference	Control	Specification and Frequency	Acceptance Criteria	Corrective Action
SM 5310C 9060, 415.1	Linearity verification	Annually	R2 ≥ 0.995	Correct problem then repeat ICAL
SM 5310C 9060, 415.1	ICV	After ICAL, prior to sample analysis	90-110%	Correct problem and verify second source standard; rerun second source verification; If fails, correct problem and repeat initial calibration.
SM 5310C 9060, 415.1	CCV	Prior to sample analysis, every 10 injections and end	± 10% Diff	Correct problem then repeat CCV or repeat ICAL
SM 5310C 9060, 415.1	CCB	Prior to sample analysis, every 10 injections and end	<MRL	If target exceeds MRL, reanalyze to determine if instrument was cause.
SM 5310C 9060, 415.1	Method Blank	Include with each analysis batch (up to 20 samples)	<MRL	If target exceeds MRL, reanalyze to determine if instrument was cause. If still noncompliant then: Re-extract or reanalyze samples containing contaminate, unless samples contain > 20x amount in blank.
SM 5310C, 415.1	Laboratory Control Sample	Include with each analysis batch (up to 20 samples)	See DQO	If exceeds limits, re-extract and re-analyze
9060	Laboratory Control Sample	Include with each analysis batch (up to 15 samples)	See DQO	If exceeds limits, re-extract and re-analyze
SM 5310C, 415.1	Matrix Spike	Include with each analysis batch (up to 20 samples)	See DQO	Evaluate data to determine if the there is a matrix effect or analytical error
9060	Matrix Spike	Include with each analysis batch (up to 10 samples)	See DQO	Evaluate data to determine if the there is a matrix effect or analytical error
SM 5310C,	Sample Duplicates	All samples in batch	≤ 10 % RPD	Re-homogenize and re-analyze if result is > 5 X the MRL
415.1	Sample Duplicates	All samples in batch	≤ 20 % RPD	Re-homogenize and re-analyze if result is > 5 X the MRL
9060	Sample Quadruplicate	All samples in batch	≤ 20 % RSD	Re-homogenize and re-analyze if result is > 5 X the MRL

DETERMINATION OF METAS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY

DOCUMENT ID: MET-6020, REV 19.0

Prepared By: _____ Date: 12/2/20
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Prepared By: Kurt Clarkson Date: 12/2/2020
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Approved By: _____ Date: 12/2/20
Laboratory Director, Charles (Pat) Byrne

1) Scope & Applicability

1.1 This procedure is used to determine the concentrations of certain elements in water, soil, tissues, aqueous and non-aqueous wastes, and sediment samples using EPA Method 6020B. The Kelso DQO table indicates analytes that are typically determined by this procedure and lists the standard Method Reporting Limits (MRLs) for each analyte in water and soil. Project-specific MRLs may apply, and if lower than standard MRLs, it is demonstrated through method detection limit determinations and analysis of MRL standards that the MRL is achievable. Method Detection Limits are referenced in the laboratory DQO tables and may change as new studies are performed.

1.2 The complexity of the technique generally requires outside study of appropriate literature as well as specialized training by a qualified spectroscopist. The scope of this document does not allow for the in-depth descriptions of the relevant spectroscopic principles required for gaining a complete level of competence in this scientific discipline.

1.3 In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DoD ELAP. QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management* (ADM-DOD5) may supersede the requirements defined in this SOP.

2) Summary of Procedure

2.1 Prior to analysis, samples must be digested using appropriate sample preparation methods. The digestate is analyzed for the elements of interest using ICP-mass spectrometry (ICP-MS).

2.2 Methods 6020B describe the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3) Definitions

3.1 For environmental laboratory quality definitions, guidance on analytical calibration and sample batches, refer to the SOP for Sample Batches, ADM-BATCH.

4) Responsibilities

4.1 It is the responsibility of the analyst to perform the analysis according to this SOP

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

4.2 It is the responsibility of the department supervisor/manager to document analyst training and method proficiency, as described in the ALS-Kelso *SOP for Training Procedure* (ADM-TRAIN).

5) Interferences

5.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). A data system must be used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Attention should be given to circumstances where very high ion currents at adjacent masses may contribute to ion signals at the mass of interest. Matrices exhibiting a significant problem of this type may require resolution improvement, matrix separation, or analysis using another isotope.

5.2 Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature. Refer to Method 6020B for further discussion.

6) Safety

6.1 Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in SDSs where available. Refer to the ALS Chemical Hygiene Plan and the appropriate SDSs prior to beginning this method.

6.2 Nitric Acid and Hydrochloric Acid is used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield, safety goggles, lab coat and gloves must be used while pouring acids. When working with solutions less than 20%, then a lab coat, goggles and gloves must always be worn.

6.3 High Voltage - The RF generator supplies up to 2000 watts to maintain an ICP. The power is transferred through the load coil located in the torch box. Contact with the load coil while generator is in operation will likely result in death. When performing maintenance on the RF generator, appropriate grounding of all HV capacitors must be performed as per manufacturer.

6.4 UV Light - The plasma is an intense source of UV emission, and must not be viewed with the naked eye. Protective lenses are in place on the instrument. Glasses with special protective lenses are available when direct viewing of the plasma is necessary.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Aqueous samples are typically collected in plastic containers. Aqueous samples are preserved with nitric acid (pH<2), then stored at room temperature from

receipt until digestion. Soil or solid samples may be collected in plastic or glass jars. Non-aqueous samples are refrigerated at 4 ± 2°C from receipt until digestion.

7.2 Samples are prepared via procedures in SOPs MET-DIG, MET-3020A, MET-3050, MET-3051M, MET-3052M, or MET-TDIG depending on matrix and project specifications.

7.3 Digestates are stored in the appropriate containers. Following analysis, digestates are stored until all results have been reviewed. Digestates are neutralized prior to disposal through the sewer system, 2 weeks after data is reviewed.

8) Standards, Reagents, and Consumable Materials

8.1 All standards are prepared from NIST traceable standards. The expiration dates are assigned according to the EPA method and the vendor's assigned expiration dates.

8.1.1 1000 ppm Single Element Stock Standard Solutions: Each stock standard is stored at room temperature on shelves located in room 113 of the metals lab. The manufacturer, lot number, and expiration date of each stock standard is recorded in a bound logbook also located in room 113. Additionally each stock standard is given a unique, identifying name.

8.1.2 Intermediate Standard Solutions: Intermediate mixed stock solutions are made from the individual stock standards described above. The individual component of each mixed solution is recorded in a bound logbook located in the ICP-MS laboratory and mixed solution is given a unique, identifying name. The expiration date for the intermediate standard is the earlier of any one of its stock components.

8.1.3 Calibration Standards: Calibration standards are made fresh daily from the intermediate standard solutions. Each individual intermediate standard used in the calibration standard is recorded in a bound logbook located in the ICP-MS laboratory, and the calibration standard solution is given a unique, identifying name. The calibration standards unique name is used on the raw data to link the data to the subsequent prepared standards and ultimately the original purchased stock standard.

8.2 Standards Preparation

8.2.1 Expiration of all standard solutions defaults to the earliest expiration date of an individual component unless otherwise specified.

8.2.2 Calibration Standards

The calibration standard is prepared from two intermediate stock solutions. These solutions are prepared in acid rinsed 1000 mL Class A volumetric flasks following the formulations laid out on the attached example standard sheet (see Attachments). The working calibration standard is made daily by aliquoting 2.5 mL of each of the intermediate solutions in to a 100 mL Class A volumetric flask and diluting to volume with DI water and the appropriate acid(s). This standard is also used as

the Continuing Calibration Verification (CCV).

8.2.3 Initial Calibration Verification (ICV)

8.2.3.1 The ICV intermediate stock solution is prepared in an acid rinsed 100 mL Class A volumetric flask. The solution is prepared by adding 2.0 mL of Inorganic Ventures QCP-CICV-1, 1.0 mL of QCP-CICV-3, 0.5 mL of 1000 ppm Boron, Bismuth, Molybdenum, Strontium, Titanium, and Uranium stock solutions, 0.25 mL of 1000 ppm Antimony stock solution and diluting to volume with the appropriate acid matrix.

8.2.3.2 The working ICV solution is prepared by aliquoting 0.5 mL of the mixed ICV intermediate solution and 0.25 mL of 10 ppm Tin standard into an acid rinsed 100 mL Class A volumetric flask and diluting to volume with the appropriate acid matrix.

NOTE: The ICV solution is not at the midpoint of the linear range which may be as high as 1000 µg/L for some elements. The ICV solution used is a premixed standard purchased from Inorganic Ventures and contains the elements of interest between 2.5 and 100 µg/L. This solution provides calibration confirmation at more representative levels, given that most ICP-MS analyses are quantifying analytes in the low-ppb to sub-ppb range.

8.2.4 Interference Check Solutions (ICSA and ICSAB)

8.2.4.1 The ICSA is prepared in an acid rinsed 50 mL Class B volumetric flask by aliquoting 1.0 mL of Elements ICSAm (CS-CAK02) and 0.250 mL of 10 ppm molybdenum solutions and diluting to volume with the appropriate acid matrix.

8.2.4.2 The ICSAB is prepared in an acid rinsed 50 mL Class B volumetric flask by aliquoting 1.0 mL of Elements ICSAm (CS-CAK02), 0.125 mL of Inorganic Ventures 6020ICS-9B, and 0.250 mL of 10 ppm Molybdenum solutions and diluting to volume with the appropriate acid matrix.

8.2.5 Post-digestion spikes are performed by adding appropriate amounts of the calibration intermediate solutions to aliquots of the sample digestate. The volumes of each standard used vary based on the native concentrations found in the field samples. Refer to the post-digestion spike in Section 12 for details.

8.2.6 Refer to the appropriate digestion SOP for details of LCSW and matrix spike solution composition and preparation.

8.2.7 Tuning / Mass Calibration Solution

8.2.7.1 A 1ppm intermediate solution containing Be, Bi, Ce, Co, In, Li, Pb, Mg, and U is prepared by adding 1.0 mL of each from 1000

ppm stock standards to an acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric acid. The expiration date for the intermediate solution is the earlier of any one of its stock components.

8.2.7.2 The working solution is prepared in depending upon the instrument:

- For the Agilent: a 10 ppb tune/mass calibration solution is prepared by adding 10 mL of intermediate solution to an acid rinsed 1000 mL volumetric flask and diluting to volume with the appropriate acid matrix.
- For the NexION (K-ICP-MS-04) instrument a 2.0 ppb tune/mass calibration solution is prepared by adding 2.0 mL of intermediate solution to an acid rinsed 1000 mL volumetric flask and diluting to volume with the appropriate acid matrix.

The expiration date for this solution is taken from the intermediate stock above.

8.3 Internal Standards Stock Solution – Prepare solutions by adding appropriate amounts of each 1000 ppm single element stock solution to an acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric. The internal standard solution is teed in by the peristaltic pump and used for the entire analytical sequence. The typical solutions are:

- Agilent Instrument: 1 ppm, Sc, Y, Ge, Ce, Tm, In, Lu, Th; 0.2 ppm ^{6}Li.
- NexION instrument: 30 ppb In, Tm, Lu, Th; 60 ppb ^{6}Li, Rh, Au; 75 ppb Sc; 100 ppb Ga, Y; 500 ppb Ge.

8.4 Additional Reagents

8.4.1 Reagent water, ASTM Type II.

8.4.2 All stocks, working solutions and sample dilutions should be prepared using deionized water (DI) conforming to ASTM Type I or ASTM Type II reagent water. For more information on reagent water generation, refer to the related SOP, Operation and Maintenance of Laboratory Reagent Water Systems.

8.4.3 "OmniTrace Ultra" Concentrated Nitric Acid (EM Science # NX0408-2).

8.4.4 Argon (Airgas Industrial Grade – 99.999% pure, bulk delivered).

9) Apparatus and Equipment

9.1 ICP/MS Instruments

9.1.1

Instrument:	NexION 300D
Nebulizer:	PFA-ST Microflow
Spray Chamber:	Cyclonic, Peltier-cooled
Cones:	Nickel Sampler (1.0 mm orifice)

		Nickel Skimmer (0.75 mm orifice)
9.1.2	Instrument:	Agilent 7700
	Nebulizer:	MicroMist
	Spray Chamber:	Double Pass quartz spray chamber
	Cones:	Nickel Sampler (1.0 mm orifice)
		Nickel Skimmer (0.75 mm orifice)
9.1.3	Instrument:	Agilent 7800
	Nebulizer:	MicroMist
	Spray Chamber:	Cyclonic, Peltier-cooled
	Cones:	Nickel Sampler (1.0 mm orifice)
		Nickel Skimmer (0.75 mm orifice)

10) Preventative Maintenance

10.1 All maintenance is documented in the instrument logbook. ALS/Kelso maintains a service contract with the instrument manufacturer that allows for an unlimited number of service calls and full reimbursement of all parts and labor.

10.2 Most routine maintenance and troubleshooting is performed by ALS staff. Preventive maintenance activities listed below should be performed when needed as determined by instrument performance (i.e. stability, sensitivity, etc.) or by visual inspection. Other maintenance or repairs may, or may not require factory service, depending on the nature of the task.

- cone removal and cleaning
- removal and cleaning of ICP glassware and fittings
- checking and cleaning RF contact strips
- checking air filters and cleaning if necessary
- checking the oil mist filters and cleaning if necessary
- checking the rotary pump oil and adding or changing if necessary
- removal and cleaning of extraction lens
- removal and cleaning of ion lens stack
- replace the electron multiplier as necessary

11) Procedure

11.1 Refer to method 6020B and the instrument manuals for detailed instruction on implementation of the following daily procedures preceding an analytical run.

11.2 The following parameters are monitored to assure awareness of changes in the instrumentation that serve as signals that optimum performance is not being achieved, or as indicators of the physical condition of certain consumable components (i.e. EMT and cones).

11.2.1 Multiplier Voltages

11.2.2 Gas Flows - Coolant Ar

11.2.3 The nebulizer and auxiliary flows are adjusted later as part of the optimizing procedure.

11.3 Optimization

11.3.1 Gas Flows

11.3.1.1 Allow a period of not less than 30 minutes for the instrument to warm up.

11.3.1.2 Aspirate a mixed tune solution into the plasma and monitor the instrument output signal at mass 115 on the rate meter. Adjust the nebulizer and auxiliary flows to obtain maximum signal. Adjust the tension screw on the peristaltic pump to obtain minimum noise in the analytical signal. Record flow rates and note any large variances.

Note: Significant differences in flow rates will be observed for different torches and cones.

11.3.2 Tuning

11.3.2.1 Ion Lens Setting - While monitoring the output signal of a mixed tune solution at mass 115 on the rate meter, adjust the ion lenses to obtain maximum sensitivity. Refer to the instrument manual for details on performing the adjustments.

11.3.2.2 Mass Calibration - Aspirate the tune / mass calibration solution described in section 8.2.7.2 and perform the mass calibration using the instrument's Mass Calibration program. (Refer to the instrument manual for details pertaining to the mass calibration procedure.) The acceptance criteria for the mass calibration is <0.1 amu from the true value. If the mass calibration fails criteria re-tune the instrument and perform the mass calibration procedure again.

11.3.2.3 Resolution Check - Using the spectra created during the mass calibration procedure; perform the resolution check to assure the resolution is less than 0.9 AMU at 5% peak height. If the resolution does not pass criteria adjust the instrument's resolution settings, run a new scan of the mass calibration solution and recheck.

11.3.2.4 Stability Check - Using the tune / mass calibration solution, perform a short-term stability check as per EPA Method 6020B. The relative standard deviations of five scans for each element in the tune solution must be < 5%. If the test does not pass criteria determine the cause (i.e. dirty cones, improper tune, etc.) correct the problem and re-run the test.

11.4 Analytical Run

11.4.1 Calibrate the instrument using a calibration blank (Standard 0), composed of reagent water, the appropriate acid matrix, and the working calibration standard (8.2.2). The masses typically monitored and those used for quantification are listed in Table 2. These masses are set as defaults in the instrument's analytical procedures. To begin select the correct method. Nebulize Standard 0 (Blank) into the plasma. Allow 1-2 minutes for system to equilibrate prior to establishing baseline. Follow directions on computer screen to perform standardization. Nebulize the working calibration standard into the plasma. The operator must sign and date the first page of standardization.

11.4.2 After the first CCB and before the ICS standards a LLCCV standard, at or below the LOQ, is analyzed. The LLCCV must recover between ±20%.

11.4.3 Perform the analysis in the order listed below.

Initial Calibration Verification (ICV)
Continuing Calibration Verification (CCV)
Initial Calibration Blank (ICB)
Continuing Calibration Blank (CCB)
LLCCV
ICSA
ICSAB
Analyze 10 Samples
CCV
CCB
Analyze 10 Samples
CCV
CCB

Repeat sequence as required to complete analytical run, analyzing CCVs/CCBs every 10 analyses and at the end of the run.

11.4.4 Water samples with silver concentrations greater than 100 ug/L require confirmation from the original sample container to ensure silver has not been lost through precipitation in the digestate.

12) QA/QC Requirements

12.1 Initial Precision and Recovery Validation

12.1.1 The accuracy and precision of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four LCS aliquots are prepared and analyzed. The average percent recovery of for each analyte must be 85-115% (for water, and within the LCS limits for soils) and the RSD <20%.

12.2 Method Detection Limits

12.2.1 A method detection limit (MDL) study must be undertaken before analysis of samples can begin. Refer to *Performing and Documenting Method Detection Limit Studies and Establishing Limits of Detection and Quantification* (ADM-MDL) for details of performing the MDL study.

12.2.2 Calculate the average concentration found (x) and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. MDL's must be verified annually or whenever there is a significant change in the background or instrument response.

12.3 The LLQC is verified initially by the analysis of at least 7 replicate samples, spiked at the LLOQ. In most cases, the mean recovery should be ±35% of the true value and the RSD should be <20%.

12.4 IDLs should be determined at least once using new equipment, or after major instrument maintenance. The IDL is determined as the mean of the blank results + three times the standard deviation of 10 replicate analyses of the reagent blank solution.

12.5 Method 6020B requires that the linear range for each wavelength be verified on a daily basis. The linear range verification must recover within 10% of the true value and can be analyzed anywhere within a particular run. If a linear range verification is not analyzed for a specific element, the highest calibration range becomes the linear range. All reporting sample measurements must fall within the linear range.

12.6 The Initial Calibration Verification (ICV) standard is analyzed immediately after calibration. The results of the ICV must agree within ±10% of the expected value. If the control limits are exceeded, the problem will be identified and the instrument recalibrated.

12.7 A Continuing Calibration Verification (CCV) is analyzed after calibration then every 10 samples thereafter with a final CCV closing the final samples of the analytical run.

12.7.1 The results of the CCV must agree within ±10% of the expected value.

12.7.2 If the control limits are exceeded, the problem will be identified and corrective action taken. The instrument recalibrated. The previous 10 samples must be reanalyzed.

12.8 A Continuing Calibration Blank (CCB) is analyzed after calibration then every 10 samples thereafter with a final CCB closing the final samples of the analytical run.

12.8.1 The CCB measured values must be less than the MRL / LOQ for each element for standard applications. Other project-specific criteria may apply (for DoD QSM projects CCB can have no analytes > the LOD).

12.9 The ICSA and ICSAB solutions are analyzed after calibration and before any field samples. The solutions are then reanalyzed every 12 hours. Results of the ICSA are used by the analyst to identify the impact of potential interferences on the quality of the data. Based on these results appropriate action should be taken when interferences are suspected in an field sample including, but not limited to, selecting and alternative isotope for quantification, manual correction of the data, elevating the MRL, selection of an alternative method (e.g. optical ICP) or flagging the result as estimated when no other action is possible. Results for the spiked analytes in the ICSAB solution must agree with ± 20% of the expected value.

INTERFERENCE CHECK SAMPLE COMPONENTS AND CONCENTRATIONS

	Solution A Concentrations (mg/L)	**Solution B** Concentrations (mg/L)
Al	20.0	20.0
Ca	60.0	60.0
Fe	50.0	50.0
Mg	20.0	20.0
Na	50.0	50.0
P	20.0	20.0
K	20.0	20.0
S	20.0	20.0
C	40.0	40.0
Cl	424	424
Mo	0.05	0.05
Ti	0.40	0.40
As	0.0	0.025
Cd	0.0	0.025
Cr	0.0	0.050
Co	0.0	0.050
Cu	0.0	0.050
Mn	0.0	0.050
Ni	0.0	0.050
Se	0.0	0.025
Ag	0.0	0.0125
V	0.0	0.050
Zn	0.0	0.025

NOTE: The concentration of interfering elements in the ICSA and ICSAB solutions are spiked at levels 5 times lower than recommended in Table 1 of Method 6020B. Running the full strength solutions as described in 6020B introduces too much material approximately 0.35 % dissolved solids into the ICP-MS system when trying to conduct low level analysis. Since the ICP-MS instrumentation is able to handle a maximum of 0.2% solids, the 6020B ICSA solution is higher in interfering components than any sample that would run through the instrument. However, the ICS solutions will be analyzed at levels that will provide approximately 0.1% dissolved solids.

12.10 Internal standards are used to correct for physical interferences. Masses used as internal standards include; ^{71}Ga, ^{72}Ge, ^{115}In, ^{6}Li, ^{175}Lu, ^{103}Rh, ^{45}Sc, ^{232}Th, and ^{89}Y. These internal standards are used in combination to cover the appropriate mass ranges. Internal standard correction is applied to the analytical isotopes by direct correlation of analyte to IS (Agilent), (NexION). This function is performed in real-time by the instruments operating system. Internal standards must be run within 50 AMU of the masses that are analyzed. Internal standard recoveries must fall between 30% and 125%. If not, then the sample must be reanalyzed after a fivefold or greater dilution has been performed.

12.11 A method blank is digested and analyzed with every batch of 20 (or fewer) samples to demonstrate that there are no method interferences. If the method blank shows any hits above the MRL for standard applications, or >½ the LOQ for DoD projects or > 1/10 the sample result, corrective action must be taken. The MB can only be rerun once. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and re-analysis,

12.12 Laboratory Control Samples are analyzed at a frequency of 5% or one per batch, whichever is greater. Refer to the current ALS-Kelso DQO spreadsheets for the LCS limits. For method 6020B, the LCS recovery limits are 80-120%. If statistical in-house limits are used, they must fall within the 80-120% range. Project, QAPP, or client-specific control limits may supersede the limits listed, but laboratory limits should be consistent with specified limits in order to establish that the specified limits can be achieved. If the control limits are exceeded, the associated batch of samples will be re-digested and reanalyzed.

12.13 A duplicate is digested one per batch, or per 20 samples (i.e. 5%). The duplicate RPD limits is ≤20%. Project, QAPP, or client-specific control limits may supersede the limits listed. If the control limits are exceeded, the samples will be re-digested and reanalyzed, unless sample non-homogeneity is established as the cause. In these instances, the data and the report will be flagged accordingly.

12.14 A Matrix Spike sample is digested one per batch, or per 20 samples (i.e. 5%). Default spike concentrations are listed in the sample digestion SOPs. Spike concentrations may be adjusted to meet project requirements. The matrix spike recovery will be calculated while the job is in progress. Where specified by project requirements, a matrix spike duplicate may be required. Matrix spike recovery criteria are derived from lab data. For Method 6020B, the recovery limits are 75-125%. If statistical in-house limits are used, they must fall within the 75-125% range. In some cases, project-specific QC limits may be required. Unless specified otherwise, for DoD QSM projects the project LCS criteria will be used for evaluation of matrix spikes. If an analyte recovery is outside acceptance limits proceed with the additional quality control tests described in sections 12.13 and 12.14. Based on results of these tests, the physical nature of the sample (e.g. homogeneity), and any specific project requirements, a determination can then be made as to appropriate corrective action (e.g. re-digestion, reporting with a qualifier, alternative methodologies, etc.). If the analyte concentration is >4x the spike level the spike control limit is no longer applicable and no action is required. For specifics on the preparation and composition of matrix spike solutions refer to the appropriate digestion SOP.

Note: For DoD projects a MS/MSD is required with every extraction batch. The %RSD should be < 20%.

12.15 Post Digestion Spike Test: The post digestion spike test is performed whenever matrix spike or replicate criteria are exceeded. An analyte spike is added to a portion of a prepared sample, or its dilution, and should be recovered to within 75% to 125% of the known value. If this spike fails, then the dilution test should be run on this sample. If both the matrix spike and the post digestion spike fail, then matrix effects are confirmed. For DOD QSM 5.0 the post digestion spike shall be recovered to within 80-120% of the known value.

12.16 Dilution Test: The dilution test is performed whenever matrix spike or replicate criteria and post digestion spike criteria are exceeded. For sample concentrations that are sufficiently high (minimally, a factor of 25 times greater than the LOQ), the analysis of a fivefold (1+4) dilution must agree within ± 20% of the original determination. If the dilution test fails then a chemical or physical effect should be suspected. Corrective action can include additional dilution of the sample, the use of alternate methodologies, etc. or the data can be flagged and reported. The exact course of action will be dependent on the nature of the samples and project requirements and should be discussed with the project manager.

12.17 Instrument blanks should be evaluated for potential carryover and rinse times need to bring the analyte signal to within the CCB criteria. Results from instrument blanks run after standards or control samples should be used to establish levels at which carryover in samples may occur. Samples exhibiting similar effects of carryover should be reanalyzed.

13) Data Reduction and Reporting

13.1 Calculations

13.1.1 Calculate sample results using the data system printouts and digestion information. The digestion and dilution information is entered into the data system. The data system then uses the calculations below to generate a sample result.

13.1.2 Aqueous samples are reported in µg/L:

µg/L (Sample) = C* x Digestion Dilution Factor x Post Digestion Dilution Factor.

C*= Concentration of analyte as measured at the instrument in µg/L (in digestate).

13.1.3 Solid samples are reported in mg/Kg:

$$mg/Kg\ (Sample) = C^{*}\ x\ Post\ Digestion\ Dilution\ Factor\ x\ \frac{Digestion\ Vol.(ml)}{Sample\ wt.(g)}\ x\ \frac{1mg}{1000ug}\ x\ \frac{1L}{1000ml}\ x\ \frac{1000g}{1Kg}$$

C*= Concentration of analyte as measured at the instrument in µg/L (in digestate).

NOTE: If results are to be reported on a dry weight basis, determine the dry weight of a separate aliquot of the sample, using the SOP for Total Solids.

13.2 Common isobaric interferences are corrected using equations equivalent to those listed in EPA Methods 6020B and 200.8. Monitoring of multiple isotopes for a single element provides a mechanism for identifying isobaric interferences. Refer to the Interferences section of EPA methods for additional descriptions of possible interferences and the mechanisms required for adequately compensating for their effects.

13.3 Data Review and Reporting

13.3.1 The ICP-MS operator reviews the MS data and signs and dates the Data Review Form. A qualified senior staff spectroscopist performs a secondary review of the data and the Data Review Form is signed and dated. The data is scanned for later compiling.

13.3.2 The data is saved on the local hard drive and is also copied to the appropriate directory on the network. The data directories are located at r:\icp\wip\data. The data is kept on the local directory for 1 month. The network files are periodically backed up on disc or network tape.

13.3.3 For "non-production" work (such as method development or research/development studies) the analyses are performed under the direction of a senior spectroscopist. All associated data is scrutinized by the senior spectroscopist. Original raw data and associated records are archived in the analytical project file.

13.3.4 The final review and approval of all data is performed by qualified spectroscopists.

14) Contingencies for Handling Our-of-Control or Unacceptable Data

14.1 Refer to the SOP for *Non Conformance and Corrective Action* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

15) Method Performance

15.1 This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2 The method detection limit (MDL) is established using the procedure described in the SOP, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification*. Method Reporting Limits are established for this

method based on MDL studies and as specified in the ALS Quality Assurance Manual.

16) Pollution Prevention and Waste Management

16.1 The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Lab Waste Management Plan.

17) Training

17.1 All analysts performing this analysis are required to read and understand this SOP.

17.2 Training is documented following the *Employee Training and New Employee Orientation* (ADM-TRAIN).

18) Method Modifications

18.1 There are no known modifications in this laboratory standard operating procedure from the reference method.

19) References

19.1 USEPA, Test Methods for Evaluation Solid Waste, SW-846, Update V, Method 6020B, Revision 2, July 2014.

19.2 Agilent and Thermo Elemental Instrument Manuals.

19.3 TNI Quality Standards, 2009, 2016.

19.4 DoD Quality Systems Manual for Environmental Laboratories, Current version.

19.5 ANSI/ISO/IEC 17005:2005/2017 American National Standard, General requirements for the competence of testing and calibration laboratories

20) Changes Since Last Revision

Summary of Revision Changes			
Revision Number	Effective Date	Document Editor	Description of Changes
19	12/2/20	T.Caron	Reformatted SOP to current ALS branding. Minor typographical changes to improve readability and consistency, not affecting technical content. Added section 11.4.

21) Attachments, Tables, and Appendices

21.1 Tables 1A, 1B, and 1C – MRLs for analyte matrix combinations.

21.2 Table 2: Target Element Masses.

21.3 Attachment A – Example Standard Sheets.

TABLE 1A
Target Analyte MRLs - Soil

METHOD	PREP METHOD	ANALYTE	MATRIX	MRL mg/kg
6020B	EPA 3050B	Aluminum	Soil	2
6020B	EPA 3050B	Antimony	Soil	0.05
6020B	EPA 3050B	Arsenic	Soil	0.5
6020B	EPA 3050B	Barium	Soil	0.05
6020B	EPA 3050B	Beryllium	Soil	0.02
6020B	EPA 3050B	Bismuth	Soil	0.05
6020B	EPA 3050B	Boron	Soil	0.5
6020B	EPA 3050B	Cadmium	Soil	0.02
6020B	EPA 3050B	Chromium	Soil	0.2
6020B	EPA 3050B	Cobalt	Soil	0.02
6020B	EPA 3050B	Copper	Soil	0.1
6020B	EPA 3050B	Lead	Soil	0.05
6020B	EPA 3050B	Manganese	Soil	0.05
6020B	EPA 3050B	Molybdenum	Soil	0.05
6020B	EPA 3050B	Nickel	Soil	0.2
6020B	EPA 3050B	Selenium	Soil	1
6020B	EPA 3050B	Silver	Soil	0.02
6020B	EPA 3050B	Thallium	Soil	0.02
6020B	EPA 3050B	Tin	Soil	0.1
6020B	EPA 3050B	Uranium	Soil	0.02
6020B	EPA 3050B	Vanadium	Soil	0.2
6020B	EPA 3050B	Zinc	Soil	0.5

TABLE 1B
Target Analyte MRLs – Water

METHOD	PREP METHOD	ANALYTE	MATRIX	MRL µg/L
6020B	MET-DIG (CLP)	Aluminum	Water	4
6020B	MET-DIG (CLP)	Antimony	Water	0.05
6020B	MET-DIG (CLP)	Arsenic	Water	0.5
6020B	MET-DIG (CLP)	Barium	Water	0.05
6020B	MET-DIG (CLP)	Beryllium	Water	0.02
6020B	MET-DIG (CLP)	Bismuth	Water	0.05
6020B	MET-DIG (CLP)	Boron	Water	2
6020B	MET-DIG (CLP)	Cadmium	Water	0.02
6020B	MET-DIG (CLP)	Chromium	Water	0.2
6020B	MET-DIG (CLP)	Cobalt	Water	0.02
6020B	MET-DIG (CLP)	Copper	Water	0.1
6020B	MET-DIG (CLP)	Iron	Water	2
6020B	MET-DIG (CLP)	Lead	Water	0.02
6020B	MET-DIG (CLP)	Manganese	Water	0.2
6020B	MET-DIG (CLP)	Molybdenum	Water	0.1
6020B	MET-DIG (CLP)	Nickel	Water	0.2
6020B	MET-DIG (CLP)	Selenium	Water	1
6020B	MET-DIG (CLP)	Silver	Water	0.02
6020B	MET-DIG (CLP)	Thallium	Water	0.02
6020B	MET-DIG (CLP)	Tin	Water	0.1
6020B	MET-DIG (CLP)	Uranium	Water	0.02
6020B	MET-DIG (CLP)	Vanadium	Water	0.2
6020B	MET-DIG (CLP)	Zinc	Water	2

TABLE 1C
Target Analyte MRLs – Tissue

METHOD	PREP METHOD	ANALYTE	MATRIX	MRL mg/kg
6020B	PSEP TISSUE	Aluminum	Tissue	2
6020B	PSEP TISSUE	Antimony	Tissue	0.05
6020B	PSEP TISSUE	Arsenic	Tissue	0.5
6020B	PSEP TISSUE	Barium	Tissue	0.05
6020B	PSEP TISSUE	Beryllium	Tissue	0.02
6020B	PSEP TISSUE	Bismuth	Tissue	0.05
6020B	PSEP TISSUE	Boron	Tissue	2
6020B	PSEP TISSUE	Cadmium	Tissue	0.02
6020B	PSEP TISSUE	Chromium	Tissue	0.2
6020B	PSEP TISSUE	Cobalt	Tissue	0.02
6020B	PSEP TISSUE	Copper	Tissue	0.1
6020B	PSEP TISSUE	Iron	Tissue	1
6020B	PSEP TISSUE	Lead	Tissue	0.02
6020B	PSEP TISSUE	Manganese	Tissue	0.05
6020B	PSEP TISSUE	Molybdenum	Tissue	0.05
6020B	PSEP TISSUE	Nickel	Tissue	0.2
6020B	PSEP TISSUE	Selenium	Tissue	1
6020B	PSEP TISSUE	Silver	Tissue	0.02
6020B	PSEP TISSUE	Thallium	Tissue	0.02
6020B	PSEP TISSUE	Tin	Tissue	0.05
6020B	PSEP TISSUE	Uranium	Tissue	0.02
6020B	PSEP TISSUE	Vanadium	Tissue	0.2
6020B	PSEP TISSUE	Zinc	Tissue	0.5

TABLE 2
Target Element Masses

ANALYTE	ISOTOPES ANALYZED	ISOTOPE REPORTED
Aluminum	27	27
Antimony	121, 123	123
Arsenic	75	75
Barium	135, 137, 138	137
Beryllium	9	9
Cadmium	111, 112, 114	111
Chromium	52, 53	52
Cobalt	59	59
Copper	63, 65	65
Lead	206, 207, 208	208
Manganese	55	55
Molybdenum	95, 97, 98	98
Nickel	60, 61, 62	60
Selenium*	77, 78, 82	82
Silver	107, 109	107
Thallium	203, 205	205
Uranium	238	238
Vanadium	51	51
Zinc	66, 67, 68	66

*Se 78 Is the default isotope on the NexION and The Agilent instruments.

ATTACHMENT A
Example Standard Sheets

Solution: **ICP-MS, 200.8 Intermediate Stock**
Matrix: **2% HNO_3**

Element	Aliquot of 1000 ppm Std/1000 mL	Concentration (µg/L)
HNO_3	50.0 mL	5%
Al	1.0 mL	1000
Sb	1.0 mL	1000
As	1.0 mL	1000
Ba	1.0 mL	1000
Be	1.0 mL	1000
Cd	1.0 mL	1000
Cr	1.0 mL	1000
Co	1.0 mL	1000
Cu	1.0 mL	1000
Fe	1.0 mL	1000
Pb	1.0 mL	1000
Mn	1.0 mL	1000
Mo	1.0 mL	1000
Ni	1.0 mL	1000
Se	1.0 mL	1000
Tl	1.0 mL	1000
V	1.0 mL	1000
U	1.0 mL	1000
Zn	1.0 mL	1000

Solution: **ICP-MS, 200.8 Silver Intermediate Stock**
Matrix: **5% HNO_3**

Element	Aliquot of 500 ppm Std/1000 mL	Concentration (µg/L)
HNO_3	50.0 mL	5%
Ag	0.5 mL	500

Solution: **ICP-MS 25 ppb Calibration Standard and CCV**
Matrix: **As required**

Source	Aliquot per 100 mL	Concentration (µg/L)
HNO_3 (Ultrex)	As Required	As Required
Intermediate Stock	2.5 mL	25.0
Silver Intermediate Stock	2.5 mL	12.5

Mercury in Solid or Semi-Solid Waste

DOCUMENT ID: MET-7471, REV 21.0

Approved By: _____ Date: 12/1/20
Inorganics Manager, Jeff Coronado

Approved By Kurt Clarkson Date: 12/1/2020
Quality Assurance Manager, Kurt Clarkson

Approved By: _____ Date: 12-1-20
Laboratory Director, Charles (Pat) Byrne

1) Scope & Applicability

1.1 This Standard Operating Procedure (SOP) describes the procedure used to determine the concentrations of Mercury in soils, sediments, freeze dried tissues, bottom deposits, and sludge-type materials using Method EPA 7471A or 7471B. If this dissolution procedure is not sufficient to dissolve a specific matrix type or sample, then this method is not applicable for that matrix. Method 7471 is a cold-vapor atomic absorption procedure.

1.2 The Method Reporting Limit (MRL) is 0.02 mg/kg. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL). Therefore, MRL=EQL. The reported MRL may be adjusted if required for specific project requirements; however, the capability of achieving other reported MRLs must be demonstrated. A Method Detection Limit (MDL) of 0.002 mg/kg has been achieved using this procedure. Refer to the ALS Kelso DQO Table for current data quality objectives.

1.3 In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DOD ELAP. QC requirements defined in the SOP *Department of Defense Projects - Laboratory Practices and Project Management (ADM-DOD/ADM-DOD5)* may supersede the requirements defined in this SOP.

2) Summary of Procedure

2.1 A representative aliquot of sample is prepared as described in this procedure. The mercury is reduced to its elemental state and aerated from solution and measured with an atomic absorption spectrometer. The mercury vapor passes through a cell positioned in the light path of the AA where absorbance is measured as a function of mercury concentration.

3) Definitions

3.1 For general definitions applicable to most analyses refer to the SOP for *Sample Batches*, ADM-BATCH.

4) Responsibilities

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

4.2 It is the responsibility of the department supervisor/manager to document analyst training and method proficiency, as described in the SOP *Employee Training and Orientation* (ADM-TRAIN).

5) Interferences

5.1 Potassium permanganate is added to eliminate possible interference from sulfide. Samples high in chlorides require additional permanganate because, during the oxidation step, chlorides are converted to free chlorine, which absorbs radiation at 253 nm.

6) Safety

6.1 Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in SDSs where available. Refer to the ALS Kelso Chemical Hygiene Plan and the appropriate SDSs prior to beginning this method.

6.2 Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Glass, plastic, and polytetraflouroethylene (PTFE) containers are suitable in most cases.

7.2 Non-aqueous samples are stored at 4 ± 2 °C from receipt until analysis, unless otherwise dictated by project specifications.

7.3 Samples must be analyzed within 28 days of sampling.

7.4 Non-aqueous samples are stored at 4 ± 2 °C from receipt until analysis, unless otherwise dictated by project specifications.

8) Apparatus and Equipment

8.1 CETAC M-6100A Mercury Analyzer. See Attachments, Tables and Appendices for instrument parameters.

8.2 Environmental Express Block Digestion Unit.

8.3 Pipettors, Eppendorf and Finn pipette fixed and adjustable volume.

8.4 Polypropylene graduated cylinders, 50 mL.

8.5 125 mL Digestion Vessel tubes.

8.6 Laboratory balance, top-loader capable of readings .001g (3-place). Mettler, Ohaus, or equivalent Standards.

8.7 Infrared Thermometer.

9) Standards, Reagents, and Consumable Materials

9.1 Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Standards, reagents and consumable material documentation shall indicate traceability to purchased neats or compounds. Refer to the SOP *Reagent/Standards Login and Tracking* (ADM-RTL) for the complete procedure and documentation requirements.

9.2 All stocks, working solutions and sample dilutions should be prepared using deionized water (DI) conforming to ASTM Type I or ASTM Type II reagent water. For more information on reagent water generation, refer to the related SOP, Operation and Maintenance of Laboratory Reagent Water Systems.

9.3 Mercury stock solution (1,000 mg/L). Commercially prepared certified solution stored at room temperature. The expiration date determined by manufacturer.

9.4 Mercury working standard (100µg/L). Prepared from the intermediate stock solution listed above. Store at room temperature and prepare a new standard daily.

9.5 Laboratory Control Sample – ERA Priority Pollutant/CLP Inorganic Soil reference material. Store at room temperature in the original container and use the vendor expiration date.

9.6 Matrix spike solution (1 mg/L) – Prepare by making a 1:1000 dilution of the mercury stock solution. Store at room temperature and prepare a new standard monthly.

Note: See the Procedure section for details on preparation of calibration and ICV standards. See the Quality Assurance section for QC sample preparation.

9.7 Reagent water - ASTM Type II water (laboratory deionized water).

9.8 Acids - Purity of acids must be established by the laboratory as being high enough to eliminate the introduction of contamination above the Method Reporting Limit.

9.8.1 Nitric Acid (HNO_3) 69-70% – JT Baker-Baker Instra-Analyzed® or equivalent.

9.8.2 Sulfuric Acid concentrated (H_2SO_4) – EMD-OmniTrace® or equivalent.

9.8.3 Hydrochloric Acid concentrated (HCL) – VWR – BHD-Aristar® or equivalent.

9.9 Potassium permanganate solution, 5% w/v. To prepare, add 50 g of solid reagent to 1000 mL of DI water and place on magnetic stir plate for approximately 30 minutes until dissolved.

9.10 Sodium chloride/hydroxylamine hydrochloride solution, 12% w/v each. To prepare, add 120 g sodium chloride and 120 g of hydroxylamine hydrochloride to 1000 mL of DI water and place on magnetic stir plate for approximately 15 minutes until dissolved.

9.11 Stannous chloride, 10% w/v in HCl (7% v/v). To prepare, add 100 g stannous chloride crystals and 70 mL of concentrated hydrochloric acid in 1000 mL of D.I. water. Seal lid on mixing bottle and shake until the stannous chloride is dissolved.

9.12 Aqua Regia – Prepare immediately before use by carefully adding 3 parts of concentrated HCL to one part of HNO_3.

10) Preventative Maintenance

10.1 All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. This includes the routine maintenance described in section 9. The entry in the log must include: date of event, the initials of who performed the work, and a reference to analytical control.

10.2 ALS staff performs all routine maintenance and troubleshooting. Preventative maintenance activities listed below should be performed when needed as determined by instrument performance (i.e. stability, sensitivity, etc.) or by visual inspection. Repairs of an extraordinary nature may or may not require factory service, depending on the nature of the task.

10.3 Keep the instrument free of dust, deposits, and chemical spills.

10.4 Replace the peristaltic and autosampler rinse tubing.

10.5 Remove and clean the Gas-Liquid separator.

10.6 Remove, dismantle, and clean the optical cells (sample cell and reference cell) including the sapphire windows.

10.7 Replace the Hg lamp bulb when the Lamp Over-Range is triggered. (The new instrument does not display a value).

10.8 Each block digester is uniquely identified and the temperature is verified with each batch of samples. The temperature is turned to the 95°C setting and the block is allowed to come to temperature. The analyst will verify that the block gives a temperature of 95 ± 2°C using a semi-annually-calibrated traceable handheld infrared thermometer. If not at the right temperature, the thermostat is adjusted until the IR thermometer reads 95 ± 2°C.

11) Procedure

11.1 Sample Preparation

11.1.1 Mix the sample thoroughly to achieve homogeneity. For soil, sediment, solids, weigh approximately 0.5 g of well-homogenized sample and place in the bottom of a 125 mL digestion tube and record the weight to the nearest 0.01g. Add 5.0 mL of reagent water and 5.0 mL of aqua regia, then heat in the Block Digestion Unit for 2 minutes at 95°C.

11.1.2 Cool then add 10 mL of reagent water and 15 mL of potassium permanganate solution. If the purple color does not persist for 15 minutes, add additional potassium permanganate until it does so. Any additional potassium permanganate solution must also be added to the blanks and standards in equal proportion.

Note: Spiking solution is added prior to acidification.

11.1.3 Mix thoroughly and place in the heating block for 30 minutes at 95°C. The temperature of the block is monitored with a thermometer that is calibrated annually.

11.1.4 Cool and add 6 mL of sodium chloride-hydroxylamine hydrochloride to reduce the excess permanganate. Perform this addition under a hood as Cl_2 could be evolved.

11.1.5 Add 27 mL of reagent water and the sample is ready for analysis. (The vapor generator does the step of adding the stannous chloride solution automatically.)

11.2 Calibration

11.2.1 To prepare calibration standards a 10 ppm intermediate stock solution is first prepared by aliquoting 1.0 mL of commercially prepared 1000 ppm stock standard into an acid rinsed 100 mL Class A volumetric flask and diluting to volume with 1% HNO3. This solution must be prepared monthly. Next, a 100 ppb working solution is prepared by aliquoting 1.0 mL of the 10 ppm intermediate stock solution into an acid rinsed 100 mL Class A volumetric flask and diluting to volume with 1% HNO3. This solution must be prepared daily.

Note: All standard aliquots are measured using calibrated fixed or adjustable volume autopipettors or calibrated disposable 5.0 or 10.0 mL pipettes.

11.2.2 Transfer 0, 0.1, 0.25, 0.5, 2.5 and 5.0 mL aliquots of the working solution to a series of labeled 125 mL digestion tubes. Add the appropriate amount of reagent water to bring each bottle to a volume of 5mL. Add 5.0 mL of aqua regia and heat in the heating block for 2 minutes at 95°C. The final concentrations of the prepared standards are 0, 0.2, 0.5, 1.0, 5.0, 10.0 ppb.

11.2.3 The Initial Calibration Verification (ICV) is prepared by first making a 1000 ppb intermediate solution. 0.10 mL of commercially prepared 1000 ppm stock standard, from a different manufacturer and lot than the calibration standard, is aliquoted into an acid rinsed 100 mL Class A volumetric flask and diluting to volume with 1% HNO_3. This solution must be prepared monthly. Prepare the ICV standard by aliquoting 0.25 mL to a labeled 125 mL digestion tube. Add the 4.75 mL of reagent water and 5.0 mL of Aqua Regia.

11.2.4 Cool and then add 10 mL of reagent water and 15 mL of potassium permanganate solution and return the tubes to the Block Digestion Unit for 30 minutes.

11.2.5 Cool and add 6.0 mL of sodium chloride-hydroxylamine hydrochloride solution. Add 27 mL of reagent water and the standards are ready for analysis.

11.2.6 CETAC Calibration and Sample Analysis

11.2.6.1 Turn on the CETAC instrument, including the Hg lamp, and autosampler. After this is done turn open the operating software (Mercury Analyzer 1.6.5).

11.2.6.2 The rinse station for the autosampler turns on automatically, but the peristaltic pump must be started manually. Make sure all sample uptake and drain tubes are placed correctly on the pump and are secured with the appropriate tension. Place the reagent uptake tube in the stannous chloride and start the pump.

11.2.6.3 From the software's main screen select "File", then: "New From". Under "Template Worksheet", click "Browse" and then select "Kelso Hg Template II". Enter the name of the worksheet and click: OK.

11.2.6.4 Go to the "Sequence Editor" tab to generate a sequence, then enter the QC and field samples to be analyzed in the appropriate order.

11.2.6.5 Transfer the solutions to be analyzed to labeled 12mL polyethylene test tubes and place them in the appropriate spaces on the autosampler trays.

11.2.6.6 Transfer the calibration blank and standards (0.2, 0.5, 1.0, 5.0, and 10 ppb) from their digestion tubes to the standard tubes located behind the autosampler trays. The calibration blank is placed in the left most tube and the other standards are placed in ascending order to the right.

11.2.6.7 Click start and the analysis will begin.

11.2.6.8 After the calibration standards have run the software will use linear regression to create a calibration curve based on the concentration and measured absorbance of each standard. The form of regression line is $y = mx + b$. If the correlation coefficient of the curve is greater than 0.995 the analysis will continue, if not the analysis will be terminated and corrective action will be needed by the analyst.

11.3 As the analysis sequence proceeds, next analyze the following QC standards.

- ICV (5.0 ppb standard prepared from a second source)
- ICB
- CRA (0.2 ppb calibration standard)
- CCV (5.0 ppb calibration standard)
- CCB

11.3.1 If either the ICV or CCV are different from their true values by more than 10% the software will terminate the analysis. If either the ICB or CCB is greater than the MRL the software will terminate the analysis. Method 7471A does not contain criteria for the CRA, however, the result must be a positive measured concentration. For 7471B analyses the criteria are ±30% of the true value. Also, specific project requirements may apply.

Note: For projects falling under DoD QSM requirements, the QSM criteria for CRA standards is ±20% and for ICB and CCB standards no analytes detected > LOD. (The ICV limit is as listed above.)

11.4 Sample Analysis

11.4.1 The samples are analyzed with the CETAC analyzer in the same manner as the calibration standards. The analyzer does the step of adding the stannous chloride solution automatically. Check the baseline between samples to verify that the spectrometer reading has stabilized at the normal baseline level.

11.4.2 The analytical sequence should be set up to include all samples, QC samples, blanks, and calibration verification standards at necessary intervals. Refer to the SOP for Sample Batches.

11.5 Sample digestion batches are analyzed with a set of CCV and CCB standards which are run at the beginning and end of the analytical run and at a minimum every 10 samples during the run. The same criteria listed above are applied to the CCVs and CCBs and if one is found to be outside these limits the analysis is terminated.

12) QA/QC Requirements

12.1 Initial Precision and Recovery Validation

12.1.1 This method shall operate under the formal Quality Assurance Program established at ALS and must maintain records that define the quality of data that is generated. Data shall be compared to established criteria in order to determine if the results of the analyses meet the performance characteristics of the method. It is required that an initial demonstration of capability and periodic analysis of laboratory reagent blanks, laboratory fortified blanks, and other QC solutions as a continuing check on performance. The accuracy and precision of the procedure must be validated *before* analyses of samples begin, or whenever significant changes to the procedures have been made.

12.1.2 Initial demonstration of capability must be performed by each analyst performing sample analysis and documented in the laboratory records.

12.2 Method Detection Limits

12.2.1 A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution near the MRL and analyze. Refer to *Performing and Documenting Method Detection Limit Studies and Establishing Limits of Detection and Quantification.*

12.2.2 Calculate the average concentration found (x) in µg/L, and the standard deviation of the concentrations (s) in µg/L for each analyte. Calculate the MDL for each analyte. Refer to ADM-MDL.

Note: Method Detection Limits are subject to change as new MDL studies are completed.

12.3 Limits of Quantification

12.3.1 The laboratory establishes a Limit of Quantitation (LOQ) for each analyte as the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements. Analysis of a standard or extract prepared at the lowest point calibration standard provides confirmation of the established sensitivity of the method. The LOQ recoveries should be within 70-130% of the true values to verify the data reporting limit. Refer to ADM-MDL.

12.4 For method 7471B, an LLQC sample (a CRA that is carried through the digestion) must be analyzed to verify accuracy at the MRL. The recovery must be ±30%.

12.5 Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual, in *Sample Batches* (ADM-BATCH). For this analysis, these include:

12.5.1 Prepare one method blank (MB) per digestion batch, or per 20 samples, or per EPA SDG group, whichever is more frequent. Use D.I. water and follow the digestion procedures. The Method Blank should be < MRL. Re-digest the associated samples if sample levels are <20X MB level.

Note: For projects falling under DoD QSM requirements, the QSM criteria for method blanks is no analytes detected > ½ MRL.

12.5.2 Prepare one Laboratory Control Sample (LCS) per digestion batch, or per 20 samples. Weigh 0.25 g of the current lot of "Environmental Resource Associates Priority Pollutant/CLP Inorganic Soil" prepared reference material in to a 125 mL Digestion vessel tube and prepare as per the procedure.

12.5.3 Calculate the LCS recovery as follows:

$$\%R = X/TV \times 100$$

Where

X = Concentration of the analyte recovered
TV = True value of amount spiked

Apply LCS recovery criteria from the DQO Table, unless project-specific or in-house limits are established. For method 7471B, the LCS recovery limits are 80-120%. If statistical in-house limits are used, they must fall within the 80-120% range.

Note: For DoD QSM projects, the QSM LCS criterion is 80-120%. If the LCS fails the acceptance criteria, re-digest the batch of samples.

12.5.4 Prepare one sample duplicate and one matrix spike sample per each digestion batch, or per twenty samples, whichever is more frequent. For the matrix spike, add 0.25mL of the matrix spike solution to the designated spike sample, resulting in a spike concentration of 0.5 mg/kg. At times, specific samples will be assigned as duplicates or spikes depending on client requirements.

Note: Duplicate samples are routinely analyzed; however some projects may require a MSD. All DOD projects require a MSD. The MSD sample is prepare as described above.

12.5.5 The RPD criterion for duplicates is 20%. If not, flag the data or re-digest samples. Apply matrix spike recovery criterion listed in the DQO Table, unless

project-specific limits are required. For method 7471B, the recovery limits are 80-120%. If statistical in-house limits are used, they must fall within the 80-120% range. For DoD QSM work, MS recoveries are assessed using the QSM LCS control limits. If the MS (and/or MSD where applicable) recovery is outside acceptance limits proceed with the additional interference tests. Based on results of these tests, the physical nature of the sample (e.g. homogeneity), and any specific project requirements, a determination can then be made as to appropriate corrective action (e.g. re-digestion, reporting with a qualifier, alternative methodologies, etc.). If the analyte concentration is >4x the spike level the spike control limit is no longer applicable and no action is required.

Note: For DoD QSM projects, the duplicate RPD limit is 20% and MS recoveries are assessed using the QSM LCS control limits 80-120%.

12.5.5.1 Calculate percent recovery (%R) as:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

12.5.5.2 Calculate Relative Percent Difference (RPD) as:

$$\%\,RPD = \frac{|R1 - R2|}{(R1 + R2)/2} \times 100$$

Where R1= Higher Result
R2= Lower Result

12.5.6 Interference Tests: Prepare one post spike for every batch of samples and if samples are sufficiently high (10x the MRL/LOQ) a serial dilution. The serial dilution must agree within 10% of the original sample result. Post spike recovery acceptance limits for method 7471A and 7471B are 80-120% for project falling under SW-846 Update IV. When both the post spike and dilution tests fail all of the samples in the associated preparation batch must be quantified via Method of Standard Additions (MSA).

13) Data Reduction and Reporting

13.1 It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified in section 12.

13.2 Record all sample weight, volumes and dilutions on an A.A. benchsheet (see Attachments).

13.3 Solution concentrations are calculated by the Mercury Analyzer software based on the linear regression calibration curve created when the calibration standards are analyzed. The absorbance measured for each sample is applied to the linear regression curve and the final solution concentration is determined and displayed as the instrument result.

13.4 Calculate sample results using the data system printouts and digestion information. The digestion and dilution information is entered into the data system. The data

system then uses the calculations below to generate a sample result. Solid samples are reported in mg/Kg:

$$mg/Kg(Sample) = C^{*} \times Post\,Digestion\,Dilution\,Factor \times \frac{Digestion\,Vol.(ml)}{Sample\,wt.(g)} \times \frac{1mg}{1000ug} \times \frac{1L}{1000ml} \times \frac{1000g}{1Kg}$$

C*= Concentration of analyte as measured at the instrument in ug/L (in digestate).

NOTE: If results are to be reported on a dry weight basis as required by certain projects, the Sample Wt. (g) component of the equation should be the dry-weight derived from a determination of %moisture of a separate aliquot of the sample using the SOP for Total Solids.

13.5 Record all concentrations determined at the instrument and calculate the final results in mg/Kg. Record the final results on the A.A. Benchsheet.

13.6 The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in the ALS network directory R:\ICP\WIP. Once the results are transferred, the report is reviewed.

13.7 A daily run log of all samples analyzed is maintained. All data should be printed and stored after operator has checked for evenness of burns. A copy of this document will go with each package of Tier III or higher data run that day.

13.8 Refer to the SOP *Laboratory Data Review Process* (ADM-DREV) for general instructions for data review.

14) Method Performance

14.1 Available method performance data is given in the reference method. In addition, this procedure was validated through single laboratory studies of accuracy and precision as in the determinative procedure. The method detection limit(s) and method reporting limit(s) are established for the determinative procedure. See *Performing and Documenting Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*.

15) Pollution Prevention and Waste Management

15.1 The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Lab Waste Management Plan.

16) Contingencies for Handling Out-of-Control or Unacceptable Data

16.1 Refer to the SOP for *Nonconformance and Corrective Action Procedure* (ADM-NCAR) for Refer to the SOP for Nonconformance and Corrective Action (ADIN-NCAR) for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

17) Training

17.1 All analysts performing this analysis are required to read and understand this SOP.

17.2 Training is documented following the *Employee Training and New Employee Orientation* (ADM-TRAIN).

18) Method Modifications

18.1 There are no known modifications in this laboratory standard operating procedure from the reference method.

19) References

19.1 USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Update IV, Method 7471B, Revision 2, February 2007.

19.2 DoD Quality Systems Manual for Environmental Laboratories, current version.

19.3 TNI Standard, Volume 1, 2009 & 2016.

19.4 ISO/IEC 17025:2017 American National Standard, General requirements for the competence of testing and calibration laboratories.

20) Changes Since the Last Revision

Summary of Revision Changes			
Revision Number	Effective Date	Document Editor	Description of Changes
21.0	12/4/2020	T. Caron	Updated SOP signatories. Sections 8 & 10: Added the use of an infrared thermometer to verify hot block operating temperature.

21) Attachments, Tables, and Appendices

21.1 Table 1: Analysis Parameters - M 6100 Mercury Analyzer.

21.2 Table 2: Summary of Corrective Actions.

Table 1
Analysis Parameters M-6100 Mercury Analyzer

Analysis Parameters

Instrument M-6100 Mercury Analyzer

Conditions

Gas flow (mL/min)	Sample Uptake (s)	Rinse (s)	Read delay (s)	Replicates (#)	Replicate time (s)	Pump speed (%)	Wavelength (nm)
40	30.00	60.00	50.00	4	2.00		253.65

Instrumental Zero

Zero before first sample: No

Zero periodically: Yes

Before each calibration

Baseline Correction

#1 Start time (s)	#1 End time (s)	#2 Start time (s)	#2 End time (s)
5.00	10.00		

Standby Mode

Enabled: Yes

Standby Options: gas off, lamp off

Autodilution

Enabled: No

Condition:

Tube # range:

If no autodilution tubes remaining

Calibration

Settings

Algorithm	Through blank	Weighted fit	Cal. Type	Recalibration rate	Reslope rate	Reslope standard
Linear	Yes	No	Normal	0	0	N/A

Limits

Calibration slope		Reslope		Coeff. of
Lower (%)	Upper (%)	Lower (%)	Upper (%)	Determination
75	125	75	125	0.99500

Error action: Stop analysis

QC

GLP Override: Yes

QC Tests

Table 2

Summary of Corrective Actions				
Method Reference	**Control**	**Specification and Frequency**	**Acceptance Criteria**	**Corrective Action**
EPA 7471A/B	ICAL	Prior to sample analysis	R2 ≥ 0.995	Correct problem then repeat ICAL
EPA 7471A/B	ICV	After ICAL	± 10%	Correct problem and verify second source standard; rerun second source verification. If fails, correct problem and repeat initial calibration.
EPA 7471A/B	CCV	Prior to sample analysis	± 10%	Correct problem then repeat CCV or repeat ICAL
EPA 7471A/B	Method Blank	Include with each analysis batch (up to 20 samples)	<MRL	If target exceeds MRL, reanalyze to determine if instrument was cause. If still noncompliant then: Re-extract or reanalyze samples containing contaminate, unless samples contain > 20x amount in blank.
EPA 7471A/B	Laboratory Control Sample	Include with each analysis batch (up to 20 samples)	See DQO	If exceeds limits, re-extract and re-analyze
EPA 7471A/B	Matrix Spike	Include with each analysis batch (up to 20 samples)	See DQO	Evaluate data to determine if the there is a matrix effect or analytical error
EPA 7471A/B	Sample Duplicates	Include with each analysis batch (up to 20 samples)	≤ 20 % RPD	Re-homogenize and re-analyze if result is > 5 X the MRL

PARTICLE SIZE ANALYSIS FOR SOIL AND SEDIMENT (SIEVE AND HYDROMETER METHOD)

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Document Authorization

Authorized By: Colby Bingham (Quality Authorization) Date: 25-Aug-19

Authorized By: Dwayne Bennett (Technical Authorization) Date: 16-Aug-19

Implementation Authorization

Saskatoon
Laboratory Location

Authorized By: _____ (Laboratory Manager) Effective Date: _____

PROVIDED BY ALS TO BERNICE KIDD, "JACOBS" APRIL 14, 2021

1.0 SCOPE AND APPLICATION

1.1 Scope

This method is the most common test used to determine the sand, silt and clay content of a mineral soil or sediment sample. Soil scientists, environmental engineers or civil engineers may use particle size data for a variety of applications.

There are many instances where other particle size methods are recommended for specific sample types or customer applications.

LIMS Product	Applicable Regulation/Guide	Soil Classification System
GRAIN SIZE-HYD-SK	General Use	ASTM D422-63
GRAIN SIZE-MBWQ-SK	Onsite Wastewater Management Systems Regulation (Manitoba Conservation)	Udden-Wentworth
GRAIN SIZE-HYD-SR-SK	Metal Mining EEM Guidance Document for Aquatic Environmental Effects	Udden-Wentworth
PSA-1-HYD-SK	General Use for Soil Classification	CSSC
Special Request-SK	General Use	Specified by Client

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1.2 Parameters and Limits of Reporting

The most common analyte lists and Limits of Reporting for ALS Canada labs are listed. All Limits of Reporting (LOR) are subject to validation by each lab. Other lists and LORs may be offered where validated.

Parameter (PSA-1-HYD-SK)	Matrix		Limit of Reporting
	Matnum	Name	%
Sand (2 mm – 0.05 mm)	2	Soil	1
Silt (0.05 mm – 0.002 mm)	2	Soil	1
Clay (<0.002 mm)	2	Soil	1
Texture Class	2	Soil	N/A
Parameter (GRAIN SIZE-HYD-SK)	**Matrix**		**Limit of Reporting**
	Matnum	**Name**	**%**
Gravel (4.75 mm-75mm.)	2	Soil	1
Coarse Sand (2mm - 4.75 mm)	2	Soil	1
Medium Sand (0.425mm - 2 mm)	2	Soil	1
Fine Sand (0.075mm - 0.425 mm)	2	Soil	1
Fines (< 0.075 mm)	2	Soil	1
Silt (0.005mm-0.075mm)	2	Soil	1

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Silt (0.002mm-0.075mm)	2	Soil	1
Clay(< 0.005 mm)	2	Soil	1
Clay(< 0.002 mm)	2	Soil	1
Grain Size Curve	2	Soil	NA
Parameter (GRAIN SIZE-MBWQ-SK)	**Matrix**		**Limit of Reporting**
	Matnum	**Name**	**%**
Gravel (4.75 mm-75mm.)	2	Soil	1
Sand (0.075mm - 4.75 mm)	2	Soil	1
Silt (0.005mm-0.075mm)	2	Soil	1
Clay(< 0.005 mm)	2	Soil	1
Grain Size Curve	2	Soil	NA
Parameter (GRAIN SIZE-HYD-SR-SK)	**Matrix**		**Limit of Reporting**
	Matnum	**Name**	**%**
% Passing 75 mm	2	Soil	1
% Passing 50 mm	2	Soil	1
% Passing 37.5 mm	2	Soil	1
% Passing 25 mm	2	Soil	1
% Passing 19 mm	2	Soil	1
% Passing 9.5 mm	2	Soil	1
% Passing 4.75 mm	2	Soil	1
% Passing 2 mm	2	Soil	1
% Passing 0.85 mm	2	Soil	1
% Passing 0.425 mm	2	Soil	1
% Passing 0.106 mm	2	Soil	1
% Passing 0.075 mm	2	Soil	1
% Passing 0.045 mm	2	Soil	1
% Passing 0.033 mm	2	Soil	1
% Passing 0.023 mm	2	Soil	1
% Passing 0.017 mm	2	Soil	1
% Passing 0.012 mm	2	Soil	1
% Passing 0.009 mm	2	Soil	1
% Passing 0.006 mm	2	Soil	1

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% Passing 0.005 mm	2	Soil	1
% Passing 0.0046 mm	2	Soil	1
% Passing 0.003 mm	2	Soil	1
% Passing 0.002 mm	2	Soil	1
% Passing 0.0015 mm	2	Soil	1
% Passing 0.001 mm	2	Soil	1
Grain Size Curve	2	Soil	NA

Parameter (PSA-1 and PSA-3)	Matrix		Limit of Reporting
	Matnum	Name	%
% Sand (2.0mm - 0.05mm)	2	Soil	1
% Silt (0.05mm - 2µm)	2	Soil	1
% Clay (<2µm)	2	Soil	1
Texture	2	Soil	NA

1.3 Method Performance Data and Measurement Uncertainty

This is not a chemistry test. Accuracy is expressed as absolute bias rather than % recovery. Uncertainty is expressed as standard deviation rather than relative standard deviation. Refer to NA-SP-0100 Initial Method Validation – Chemistry and NA-SP-0102 Method Revalidation and MU – Chemistry for policies on determination and storage of method performance data.

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2.0 PRINCIPLE OF METHOD

Particle size distribution is determined by a combination of techniques. Dry sieving is performed for coarse particles, wet sieving for sand particles and the hydrometer method for clay particles.

The air-dried sample must be broken down into individual soil particles before it is ready for analysis. This is accomplished by first passing the sample through a flail grinder, which breaks up the larger soil lumps. Mixing the soil with sodium hexametaphosphate then disperses the smaller clay lumps.

The sedimentation part of the test is based on Stokes' Law, which relates the radius of soil particles to the velocity of their sedimentation. After homogenizing 1 L of suspension, a hydrometer is used to measure soil suspension density at two specific times when the weight per L of particles < 50 µm and (later) of particles < 2 µm is about to decline, at the effective reading depth of the hydrometer.

3.0 DEFINITIONS

Refer to NA-SP-0001 Definitions of Key Terms for definitions of common terms.

PSA-1: Particle size analysis providing size fractionation based on CSSC soil classification. Sand (2mm-0.05mm), Silt (0.05mm-0.002mm), Clay (<0.002mm). The texture classification is reported based on the CSSC soil triangle (Appendix 3)

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PSA-3: Particle size analysis providing size fractionation based on PSA-1. The PSA 3 includes additional pretreatment steps for removal of organic matter and carbonates.

4.0 INTERFERENCES

4.1 Foreign Material

Samples with more than approximately 20% foreign material such as sea shells, wood chips, peat or organic sludge are not suitable for this test. It may or may not be possible to remove this material prior to analysis. Contact the account manager for instructions on how to proceed.

4.2 Crushing Gravel

If a sample contains gravel, dry soil lumps are manually disaggregated to pass a 2 mm sieve using a mortar and rubber-tipped pestle. Refer to Techsys # SK.I.0069 demonstrating that a flail grinder may begin to crush gravel after only a few seconds of operation.

4.3 Crushing Primary Soil Particles

If a sample does not contain gravel, dry soil lumps are mechanically disaggregated to pass a 2 mm sieve using a flail grinder. Refer to SK.I.0063, which demonstrates that flail grinders do not alter the size of primary soil particles.

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4.4 Carbonates

High levels of insoluble carbonates can act as a cementing agent thereby interfering with the dispersion of clay particles. Carbonates can be detected after the soil has been prepared, but prior to laboratory analysis. Before performing the test, refer to section 9.7 for guidance on identifying soils that are high in carbonates and section 9.6.5 for instructions on how to remove carbonates from the soil.

4.5 Organic Soil

Organic matter within the soil can also act as a cementing agent thereby interfering with the dispersion of clay particles. Organic soils must be identified and then pretreated with hydrogen peroxide before performing this test. Highly organic soils are not suitable for this test, due to the limited effectiveness of the peroxide treatment. Before performing the test, refer to section 9.8 for guidance on identifying soils that are high in organic matter and section 9.6.6 for instructions on how to remove organic matter from the soil.

Note:

1. If the sample is found to be high in carbonates and organic matter for PSA-1, then
   PSA-3 test code must be applied to the sample. Contact account manager
   and request that the particle size analysis procedure be changed from PSA-1 to PSA-3 test, which includes removal of carbonates and organic matter.
2. If the sample is found to be high in carbonates and organic matter for Grain size, follow the procedure outlined in section 9.9 and 9.10 to remove carbonates and organic matter.

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4.6 High Salinity

Highly saline soils may cause a significant change in the density of the Calgon solution. This does not allow complete settling of the clay particles as shown in Appendix 1. This problem will not be detected until the test has begun, but can be corrected by discarding the clear liquid after the soil has settled, replacing with fresh liquid, and then performing the test again.

4.7 High Clay Content

Clay lumps that have not been fully dispersed using 5% Calgon must be manually dispersed using a PTFE stirring rod. Refer to step 9.9.2.

5.0 SAMPLE HANDLING AND STORAGE

Parameter(s) and Matrix: All Parameters – Soil	
Container	250 mL – 500mL glass jar. Plastic (ldpe) bag
Preservative	None
Min. Sample Amount	100 grams of dry sample
Sample Pre-treatment	1. Dry at <38 ˚C as per SK-WI-009 Soil Drying and Grinding Protocols 2. Disaggregate the dry soil to as per SK-WI-011 Elwood Grinder Operation. Both >2mm and <2mm portion must be retained.
Lab Storage Conditions	>0-6ºC wet (CCME Vol 4) Ambient temperature dried (CSSS. 1993 section 3.4)
Maximum Sample Holding Time	30 days as received (without lab drying), indefinite when dried. Reference: CCME Vol 4, Table 3A

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Note: If less than 100g is used for PSA analysis due to insufficient sample amount, add the qualifier '**PSAL**' (Limited sample was available for PSA (100g minimum is standard)).

5.1 Sampling Instructions

Sample collection is not conducted by the laboratory.

5.2 Sampling Handling and Transportation

Refer to ALS Sample Handling and Preservation Guidelines for further details on the sample handling procedures recommended by ALS.

If holding time violations occur, Account Manager(s) must be notified.

6.0 EQUIPMENT AND SUPPLIES

The following equipment and supplies have been shown to meet the performance specifications of this test method. Alternative equivalent supplies may be substituted for those listed below if suitability is demonstrated.

6.1 Sample Preparation Equipment

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1. Forced-air soil drying oven. Custom made. Set to 35±3˚C.
2. Stainless steel flail grinder with 2 mm perforations. Custom made and henceforth known as "Elwood". The design of this grinder is similar to a commercially available grinder (Humboldt Manufacturing Ltd. Model H-4199).
3. Mortar, Porcelain 180mm. Fisher catalog # 12-961-D MEOL #4420028
4. Pestle, Porcelain 180mm for mortar. Fisher catalog # 12-961-5B MEOL #4420027
5. Rubber-tipped pestle. M & T Testing Equipment catalog # H-4258 MEOL # 4420234

6.2 Large Sieve Set

1. 75 mm stainless steel sieve, 8" diameter (VWR catalog # 57334-204 MEOL #4560269)
2. 37.5 mm stainless steel sieve, 8"diameter (VWR catalog # 57334-214 MEOL # 4560284)
3. 25 mm stainless steel sieve, 8" diameter (VWR catalog # 57334-220 MEOL # 4560270)
4. 19 mm stainless steel sieve, 8" diameter (VWR catalog # 57334-224 MEOL # 4560271)
5. 9.5 mm stainless steel sieve, 8" diameter (VWR catalog # 57334-234 MEOL # 4560272)
6. 4.75 mm stainless steel sieve, 8"diameter (VWR catalog # 57334-246 MEOL # 4560273)
7. 2 mm stainless steel sieve, 8" diameter (VWR catalog # 57324-442 MEOL #4560022)
8. Stainless steel bottom pan, 8" (VWR catalog # 57334-652 MEOL #4560007)
9. Cover, sieve ring stainless steel fits 8"(VWR catalog # 57334-662 MEOL #4560228).

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6.3 Laboratory Equipment

1. Top loading balance with ± 0.01g sensitivity. Sartorius Practum Model 2102-1S. Fisher catalog # 14-558-111. MEOL # 3091713 or equivalent.
2. Analytical balance with ± 0.1mg sensitivity. Sartorious Practum Model 224-1S. Fisher catalog # 14-557-422. MEOL # 3091661 or equivalent.
3. Orbital shaker. VWR Advanced Digital Shaker model 10000-2. VWR catalog # 80081-136. MEOL # 3091332.
4. Multi-channel timer. VWR catalog # 62344-600. MEOL # 4550697.
5. Drying oven set to 110 ± 5˚C. Yamato model DKN812C. Fisher catalog # 361023113.
6. Additional shelves for oven. Fisher catalog # 1326377. MEOL # 2190745.
7. Hot plate. VWR catalog # 11301-058 or equivalent.
8. Dispenser, VWR labmax bottle top 20-100ml .VWR catalog # 40000-070. MEOL # 4120187 or equivalent.

6.4 Small Sieve Set

1. 0.850 mm stainless steel sieve, 6" diameter (Fisher catalog # 3867 Special MEOL # 4560258)
2. 0.425 mm stainless steel sieve, 6" diameter (Fisher catalog # 3871 Special MEOL # 4560259)
3. 0.25 mm stainless steel sieve, 6" diameter (Fisher catalog # 3874 Special MEOL # 4560260)
4. 0.149 mm stainless steel sieve, 6" diameter (Fisher catalog # 3877 Special MEOL # 4560261)

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5. 0.075 mm stainless steel sieve, 6" diameter (Fisher catalog # 3881 Special MEOL #4560056)
6. Stainless steel bottom pan, 6" (Fisher catalog # 8467 Special MEOL # 4560263)

6.5 Software

ALS Balance Talk software

6.6 Sample Preparation Supplies

Teflon coated baking trays (13 X 9"). Purchased locally.

6.7 Laboratory Analysis Supplies

1. Medium Anti-static Weight boats, white. VWR catalog #89106-766.MEOL # 4220021
2. P.E.T clear Bottles, 250ml Wide Mouth w/Unlined 53mm cap. (Systems Plus catalog # P250/US MEOL # 4010481).

   Note:

   a. These bottles are to be calibrated by adding 50±1ml water and marking the water level with permanent marker.
   b. PSA-3 method bottles are to be calibrated by adding 50±5g of sample and 100 ±1ml of 5% Calgon and marking the liquid level with permanent marker.

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3. Glass sedimentation cylinders marked at 1000 ml (VWR catalog # 34795-000 MEOL# 4000623 or equivalent.
4. ASTM No. 1. 152 H soil hydrometer (Fisher catalogue # 13202133 MEOL # 3300320).

   Note:

   c. It is important to purchase a high quality soil hydrometer such as the hydrometers produced DURACR by H-B Instrument Company. Cheaper hydrometers often do not meet the accuracy requirements of the method.
   d. Before using a hydrometer for the first time, verify the accuracy of the hydrometer: 1000ml of DI water in 1000mL cylinder at 20±1˚C should read "0".
   e. Record the reading in form SK-FM-1023a Hydrometer Verification according to the serial number.
   f. Before first use, and additionally every six months, perform a Hydrometer Standard Dimensions check, using a 30 cm ruler and calipers.
   g. Record the data in for SK-FM-1023a Hydrometer Verification

5. Aluminum dishes (70 mL capacity). VWR catalog # 25433-089. MEOL # 4220025.
6. Aluminum dishes (57 mL capacity). VWR catalog # 16005-126. MEOL # 4551455.
7. Cut-resistant gloves. Fisher catalog # 29-635-2165 or equivalent.
8. Metal spatula. Custom made. Refer to ASTM method D 7928

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9. 750ml wash Bottle. VWR catalog # 16651-609. MEOL #4018902.
10. 1000ml flasks. Fisher catalog # 10-090D MEOL #4000639 or equivalent
11. 250 mL graduated cylinder. VWR catalog # 89090-452. MEOL # 4000824.
12. 23ml transfer pipet. VWR catalog # 14670-405. MEOL #4430539.
13. Magnifying glass (optional)
14. Thermometer with 0.1 ºC sensitivity. VWR 89094-746, MEOL 3290048, or equivalent
15. Watch Glass 100mm VWR 66110-087, MEOL 4001506, or equivalent

6.8 Reagent Preparation Supplies

1. 20 liter appropriate container.
2. 20L amber HDPE carboy with versatile cap and spigot (VWR catalog # 89217-234, MEOL# 4019185), or equivalent.
3. 100 mL graduated cylinder. Fisher catalog # 08-553B. MEOL # 4000502.
4. Class A Volumetric Flask, 1L. VWR catalog # 89001-920. MEOL # 4000159.

7.0 REAGENTS AND STANDARDS

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The following reagents and standards have been shown to meet the performance specifications of this test method. Alternate sources may be substituted where equivalent performance and suitability has been verified. Refer to site-specific reagent and standard preparation logs and analytical records to identify the reagents and standards in current use at each lab.

7.1 Reagents

All solvents must be ACS Grade or better.

1. DI water. Fill 20L amber HDPE carboy with DI water in room temperature for overnight.
2. Isopropyl or Ethyl alcohol denatured anhydrous 4L. VWR catalog # CA71007-230. MEOL # 6010154. (or other anti-foaming agent)
3. Sodium Hexametaphosphate (Calgon). Min. 65% P2O5. VWR catalog # SX0583-3. MEOL # 6030481.
4. Concentrated hydrochloric acid, ACS grade. Fisher Cat# 351282-4. MEOL # 6000201. Store in an acid cabinet.
5. 3M Hydrochloric acid (HCl)
   a. Fill a one liter Class A volumetric flask half full with DI water.
   b. Add 250±1ml of concentrated HCl using a 250 ml graduated cylinder. Make up to volume with DI water, cap and then invert several times to mix.
   c. Transfer to a labeled plastic bottle and store in the acid cabinet.
   d. This solution is stable for 6 months. Record the preparation data on form # SK-FM-1009a - HCl Prep for PSA.

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6. Hydrogen Peroxide, ACS grade 30-35%, Fisher catalog # H325-4 MEOL #6030197 or equivalent. Store in the acid cabinet. Keep away from combustible material such as wood or paper. The expiry is based on the manufacturer expiry date.
7. 5% Calgon Dispersing Agent (record data in form # SK-FM-1023b-PSA 5% Calgon Reagent Prep)
   a. Transfer 200.0 ± 0.1 grams of to a 4 liter vessel containing about 3 liters of DI water.
   b. Stir vigorously until dissolved. Dilute to approximately 4 liters ±5ml with DI water and stir.
   c. Transfer to a 20 liter appropriate container. Affix a label to the carboy. Write the ID number of the solution as indicated in form # SK-FM-1023b-PSA 5% Calgon Reagent Prep.
   d. Store at room temperature. Solution expires after 7 days.
8. Determination of Calgon Blank Value
   a. Prepare a blank measuring cylinder by making 100 ± 0.5 ml of stock Calgon up to the 1,000 ml mark with stock de-ionized water. Carefully immerse the hydrometer and read it at the upper level of the meniscus. Record this initial blank reading to the nearest 0.25 g/L . If necessary use a magnifying glass. Record on the "Hydrometer (Calgon Blank)" tab in the GRAIN SIZE-HYD-SK spreadsheet.

8.0 QUALITY CONTROL

8.1 Method QC

Maximum (Sample Preparation) Batch Size =	20 Client Samples

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Method QC	Min. Frequency	Data Quality Objective (units)
Reagent Blank (RB)	1 per batch	5 ± 1 g/L
Reference Material (RM)	1 per batch	± 5% from long term mean If RM pretreated for OM and carbonate, the DQO will be ± 10%
Sample Duplicate	1 per batch	Difference < 5% sand, silt or clay. If sample has been pretreated for OM and carbonate, the DQO will be<10%

9.0 TEST PROCEDURE

9.1 Sampling Suitability

1. Foreign Matter
   a. Samples with more than approximately 15-20% foreign material such as sea shells, wood chips, peat or organic sludge are not suitable for this test. It may or may not be possible to remove this material prior to analysis. Contact the account manager for further instructions.
2. Minimum Sample Size
   a. Refer to the D6913-04(2009)-TABLE 2 Minimum Mass Requirement for Specimen, as show on 16.2 Appendix 2. For the minimum Dry Mass of Specimen required is 50g of soil sample

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and 75g of sandy soil sample. If the minimum amount of sample is not available, contact the account manager for instructions on how to proceed and add qualifier "PSAL" on the sample in LIMS.

9.2 Drying the Sample

Samples are dried and ground by the samples preparation department, according to SK-WI-009 Soil Drying and Grinding Protocols.

The sample is spread onto a baking tray, and then placed into the forced air drying oven overnight at 35±3˚C. The sample may be dried at up to 60 ˚C, if other test codes being performed allows for the higher temperature.

Note: Do not take subsamples from watery samples. The entire watery sample must be dried.

9.3 Verifying the Balance (daily when used)

1. Tare the balance.

2. Place designated weights on the balance and record the weights on the appropriate form SK-FM-0075a Daily Balance Verification. If the weights are outside of the acceptance range, seek assistance from your supervisor.

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9.4 Dispenser and Oven (daily when used), Thermometer, and Orbital shaker (Annually) Verification

9.4.1 Dispenser Check

Use the dispenser to transfer 100 mL of 5% Calgon solution into a 100ml graduated cylinder. Record the volume of Calgon on form SK-FM-0075af PSA Volumetric Device Verification Form. The acceptance criterion is 100mL ± 1.0mL.

9.4.2 Oven Check

1. Turn on the oven if not already on, and wait 1 hour for it to warm up.
2. Record the initial oven temperature on the form SK-FM-0075ad Daily Oven Checks.
3. If the oven temperature is outside the 105-115˚C range, then adjust the set temperature.
4. Wait 30 minutes, then record the final temperature to the "Set Temp" on the form.

9.4.3 Annual Thermometer Calibration

The thermometers are annually calibrated to be within ± 0.5 ºC of the reference thermometer. If it is out of range, that thermometer can be recalibrated or serviced. Form "ALS Thermometer Calibration Record "can be used.

9.4.4 Orbital Shaker Check

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1. For the 70 rpm orbital shaker the count needs to be 70 ± 4 rpm.

2. Set a timer for 30 seconds and place your hand at one corner of the shaker. Start the timer and count how many times the shaker hits your hand in the 30 second time frame. Multiply this number by 2 to get your result. Record results on form SK-FM-1009d Orbital Shaker Verification Log.

3. Inform your supervisor if the shaker is out of range so that the shaker can be Calibrated or serviced

CALIBRATION PROCEDURE:

1. The tray should be free of any samples, vessels, and accessories prior to calibrating.

2. Turn unit on. Speed and time displays will be illuminated.

3. Press and hold the standby button and momentarily press the speed on/off button. The speed display should read “CAL”.

4. The unit will run for approximately one (1) minute and automatically calibrate. Refer to the “standard Orbital shaker Instruction Manual”

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9.5 Select the Appropriate Data Transfer Spreadsheet

From the G:\Samson Lab Sheets folder, select one of the following GRAIN SIZE-HYD-SK or PSA-1-HYD-SK Method spreadsheets for data entry and report generation.

9.6 Sample Preparation Procedure

9.6.1 Preparation for Grain Size:

9.6.1.1 Dry Sieving Gravel

1. First, use a rubber pestle and mortar to disaggregate the sample. If the particles are heavy clay aggregates, a porcelain pestle may be used to break down the aggregates.
2. Pass the > 2 mm gravel through the “Large (8”) sieve set”. Shake each sieve for 5-10 seconds. Weigh the gravel or stones on each sieve using 2 decimal place top-loading balance. Enter the weights in the “Sieving (>2mm)” tab and manually enter the total amount in the “Prep (>2mm)” tab in the GRAIN SIZE-HYD-SK spreadsheet.
3. If any soil <2mm is present in bottom pan, add it to the <2mm soil bag.
4. Weigh the < 2 mm soil using the same balance and enter the total weight to the “Prep (<2mm)” tab in the GRAIN SIZE-HYD-SK spreadsheet.

9.6.1.2 Moisture Test

1. Weigh an empty aluminum dish (57ml).

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2. Take a 10±5 gram portion of < 2 mm soil and place into the aluminum dish. Transfer the weight nearest to 0.1mg into the "moisture" tab in the GRAIN SIZE-HYD-SK spreadsheet.
3. Place the dish into a drying oven overnight at 110 ± 5 °C.
4. Cool in a desiccator. Weigh the dry sample and transfer the weight nearest to 0.1mg into the "moisture" tab in the GRAIN SIZE-HYD-SK spreadsheet.

9.6.1.3 Preparing Aluminum Dishes

Set aside one sets of numbered aluminum dishes (70ml) for sand sieving. This must be done prior to analysis.

9.6.2 Preparation for PSA-1/PSA-3

Obtain < 2mm portion of sample from Sample Preparation department.

9.6.3 Checking for High Levels of Carbonates

Carbonates can be detected visually by checking for a whitish color. Not all whitish samples contain carbonates, but should be checked by adding a few drops of 3N hydrochloric acid to approximately ½ gram sample. Excessive bubbling indicates high levels of carbonates. Follow the procedure outlined in section 9.**6.5** to remove carbonates.

9.6.4 Checking for High Levels of Organic Matter

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An experienced analyst can usually detect highly organic samples visually (ie. dark colored soil or fluffy brown material present). In cases where less experienced analysts are involved, the 50 ml calibration line on the sample bottle shall be used as an indicator of unacceptably high organic matter content as seen in **Appendix 4.** This assumes that sample with a bulk density of < 1g/cc has an organic content of > 20%. Refer to section 9.6.6 to remove organic matter.

9.6.5 Removal of Carbonates

1. Weigh 50±5g of < 2mm soil into a 1000 mL flask.
2. In a fume hood, add 60±10 mL 3M HCl, swirl and let stand until all carbonates are removed (fizzing stops). Samples with a high level of carbonates will require more HCl.
3. To remove acid from samples:

Option 1:

1. Wash soil into 50 mL centrifuge tubes with DI water. Centrifuge for 7 min @ 2100±5 rpm and then pour off supernatant. Repeat the washing and centrifuge step 2 more times. The soil is now acid free.
2. If the samples are high in organic matter, transfer the sample back to the 1000 mL flask using minimal DI water. Continue to step 9.10 below if the sample is highly organic.
3. If the samples are low in organic matter, transfer the sample into 250ml P.E.T clear (PSA-3 method) bottles using 100 ± 1mL of 5% Calgon solution, and then allow to settle, then pour off supernatant.

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4. Add 5% Calgon to PSA-3 bottle mark (50 g of sample+ 100 ± 1.0 mL Calgon mark). Screw the cap on tightly and mix.
5. Proceed to step 9.8.3

Option 2:

1. Add DI water to 300 ± 10mL mark, mix with a stir rod, and then allow to settle, then pour off supernatant. Repeat the washing 2 more times. The soil is now acid free. If the samples are high in organic matter. Continue to step 9.10 below.
2. If the samples are low in organic matter, transfer the sample into 250ml P.E.T clear (PSA-3 method) bottles using 100 ± 1.0 mL of 5% Calgon solution, and then allow to settle, then pour off supernatant.
3. Add 5% Calgon to PSA-3 bottle mark (50 g of sample + 100 ± 1.0 mL Calgon mark). Screw the cap on tightly and mix. Proceed to step 9.8.3.

9.6.6 Removal of Organic Matter

1. Slowly add 20 ±10.0 mL of 30-35% H_2O_2, depending on the estimated amount of organic matter in the sample. Samples high in organic matter (i.e. peat moss) will need more H_2O_2.
2. Stir the sample with a stirring rod, then allow a few minutes for the soil to settle.
3. If there still appears to be a lot of organic matter left in the soil layer, place the sample into a hot plate at 75±5.0 ˚C. Keep adding H_2O_2 until fizzing stops.
4. Remove from heat and allow the sample to cool down.
5. If there is a layer of H_2O_2 on top, remove with a 23ml transfer pipet and discard down the drain with running water. Add DI water to 300± 10mL mark, mix with a stir rod, and then allow to settle. Repeat removal of H_2O_2 layer and mixing steps twice more.
6. Transfer the sample back to a 250ml P.E.T clear (PSA-3 method) bottle. Add 100mL of 5% Calgon solution, and then allow to settle, then pour off supernatant.
7. Add 5% Calgon to PSA-3 bottle mark (50 g of sample + 100 ± 1.0mL Calgon mark). Screw the cap on tightly and mix. Proceed to step 9.8.3 of this method.

9.6.7 Removal of Salt

1. After step 9.8.6, when the sample is transferred to the 1000 mL graduated cylinder and DI Water is added, refer to appendix 1 to identify salinity in the sample.
2. If salt is present, Remove the clear liquid from the container using a pipette after the soil has settled, replace with fresh liquid, and then perform the determination again.

9.7 Method QC Preparation

The same method QC procedure is used for grain size, PSA-1, and PSA-3.

9.7.1 Method Blank

Add 100 ± 1.0 ml of stock Calgon to the cylinder and make the volume up to 1,000 ml mark with stock de-ionized water. Carefully immerse the hydrometer and read it at the upper level of

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the meniscus. Record this initial blank reading to the nearest 0.1 g/L (estimated). If necessary use a magnifying glass. Record on the "Hydrometer (Calgon Blank)" tab in the GRAIN SIZE-HYD-SK spreadsheet or PSA-1-HYD-SK.

9.7.2 IRM

Prepare an IRM by weighing 50±5g of IRM standard into a 250ml P.E.T clear bottle. The IRM must go through all steps of the method including optional steps such as carbonate removal.

9.7.3 Duplicate

Randomly select one sample from the analytical batch as a duplicate. This sample should go through all steps that the original sample required including any required pretreatments.

9.8 Sample Dispersion Using Calgon

This procedure applies to grain size, PSA-1, and PSA-3.

1. Transfer 50±5g of < 2mm soil into a 250ml P.E.T clear bottle. Electronically transfer the sample weight to the "Hydrometer (Sample weight)" tab in the GRAIN SIZE-HYD-SK or PSA-1-SK spreadsheet. If the sample appears to be > 90% sand, then use 80 grams sample.
2. Add 100 ± 1.0 ml of Calgon solution, screw the cap on tightly and mix.
3. Place the samples in the rack.
4. Place the samples horizontally on the orbital shaker overnight at 70 ± 4 rpm (minimum 16 hours).
5. Remove the samples from the shaker. Break down any lumps using a PTFE stirring rod.
6. Transfer the entire slurry to a clean measuring cylinder and fill to the 1,000-mL mark with room temperature de-ionized water.

9.9 Grain Size Procedure

This section outlines the procedure for both, grain size hydrometer readings and the subsequent grain size sieving.

9.9.1 Taking Grain Size Hydrometer Readings

1. Insert the plunger and stir the suspension by moving the plunger up and down the whole length of the cylinder. Be sure to loosen all soil from the bottom of the cylinder. Do not remove plunger from sample on upward strokes. Use a consistent number of strokes (8-12) or consistent time (15 sec).
2. Remove the plunger and, immediately, start the stopwatch.
3. Quickly but carefully place the hydrometer in the cylinder. Use your hand to steady any bobbing of the meter. If necessary, add a few drops of amyl or isopropyl alcohol to disperse foam.

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4. When the timer indicates 1 min, read the hydrometer. The reading is transient and should be recorded to the nearest 0.5 g/L. This reading is recorded on “Hydrometer” tab in the GRAIN SIZE-HYD-SK spreadsheet.
5. Continue taking readings at the 2, 4, 8, 15, 30, 60 minute mark without removing the hydrometer. After taking the 60-minute hydrometer reading remove the hydrometer and rinse with water.
6. 20-30 seconds prior to the 120, 240, 480, and 1440 min. mark, carefully put the hydrometer back into the cylinder. Take care not to disturb the settled material.
7. Read the hydrometer for the appropriate time (120, 240, 480, and 1440 min. mark) and record. Carefully remove the hydrometer and rinse with DI water between readings.
8. Cover the cylinder using a glass watch glass between the 480 minute and 1440 minute readings to prevent evaporative losses.
9. Note: If the suspension flocculates as shown in **Appendix 1**, the sample may be saline. Check by measuring the electrical conductivity of a 1:2 (soil to water) suspension. An electrical conductivity reading > 5 ds/m indicates a saline soil. Contact the account manager if the soil is saline. Special pre-treatment may be required to continue the analysis (Removal of Salt. Step 9.11).
10. Take temperature readings at 1, 60, 480 and 1440 min, recorded on “Hydrometer (Temperature Reading)” tab in the GRAIN SIZE-HYD-SK spreadsheet.

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9.9.2 Grain size Sieving

1. To determine sand content, pour the slurry from the 1000ml cylinder through a 75 µm sieve. Rinse the sand on the sieve with tap water until the water that passes through the sieve is clean. If you notice lumps of clay on the sieve, then do not proceed with the test. The test must be repeated from the beginning, making sure that clay lumps are broken down using the PTFE stirring rod.
2. Transfer the clean sand to an aluminum dish (75mL) using a DI water bottle. Place the dish into the 110±5˚C drying oven for at least 2 hours. Remove the dishes from the drying oven and cool for 10 minutes.
3. Pass the dry sand trough “Small Sieve set”. Shake each sieve for 10±1 seconds with tapping. Electronically transfer the weight on each sieve to the “Sieving (2mm-75µm)” tab in the GRAIN SIZE-HYD-SK data transfer spreadsheet.
4. Using a Magnifying glass (optional) to identify the sample “Shape and “Hardness", enter into the “Prep” tab in the GRAIN SIZE-HYD-SK. Do not discard the dish with all of the sand until data review is complete.

9.10 PSA Procedure (PSA-1, PSA-3)

9.10.1 Taking PSA Hydrometer Readings

1. If necessary, add a few drops of amyl or isopropyl alcohol to disperse foam.

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2. Within 30 seconds, transfer the hydrometer from the blank Calgon solution into the soil suspension. (There is no need to dry it since the volume transferred is negligible.) Use your hand to steady any bobbing motion.
3. When the stopwatch or timer indicates 39±1 seconds, read the hydrometer at the upper level of the meniscus. The reading is transient and should be recorded to the nearest 0.5 g/L.
4. Transfer the hydrometer to its wash-water. Let the suspension stand undisturbed for 6 hours.
5. Prepare a blank measuring cylinder by making 100 ± 0.5 ml of stock Calgon up to the 1,000 ml mark with stock de-ionized water. Carefully immerse the hydrometer and read it at the upper level of the meniscus. Record this initial blank reading to the nearest 0.1 g/L (estimated). If necessary use a magnifying glass.
6. After 6 hours - DO NOT REMIX THE SUSPENSION - carefully immerse the hydrometer in the settled suspension as in step 9.10.1.2. The 6-h (upper meniscus) reading is stable and should be recorded to the nearest 0.1 g/L. At about the same time, also record the final blank Calgon solution reading, to the nearest 0.1 g/L. Again use a magnifying glass if necessary to make these readings.
7. Alternatively, the 6-h reading can be estimated from a 2-h reading for samples such as agricultural soils, for which fast turn-around time is essential and slight loss of accuracy can be tolerated.
8. To predict the raw (uncorrected) 6-h reading from the raw 2-h reading, use the following equation (obtained for a range of soils analyzed by three different technicians; Ashworth, private communication):

   Raw 6-h reading = 0.87 * (raw 2-h reading)

   When using a 2-h reading, a blank Calgon solution reading must also be made after 2 hours, and subtracted from the calculated raw 6-h reading, in order to obtain R6h for insertion in equation B below.

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10.0 DATA CALCULATION AND REPORTING

10.1 Grainsize Data Calculations

10.1.1 PERCENT OF SAMPLE PASSING EACH COARSE SIEVE (SIEVE SET #1)

1. Percent passing 75 mm sieve: (Mass of sample< 2mm + Masses retained on 50, 25, 19, 9.5, 4.75 and 2 mm sieves) / (total mass of sample) x 100
2. Percent passing 50 mm sieve:(Mass of sample< 2mm + Masses retained on 25, 19, 9.5, 4.75 and 2 mm sieves) / (total mass of sample) x 100
3. Percent passing 25 mm sieve: (Mass of sample< 2mm + Masses retained on 19, 9.5, 4.75 and 2 mm sieves) / (total mass of sample) x 100
4. Percent passing 19 mm sieve: (Mass of sample< 2mm + Masses retained on 9.5, 4.75 and 2 mm sieves) / (total mass of sample) x 100

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5. Percent passing 9.5 mm sieve: (Mass of sample< 2mm + Masses retained on 4.75 and 2 mm sieves) / (total mass of sample) x 100
6. Percent passing 4.75 mm sieve: (Mass of sample< 2mm + Masses retained on 2 mm sieve) / (total mass of sample) x 100

10.1.2 PERCENT OF SOIL IN SUSPENSION FOR EACH HYDROMETER READING

1. Percent passing 2 mm sieve: Mass of sample < 2mm / (mass of sample < 2mm + mass of sample > 2mm) x 100
2. Oven dry mass of sample used for hydrometer test: Mass of air dried sample used in hydrometer test x (100 - %moisture of sample) / 100
3. Corrected sample mass for hydrometer test: Oven dry mass / % passing 2 mm sieve x 100
4. Percent of sample still in suspension at any given hydrometer reading: (Hydrometer reading – Calgon blank) / Corrected sample mass x 100

10.1.3 DIAMETER OF SOIL PARTICLES FOR EACH HYDROMETER READING

Diameter (mm) = K X SQRT(L/T), where

K = temp constant = (-0.000156 x Temp °C) + 0.0168 [ASTM D422-63, Table 3, 2.65 particle density col.)

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L = Effective depth of hydrometer reading = (-0.164 x hydrometer reading) + 16.298 [ASTM D422-63, Table 2, Hydrometer 152H columns)

T = Temperature (°C)

10.2 PSA Data Calculations

All test data is entered into an Excel spreadsheet specifically designed for calculation of % sand, silt and clay and assigning a soil texture based on raw data provided. Calculations performed in the spreadsheet are described below. Where R40s = hydrometer reading at 40 seconds and R6h = hydrometer reading at 6 hours.

1. Adjusted hydrometer reading R40s = R40s (sample) - R40s (Calgon blank)
2. Adjusted hydrometer reading R6h = R6h (sample) - R6h (Calgon blank)
3. % silt & clay = R40s (Adjusted) * 100 / sample wt. (g)
4. % clay = R6h (Adjusted) * 100 / sample wt. (g)
5. % silt = % silt & clay – % clay
6. % sand = 100 – % silt & clay
7. Soil texture is obtained from % clay and % sand. The soil texture triangle, figure 1, shows different textural classes. Where the calculated % clay and % sand cross, read the soil texture rating.

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10.3 Data Transfer to LIMS

All weights are transferred electronically to the spreadsheets from section 9.12.1 using BalanceTalk software. The spreadsheets will send a copy of the final results to LIMS and also save a copy of the spreadsheet in G:\Enviro\Physical\Physical Data folder.

10.4 Data Validation

Peer review and sign off is a requirement, refer to NA-SP-0175 “Data Validation” for data.

10.5 Data Reporting

1. Enter all data from lab sheet and from balance to the GRAIN SIZE-HYD-SK or PSA-1-SK spreadsheet (G:\Enviro\Physical\ELECTRONIC WIP) to verify results. After QC has been visually verified then lab sheet is approved.
2. LIMS will calculate and report soil texture along with the test results. See Appendix 3.

10.6 Particle Size Distribution Curve

Grain Size-HYD-SK the reported parameter is called “Grain Size Curve”. The test result is listed as “See attached”, which refers to the particle size distribution report which is electronically attached to the file and included with the test report by:

1. Select “Create PDFs” button on “Graph” tab in the GRAIN SIZE-HYD-SK spreadsheet.
2. Move your PDF file from the G:\Enviro\Physical\Grain Size Graph to L:\Client_Attachments\Sublets.
3. Export and Save the spreadsheet from “Generic Export” tab under G:\Enviro\Physical\Physical Data.
4. Make sure the graph is attached on the file.

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10.7 Modifying Limits of Reporting

Where Method Blank contamination above the LORs occurs. Re-prep entire batch.

11.0 REFERENCES

11.1 Primary References

ASTM D6913-04 (2009) Standard Test Methods for Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis

ASTM D7928-16 (2016) Standard Test Methods for Particle-Size Distribution (Gradation) of Fine-Grained Soils Using the Sedimentation (Hydrometer) Analysis

11.2 Secondary References

1. Soil Survey Staff. 2014. Soil Survey Field and Laboratory Methods Manual. Soil Survey Investigations Report No. 51. U.S. Department of Agriculture. Natural Resources Conservation Service. Method 3.2.1
2. Ashworth J et al. 2001. Comm. Soil Sci. Pl. Analysis 32: pp 633-642. Reference for:
   a. Settling time for < 2 micron (clay) particles = 6h, rather than 7h (Kroetsch & Wang).

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b. Validation of the 40-s sand reading time (agreement with % retained on 53 µ sieve).
c. Rationale for blank-adjustment of readings made at ambient temperature, rather than adjusting to a standard temperature.
d. Evidence for method ruggedness towards settling cylinder dimensions.
e. Equivalence of mixing suspensions by inversion, rather than with a plunger (Kroetsch & Wang)

3. Corporate ETLSOP07_101. Reference for calculation of estimated 6h reading based on a 2h reading using a multiplication factor of 0.87 accompanied by a subtraction of a Reagent Blank.
4. Kunze, G.W. (1965). Pretreatment for Mineralogical Analysis. In: C.A. Black(ed.) Methods of Soil Analysis. Physical and Mineralogical Properties. American Society of Agronomy Inc.
5. ASTM D422-63 (2007) Standard Test Method for Particle-Size Analysis of Soils. Previous standard, used for reporting purposes and to maintain compatibility with regulations which may not be updated.

11.3 Supporting ALS Documents and Forms

1. SK-FM-1023b PSA 5% Calgon Reagent Prep
2. SK-WI-009 Soil Drying and Grinding Protocols
3. SK-FM-1009d Orbital Shaker Verification Log
4. SK-FM-1009a –HCl Prep for PSA
5. SK-FM -0075ad Oven Checks
6. SK-FM-0075af PSA Volumetric Device Verification Form
7. SK-FM-1023a -Hydrometer Verification
8. SK-FM-0075a Daily Balance Verification
9. SK-WI-011 Elwood Grinder Operation

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11.4 Instrument Manuals

1. Balance (MS204S/03 , ML204T, ED224s and MS3002S Operating and Service Manual
2. Oven(DNK812C) Operating and Service Manual
3. Standard Orbital shaker (10000-2) Operating and Service Manual.

12.0 DEVIATIONS FROM THE PRIMARY REFERENCE

1. Organic matter is known to cause clay particles to flocculate. The exact OM content that causes flocculation will vary from sample to sample, but a general guide is needed. Although Carter recognizes that soil particle size analysis is routine performed without pre-treatment (except Calgon), he suggests that organic matter (OM) be removed from the sample if OM content is greater than 3.5% by weight. This procedure allows a limit of 10% organic matter based on the work of Ashworth (section 11.2). In addition, tests performed on a highly organic sediment sample show that a 10% OM limit is reasonable. See Appendix 1.

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2. This procedure does not include a step for removal of salts for highly saline samples. Since most saline prairie soils are dominated with sodium chloride, salinity effects should be minimal even at a level of 1000 mg/kg sodium or more. Refer to section 11.2.6.
3. Carter specifies that a second sample be taken for moisture analysis at 110 ± 5 ºC. This moisture value would be used to calculate PSA results on an oven dry weight basis. This moisture value would be used to calculate PSA results on an oven dry weight basis. In this procedure moisture correction is not incorporated, because moisture content of most air-dried soil samples is less than 3%. The absence of moisture correction will cause a maximum error of 2% sand, silt or clay. This is well within the measurement uncertainty specified in section 1.3.
4. Carter suggests a clay reading at 7 hours. In this procedure, we use a clay reading at 6 hours as indicated by Ashworth (listed in section 11.2.3).
5. For grain size analysis, hydrometers are not removed from sedimentation cylinder between readings prior to the 60-minute mark. This is justified as the time between these readings are close together and would result in constant adding and removing of hydrometers which would lead to disturbing the settling rate. This part of the reference is reflective of locations using a single hydrometer for multiple samples. This method uses individual hydrometers for each sample.

13.0 REVISION HISTORY

Version #	Date	Summary of Major Changes Made / By
01	01April, 2019	Initial SOP in national format / HSL

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14.0 WASTE MANAGEMENT

The laboratory has a soil collecting sump system for waste soil slurry. Waste information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

15.0 SAFETY

This task may include CHEMICAL, BIOLOGICAL, OPERATIONAL and/or EQUIPMENT hazards. Staff must review and understand the following hazards and their preventive measures prior to proceeding with this activity. Refer to Material Safety Data Sheets (MSDS) for detailed information about safe handling guidelines and hazards associated with chemicals used in this method.

HAZARD ASSESSMENT		
Job Task #1: Sample Preparation	**Hazards**	**Preventative Measures**
Sample grinding	Flying debris	Wear PPE
Sample grinding	Noise	Wear appropriate ear protection.
Job Task #2: Reagent preparation and usage	**Hazards**	**Preventative Measures**

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Preparing 0.5% Calgon solution	Skin or eye irritation		Wear PPE
Preparing 3 M HCl solution	Danger: Toxicity and skin burns.		Wear gloves, safety glasses and lab coat. Work in fume hood. Avoid skin contact. . Wear a full face shield when working with concentrated acids
Hydrochloric Acid usage	Danger: Reacts with oxidizing materials to produce chlorine gas.		Wear appropriate PPE. Avoid contact with organic materials such as isopropanol. For small spills, neutralize using sodium bicarbonate and wipe up using a wet sponge. For large spills, contact the spill response team. Use an acid spill kit.
Job Task #3: PSA Analysis	**Hazards**		**Preventative Measures**
Plunging to mix soil suspension	Skin or eye irritation	Wear PPE	Plunging to mix soil suspension

Job Task #4:	**Hazards**	**Preventative Measures**
Removing dishes from the oven	Burns on hands.	Wear heat resistant gloves when handling hot sample trays.

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Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

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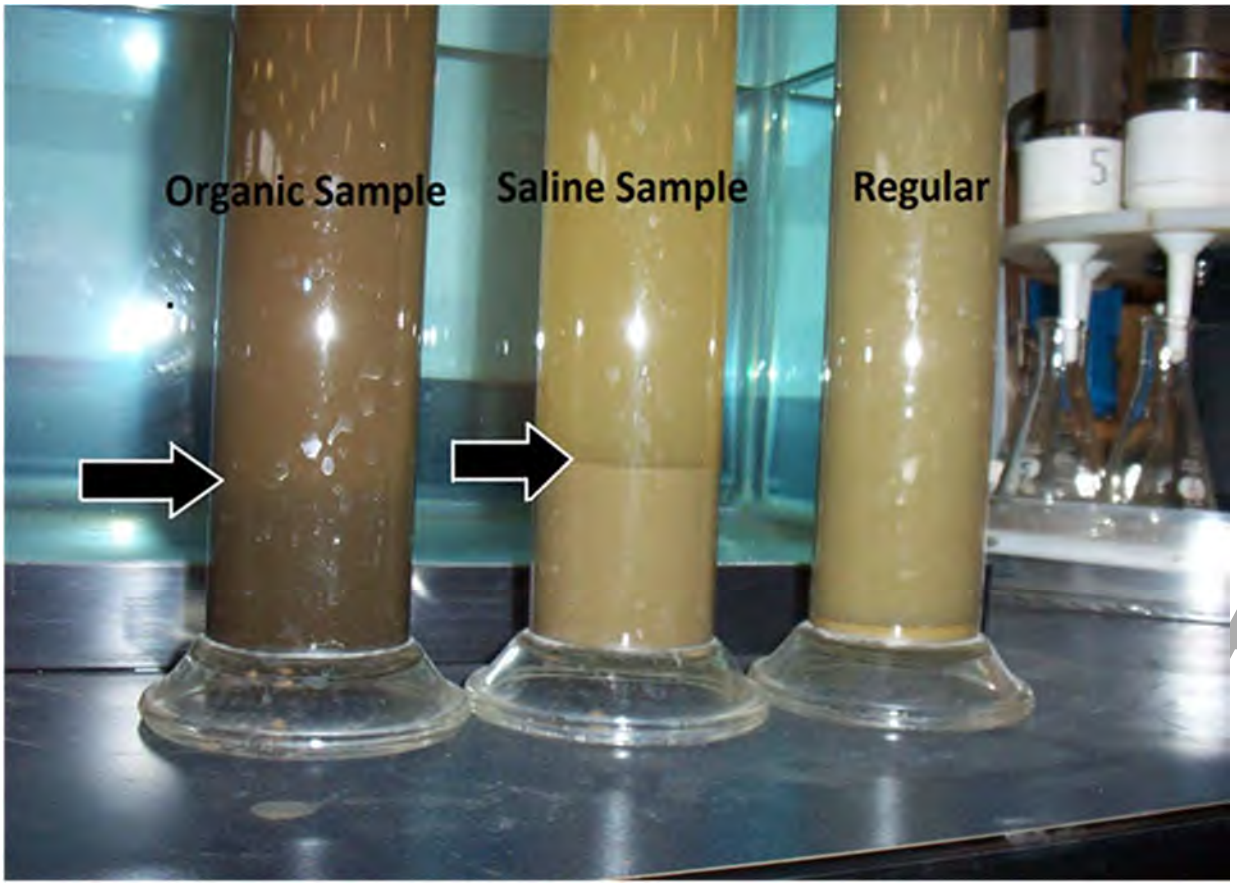
15.0 APPENDICES

Appendix 1: The Effect of Organic Matter and Salinity on Clay



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Appendix 2: Minimum Mass Requirement for Specimen

D6913 – 04 (2009)$^{\varepsilon 1}$

TABLE 2 Minimum Mass Requirement for Specimen

Maximum Particle Size of Material (99 % or more passes)		Minimum Dry Mass of Specimen, g or kg[A]	
Alternative Sieve Designation	Maximum Particle Size, mm	Method A Results Reported to Nearest 1 %	Method B Results Reported to Nearest 0.1 %
No. 40	0.425	50 g	75 g
No. 10	2.00	50 g	100 g
No. 4	4.75	75 g	200 g[B]
⅜ in.	9.5	165 g[C]	D
¾ in.	19.0	1.3 kg[C]	D
1 in.	25.4	3 kg[C]	D
1-½ in.	38.1	10 kg[C]	D
2 in.	50.8	25 kg[C]	D
3 in.	76.2	70 kg[E]	D

[A] Specimen masses should not significantly exceed (by more than about 50 %) the presented values because excessively large specimens may result in sieve overloading, (see 11.3) and increase the difficulty of specimen processing.
[B] The same as "C," except multiplied by 10.
[C] These values are based on the mass of an individual spherical shaped particle, at the given sieve, multiplied by 100 then 1.2 (factor to account uncertainty) and finally rounded to a convenient number.
[D] Specimens of this size require composite sieving. The sample sizes required for reporting results to 0.1 % are not practical and the possible errors associated with composite sieving causes this sensitivity to be unrealistic for specimens with these larger size particles.
[E] Same as "C," except 1.2 factor is omitted.

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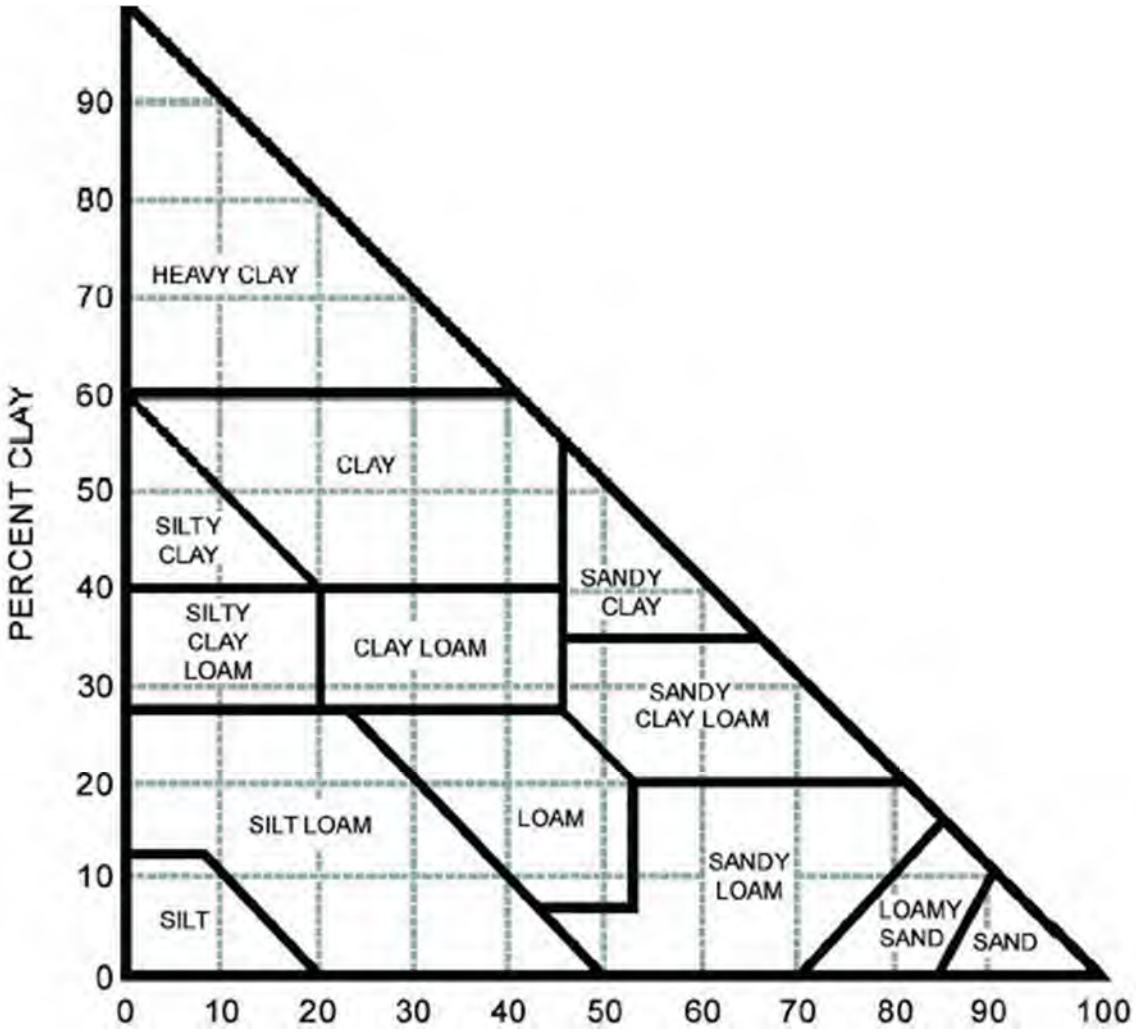
Appendix 3: Soil Textural Triangle (Canadian Soil Survey Committee)



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Appendix 4: Checking for High Levels of Organic Matter

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Analysis of Water, Solids, and Soluble Waste Samples for Semi-volatile Fuel Hydrocarbons

DOCUMENT ID: PET-SVF, REV 17.0

Prepared By: _____ Date: 12/2/2020
Organics Manager, Jonathon Walter

Prepared By: Kurt Clarkson _____ Date: 12/2/2020
Quality Assurance Manager, Kurt Clarkson

Approved By: _____ Date: 12-2-20
Laboratory Director, Charles (Pat) Byrne

1) Scope & Applicability

1.1 This Standard Operating Procedure (SOP) describes the method used for analysis of semi-volatile petroleum hydrocarbons using EPA Method 8015C and state-specific methods, as listed in Table 1.

1.2 The analysis is used for detection and quantification of semi-volatile fuel hydrocarbons ranging from gasoline through fuel oils and lubricating oils. The SOP is applicable for quantitative analysis of Total Petroleum Hydrocarbons as Diesel, Diesel Range Organics, Motor Oil and Residual Range Organics. The SOP is applicable for semi-quantitative analysis of more volatile petroleum products ranging chiefly from n-C8 through n-C11 (e.g., Gasoline). The SOP is also applicable for qualitative identification of petroleum products ranging from n-C8 through n-C44. Normally only the following analytes are reported under this SOP: Diesel Range Organics (various ranges), Residual Range Organics (various ranges) and C8-C12 Gasoline Range Organics. Other petroleum products are reported (quantitatively, semi-quantitatively or qualitatively) when and as requested by the client.

1.3 This SOP describes both the sample preparation and chromatographic procedures used to determine the target analytes in water, soil, sediment and unknown waste (product) matrices. The SOP may be applied to other miscellaneous sample matrices providing that the analyst demonstrates the ability of the SOP to give data of acceptable quality in that matrix. The Method Reporting Limits (MRLs) for target analytes in water and soil are presented in Table 2. Method Detection Limits which have been achieved are also given.

2) Summary of Procedure

2.1 Samples are solvent extracted using methylene chloride; the extracts are injected into the GC system using a micro-syringe. Target analytes are separated using a chromatographic column and quantitation is made by comparison of target analyte response to the response of calibration standards over a known concentration range. Integrations are performed over an extended hydrocarbon range. Chromatographic fingerprints are compared to known fuel and other hydrocarbon and petroleum product fingerprints for qualitative identification. Table 3 provides a list of fingerprints maintained on file in the lab. More fingerprints are generated as necessary, from purchased materials and from suspected contaminants often supplied by clients.

2.2 The SOP is applicable to the analysis of target analytes and meets method criteria for the methods listed in Table 1. The SOP may be applied to qualitative analysis and identification of a wide variety of hydrocarbon products.

2.3 The requested target analyte list is analyzed meeting established method criteria for the compounds of interest as set forth in sections 11 and 12 of this SOP. Calibration standards used to quantitate the various target analytes are listed in Table 1..

3) Definitions

3.1 For laboratory definitions applicable to most analyses, refer to the SOP for *Sample Batches*, ADM-BATCH.

4) Responsibilities

4.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the

results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

4.2 It is the responsibility of the department supervisor/manager to document analyst training and method proficiency, as described in the SOP *Employee Training and New Employee Orientation* (ADM-TRAIN).

5) Interferences

5.1 Solvents, reagents and labware may yield artifacts that will cause responses in various hydrocarbon ranges integrated in the SOP. These materials should be checked and cleaned to prevent positive biases to the data. For interpretive analyses, obvious artifacts resulting from contaminated solvents, reagents, and glassware will not be reported as petroleum products.

5.2 Direct exposure of samples and sample extracts to unregulated labware (such as polymeric tubing attached to a laboratory reagent water spigot), personal protective equipment (such as surfactant-coated nitrile gloves) and human contact (such as skin oils and personal hygiene products) generally yields artifacts that will cause responses in various hydrocarbon ranges integrated in the SOP. Such exposures are to be avoided.

5.3 Contamination by carryover can occur when high level samples immediately precede samples containing significantly lower levels of contamination. This can be minimized by running system blanks after samples that are suspected to contain high levels of target analyte. For purposes of evaluating sequencing criteria during an analytical batch, these system blanks are not defined as samples and therefore are not counted in the ten sample groupings.

5.4 An optional sulfuric acid/silica gel cleanup procedure is provided for removal of chiefly biogenic polar interferences. A state specific (AK 102 and AK 103 tests only), silica gel only cleanup procedure is also provided for, usually to be performed only upon client request.

5.5 This SOP provides for the qualitative identification and quantitative or semi-quantitative measurement of various petroleum products in samples. In practice, a great variety of different organic compounds, many of which are not part of petroleum products, are analyzed. In all cases, all chromatographic responses (exclusive of those due to surrogate compounds) that occur within a hydrocarbon range are reported; a qualitative evaluation of the result is then made and an appropriate Petroleum Hydrocarbon Specific Data Qualifier is added to the reported result.

6) Safety

6.1 Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in SDSs where available. Refer to the ALS Chemical Hygiene Plan and the appropriate SDSs prior to beginning this method.

6.2 This method uses Methylene Chloride, a suspected human carcinogen. Viton gloves should be used while rinsing, pouring or transferring the solvent; if solvent is spilled on the glove surface, remove the glove immediately. Nitrile gloves are more practical for sample preparation but provide only momentary protection; if solvent (especially methylene chloride) is spilled on the glove surface, remove the glove immediately. Latex and vinyl gloves do not provide adequate protection from methylene chloride and

should not be worn during procedures involving large quantities of solvent. All procedures involving the transfer of organic solvents should be performed in a fume hood.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Aqueous Samples:

7.1.1 For methods other than AK102/103 (i.e. 8015, NWTPH-Dx) the default sample size is 500 mL. Samples should be collected (received) in 500 mL glass amber bottles.

7.1.2 For AK102/103 the default sample size is 1L for aqueous samples and it is intended that samples are collected in 1L glass amber bottles, and these are the size provided by the laboratory.

7.1.3 This procedure also includes a "small volume" option for method AK102 only. When requested by the client, 250 mL is the intended sample size and 250 mL glass amber bottles will be used.

7.1.4 Samples should be preserved to pH <2 with hydrochloric acid. Samples will be refrigerated to 4 ± 2°C upon sample login.

7.2 Solid samples should be collected (received) in glass jars with PTFE-lined septa. Alternatively, soil samples may be received in brass sleeves prepared in the field. Samples will be refrigerated to 4 ± 2°C upon sample login.

7.3 Waste samples such as products can be collected (received) in a variety of sample containers. Typically, glass bottles with PTFE-lined septa are preferred.

7.4 Analytical and Sample Preparation Holding Times: See Table 4 for method specific maximum recommended holding times.

8) Standards, Reagents, and Consumable Materials

8.1 Reagents

8.1.1 Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP *Reagent/Standards Login and Tracking* (ADM-RLT) for the complete procedure and documentation requirements.

8.1.2 Acids and Bases

8.1.2.1 Hydrochloric Acid, concentrated

8.1.2.2 Sulfuric Acid, concentrated

8.1.3 Glass wool: baked overnight at 280°C.

8.1.4 Reagent Water: Laboratory reagent water that is free of contaminants of interest. The laboratory reagent water is produced by passing water through a series of deionizers followed by activated carbon filters in series.

8.1.5 Sodium Sulfate (Na_2SO_4): Powdered and granular, baked overnight at 400°C.

8.1.6 Solvents: Reagent Grade acetone, hexane and methylene chloride (DCM). Histological grade acetone (for rinsing prior to DCM rinsing only).

8.1.7 Silica Gel: 100-200 Mesh, Baked for 4 hours at 130°C before use.

8.2 Standards: Standards should be labeled as follows: logbook ID number, compounds or compound mix, concentration, solvent, date prepared, expiration date, and analyst's initials.

8.2.1 Neat Standard Material: non-fuel standards are purchased from a reliable source (e.g., Chem Service). Neat petroleum product is only routinely used to calibrate for Residual Range Organics (30 weight motor oil) but can be used when requested to calibrate for a suspected petroleum product contaminant (e.g., mineral spirits). Neat material used for quantitative analysis is given an expiration date equal to the manufacturer's recommendation, or if no expiration date is provided, the expiration date shall be five years.

8.2.2 Stock Standards: Purchased as solutions from a reliable source (e.g., Supelco) or prepared in high concentration from neat material. Stock standards are generally prepared in methylene chloride. A stock standard is defined as any standard that requires additional dilution prior to being used in the analysis. To prepare stock standards from neat material, accurately weigh the neat material using a four-place analytical balance into a tared volumetric flask. Dilute to volume using methylene chloride. Stopper and invert several times to mix. Transfer the solution to an appropriate storage vial, label and refrigerate. When preparing stock standard mixes from neat material, it is recommended that each component of the mix be prepared individually at high concentration and then combined as a complete mix at a secondary dilution. Stock standards used for quantitative analysis are given an expiration date equal to the manufacturer's recommendation (purchased, sealed solutions) or one year for laboratory prepared stocks and purchased solutions that have been opened for use. The following stock standards are used in this SOP:

8.2.2.1 Aliphatic Hydrocarbon Marker Mix: Multi-State hydrocarbon window defining standard from AccuStandard (or equivalent vendor) containing n-Alkanes from C8-C40, Pristane, and Phytane.

8.2.2.2 Aromatic Hydrocarbon Marker Mix: Purchased or prepared solution containing discrete aromatic and non-aromatic hydrocarbons in methylene chloride. The solution must include Ethylbenzene, *o*-Xylene, *m*-Xylene, *p*-Xylene, 1,2,3-Trimethylbenzene, Fluoranthene, Pyrene, α-Pinene, β-Pinene. It is useful, but not necessary, to include other compounds for characterization of products.

8.2.2.3 Gasoline: Purchased solution containing a three-source composite of gasoline at 50,000 ppm (total) in methanol. Alternately, may be purchased at 5,000 ppm (total).

8.2.2.4 Kerosene: Purchased solution containing kerosene at 50,000 ppm in methylene chloride. Alternately, may be purchased at 20,000 ppm.

8.2.2.5 Diesel #2: Purchased solution containing a three-source composite of diesel #2 at 50,000 ppm (total) in methylene chloride. Alternately, may be purchased at 20,000 ppm (total).

8.2.2.6 SAE 30 Motor Oil: Prepared solution containing 30-weight motor oil at 10,000 ppm in methylene chloride.

8.2.2.7 SAE 30/SAE 40 Motor Oil Mixture: Purchased solution containing 30-weight motor oil and 40-weight motor oil at 50,000 ppm (total) in methylene chloride.

8.2.2.8 Other petroleum products (e.g., Mineral Spirits, Bunker C) may be purchased at a high concentration from a reliable vendor (usually in methylene chloride), or may be prepared from neat material.

8.2.2.9 Fuel Fingerprints: A wide variety of petroleum and other hydrocarbon products may be characterized by this SOP. Table 3 lists fuel fingerprints currently on file or available for qualitative analysis.

8.2.3 Working Standards: Purchased as solutions from a reliable source (e.g., Supelco) or prepared from stock standards. A working standard is defined as any standard that is used directly in sample analysis. Working standards are prepared from stock solutions by accurately transferring a volume of stock standard into a volumetric flask or graduated cylinder partially filled with the dilution solvent (usually methylene chloride). The volume is further adjusted using the dilution solvent to a known level. Working standards used for quantitative analysis are given an expiration date equal to the manufacturer's recommendation (purchased, sealed solutions), or 6 months for purchased solutions that have been opened and for laboratory prepared solutions.

8.2.3.1 Calibration Standards: A series of standards prepared from a common stock ranging in concentration from a value representing the method reporting limit (or lower) to a value near the high end of the linear calibration range. Calibration standards (and continuing calibration standards) are prepared in methylene chloride. Calibrations will contain a minimum of five concentration levels. Recommended calibration standards are listed in Table 5.

8.2.3.2 Surrogate Solution: Using neat products, prepare a solution containing 500 ppm each of *o*-Terphenyl, 4-Bromofluorobenzene, and n-Triacontane in methylene chloride. Since the solution is in a relatively volatile solvent, it is suggested that the working solution be stored in a bottle with a neck that is as narrow as practical, in order to minimize concentration.

8.2.3.3 Working Analyte Spike Solution: Using neat products, prepare a solution in methylene chloride containing Diesel #2 at a concentration of 16,000 ppm and 30-weight Motor Oil at a concentration of 8,000 ppm. Since the solution is in a relatively volatile solvent, it is suggested that the working solution be stored in bottles with necks that are as narrow as practical, in order to minimize concentration.

8.2.3.4 Working AK Analyte Spike Solution: Prepare a solution in methylene chloride containing Diesel #2 at 16,000 ppm, 30-weight motor oil and 40-weight motor oil at 8,000 ppm (total).

8.2.4 Independent Calibration Verification Standard (ICV), also known as Second Source Verification Standard (SSV): A stock standard should be prepared for

each analyte from a second source independent of that used in preparing the calibration curve; this is subsequently used to create a working standard to verify the curve accuracy. Recommended ICVs for the various calibration standards used in this SOP are listed in Table 1.

9) Apparatus and Equipment

9.1 Balance

9.1.1 Analytical Balance

9.1.2 Top Loading Balance

9.2 Centrifuge

9.3 Gas Chromatography System

9.3.1 Gas Chromatograph (GC): analytical system equipped with gas supplies, column inlet, programmable oven, heated detectors (FID), and a data system for determining peak areas. Hewlett-Packard 5890, Agilent 6890, or equivalent.

9.3.2 Chromatographic Column: Phenomenex ZB-1, 15 m x 0.25 mm ID, 1.0 µm film thickness, or equivalent column of comparable chromatographic performance.

9.3.3 Autosampler Device: Designed for operation in conjunction with the gas chromatographic systems used in this SOP, Hewlett-Packard 7673 or Agilent 7683.

9.3.4 Compressed Gases or Gas Generators: Hydrogen (column flow and flame gas), Air (flame gas), and Nitrogen or Helium (make-up gas).

9.4 Labware and Syringes

9.4.1 Beakers: various sizes (125 mL, 250 mL, 400 mL, 600 mL)

9.4.2 Disposable Test Tubes: 15 mL (100 mm x 10 mm), 20 mL (150 mm x 10 mm)

9.4.3 Disposable Test Tubes Caps: Polypropylene, 10 mm

9.4.4 Disposable Pipettes: Pasteur and serological (1mL, 2 mL, 5 mL, 10 mL)

9.4.5 Erlenmeyer Flasks: 500 mL

9.4.6 Brandtech Pipetters (or equivalent): 50µl, 100µl, 500 µl, and 1000 µl

9.4.7 Fleakers: 1000 mL and 500 mL

9.4.8 Graduated cylinders, 1 liter and 50 mL, Class A, TC

9.4.9 PTFE stoppers: of appropriate size for 50 mL graduated cylinder

9.4.10 Inject Vials: 1.8 mL glass autosampler vials with aluminum crimp tops with PTFE-lined silicone septa

9.4.11 Kuderna Danish (KD) Concentration Units:

9.4.11.1 Flask: 500 mL

9.4.11.2 Keck Clip (blue)

9.4.11.3 Receiving Tube (collector): 15mL

9.4.11.4 Snyder Column: 3 or 2 Ball

9.4.12 Microsyringes: various sizes (10 µl, 25 µl, 100 µl, 250 µl, 500 µl, 1000 µl)

9.4.13 Separatory Funnels: 1 liter, 2 liter

9.4.14 VOA vials (40 mL): capped, with PTFE-Lined Septa

9.4.15 Volumetric Flasks: various sizes (1 mL, 2 mL, 5 mL, 10 mL, 25 mL, 50 mL, 100 mL)

9.4.16 Large stainless steel tablespoon

9.5 N-EVAP Nitrogen Evaporation Unit

9.6 pH Paper: narrow range (0-4 pH units) for measuring aqueous sample pH

9.7 PTFE Filters: leur-locking, 0.45 micron

9.8 S-EVAP Concentration Unit

9.9 Sonication Device

9.9.1 Lab Jack

9.9.2 Sonic Controller

9.9.3 Sonic Horn (4 x 1/4" microtip couple or ¾" tip)

9.9.4 Sonibox

9.10 Stainless Steel Scoopulas

9.11 Ultrasonic Bath

9.12 For AK 102 and AK 103 cleanup: Pre-packed Restek silica gel column: 0.5 g silica gel in a 3 mL total volume, PP barrel with PE frits.

10) Preventative Maintenance

10.1 A maintenance log will be kept documenting maintenance performed on each analytical system. Log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements (defined as any non-consumable part essential to operation), phone logs from technical support contacts, a description of the maintenance performed, a description of the check performed to assure the system has returned to acceptable levels of operation, and the analyst's initials.

10.2 Autosampler: Maintenance is generally limited to cleaning or replacing dirty, clogged or otherwise malfunctioning syringes, and cleaning other moving components with methanol when necessary.

10.3 GC System: Maintenance of the GC system is generally limited to, in declining order of frequency:

10.3.1 Replacement or cleaning of the injection port liner, or (more frequently) replacement of the glass wool plug therein. This is indicated by a significant drop in heavier range product (Motor Oil) and alkane (Triacontane) responses without a coincident drop in lighter range product and surrogate responses.

10.3.2 Replacement of the injection port septum. Leaks may occur here.

10.3.3 Replacement or cleaning of the injection port liner seal. The can be indicated by a change in surrogate responses without a coincident or proportionate change in hydrocarbon product responses. Note that the seal usually

functions satisfactorily even when obviously dirty in appearance because petroleum products are generally composed exclusively of 'non-polar' components and are thus not significantly affected by active sites; two notable exceptions are brake fluids, which are composed exclusively of glycol ethers, and Fuel Oil #6 (Bunker C), which contains significant quantities of sulfur-containing compounds.

10.3.4 Column trimming. This is indicated by surrogate peak broadening. Removal of less than two inches of the column at a time is usually sufficient.

10.3.5 Replacement of ferrules or o-rings (or tightening associated connections). Leaks may occur at these points.

10.3.6 Detector cleaning – scrubbing of the detector jet, collector and (less importantly) chimney. This is indicated by a steady increase or decrease (depending on the nature of the acquisition method) in all responses over time with no other discernable cause; or by the appearance of random spikes in chromatograms. This is also necessary if the flames fail to remain lit.

10.3.7 Column replacement. This generally becomes necessary if significant broadening and diminution of responses of early-eluting peaks (and responses of products), especially at lower concentrations (resulting in quadratic curves) is observed. Ready disintegration of the column upon handling is another indication.

11) Procedure

11.1 Cleaning of Labware: Non-disposable labware (except for syringes) used in sample analyses is washed with hot soapy water, rinsed three times with hot tap water, rinsed three times with reagent water, and either allowed to air dry or rinsed with histological grade acetone. Immediately prior to sample preparation, labware must be rinsed with methylene chloride. Syringes are rinsed with the appropriate solvent(s).

11.2 Sample Preparation: A preparation (or extraction) batch is defined as a group of field samples and QC samples processed together with the same reagents. Each preparation batch will consist of no more than 20 field samples. Every preparation batch will include one laboratory method blank (MB) and one laboratory control sample (LCS) at a frequency of not less than one each per 20 client samples. Additional QC sample requirements depend on the methodology (or matrix) employed:

- EPA 8015C: matrix spike (MS) and duplicate matrix spike (DMS) samples are required at a frequency of not fewer than one each per 20 client samples. If insufficient sample is available for a DMS or both MS and DMS, a duplicate laboratory control sample (DLCS) must be prepared at the same frequency.
- AK102/AK103: MS/DMS and DLCS are all required at a frequency of not fewer than one each per 20 client samples.
- NWTPH-DX: a duplicate client sample (DUP) is required at a frequency of not fewer than one per 10 client samples (note higher frequency requirement). If insufficient sample is available for a DUP, a DLCS must be prepared at a frequency of not less than one per 20 client samples. Note that although a client may request MS/DMS as additional QC, the DUP (or DLCS) is still required.
- WASTE or PRODUCT dilutions, all methods: a duplicate client sample (DUP) should be prepared with each batch. It is not necessary to prepare a MS or MS/DMS set (but do prepare a DLCS for EPA 8015C and AK102/AK103).

11.2.1 Aqueous Samples: EPA 3510 Liquid-Liquid Extraction

11.2.1.1 Prepare KD extract collection apparatuses: For each sample, tightly fit a 500 mL KD flask with a 15 mL collector, and secure these with a blue Keck clip. Place this assembly in a secure rack. Drop a clean PTFE boiling chip into the collector. Prepare a modified funnel thus: place a small glass wool plug at the bottom of the funnel (rinse the assembly 3 times at this point with methylene chloride, as the glass wool can be a significant source of contamination), and add about 3 or 4 heaping tablespoons of muffled, granular Na_2SO_4 to the funnel. Place the funnel in the top of the KD flask. Arrange the KD apparatuses in a fume hood.

11.2.1.2 Allow the sample to warm to room temperature and shake to mix. Note that mixing of the sample may in some cases not be desirable. If a significant portion of the sample volume is solid material, consult with the supervisor or Project Manager for guidance.

11.2.1.2.1 Method 8015, NWTPH-Dx (500 mL or 1 L samples) and default AK102/103 (1L samples): Mark the meniscus on the side of the bottle and pour the entire contents of the bottle into a 1 L separatory funnel (2L funnel if more than approximately 600 mL of sample was received in one bottle). Save the bottle for solvent rinsing and sample volume determination (11.2.1.5). Laboratory QC (MB, LCS and DLCS) are each prepared from 500 mL (or 1 L, if any client samples are greater than approximately 600 mL in volume) laboratory reagent water.

11.2.1.2.2 Small volume option (AK102 only - 250 mL bottle): Mark the meniscus on the side of the bottle and pour the entire contents into a 1L separatory funnel. Save the bottle for solvent rinsing and sample volume determination (11.2.1.5). Laboratory QC (MB, LCS and DLCS) are each prepared from 250 mL laboratory reagent water.

NOTE: For AK102, if the client provides less than approximately 600 mL (e.g. limited volume at sampling site) in a 1 L or smaller bottle, the small volume option will be used. In this case, 250 mL of sample is measured in a graduated cylinder, transferred to the separatory funnel, and the graduated cylinder solvent rinsed and transferred to the funnel.

11.2.1.3 Check pH of sample. If sample pH is not < 2, acidify to pH < 2 with concentrated HCl.

11.2.1.4 Add the working surrogate spike to all client and QC samples. Add the working analyte (matrix) spike to all MS, DMS, LCS and DLCS samples. Use the working AK analyte (matrix) spiking solution for the AK102 and 103 methods.

11.2.1.5 Add 60 mL of methylene chloride (DCM) to the empty sample bottle, swirl (in order to extract organics adhering to the bottle), and then pour the mixture into the funnel containing the sample; for laboratory QC, add 60 mL directly to the funnel.

11.2.1.6 Determine the sample volume (when the entire sample volume was used) by filling the sample bottle with water to the marked meniscus. Transfer the contents of the bottle to a graduated cylinder to measure the volume of the sample prepared. Record the volume prepared to at least 2 significant figures.

11.2.1.7 Stopper and shake the funnel for 1-2 minutes with periodic venting. Allow the solvent layers to separate.

11.2.1.8 Drain the Extract into an Erlenmeyer flask.

11.2.1.9 If an unresolvable emulsion is encountered, first drain the emulsion into another vessel for mechanical resolution, preliminary drying employing granular Na_2SO_4, or centrifugation, followed by pipetting off of aqueous layer; then collect the DCM layer as in 11.2.1.8.

11.2.1.10 Repeat the extraction twice more in sequence, starting each with a fresh 60 mL portion of DCM added directly to the sample in the separatory funnel. Pour the 3 extracts in the KD apparatus for concentration. Rinse the extract flask 3 times with DCM, adding to the appropriate KD apparatus each time. Finally, rinse the contents of the modified with DCM thoroughly and use a vacuum to pull remaining extract into the KD apparatus.

11.2.1.11 Remove the modified funnel from the KD and fit the KD with a Snyder column. Concentrate the extract by means of a hot water bath (S-Evap) at 70°C-75°C. Remove the KD apparatus from the bath when the apparent solvent level in the collector is approximately 5 mL. Do not allow the extract in the collector to evaporate to dryness.

11.2.1.12 Carefully remove the collector from the KD and further concentrate the extract, using a gentle N2 stream and < 40°C water bath (N-Evap) or TurboVap or micro-snyder, to 1 mL in DCM (if the sample volume was approximately 500 mL; 2 mL if sample volume was approximately 1L). Again, do not allow the extract to evaporate to dryness. If the extract is particularly dark, viscous and/or obnoxious smelling, it may be advisable to take the extract to a higher final volume. A graduated, disposable pipet or syringe may be used to measure the final volume.

11.2.1.13 Place 1 mL of extract in a 1.8 mL autosampler vial and cap tightly with a PTFE-lined, silicone septum (natural rubber septa introduce irremovable, MDL/MRL level artifacts into the extract upon injection, and should not be used. Archive any remaining extract in a capped, disposable test tube.

11.2.1.14 Mark the extract meniscuses. Complete paperwork, taking care to note all problems, difficulties or deviations from the standard operating procedure.

11.2.2 Solid Samples: Sonication

11.2.2.1 Prepare and arrange KD extract collection apparatuses as in 11.2.1.1.

11.2.2.2 Sample aliquots are prepared by the appropriate group according to (SOILPREP-ALIQUOT) and (SOLPREP-SUBS). Determinations of sample % dry weight - In certain cases, sample results are desired based on dry

weight basis. Refer to the SOP for *Total Solids* (GEN-160.3) for water samples or *Total, Fixed, and Volatile Solids in Solid and Semisolid Samples* (SOILPREP-SOLIDS) for solid samples. If the determination is performed by the organics preparation personnel, a portion of the sample for this determination should be weighed out at the same time as the portion used for analytical determination..

11.2.2.3 Less sample may be prepared if the sample obviously contains high levels of petroleum product, and a homogeneous mixture can be attained. Muffled laboratory matrix sand is employed as the LCS/DLCS matrix; no matrix is employed for the MB (except a typically employed amount of muffled Na2SO4, which is added to all samples). Record weight prepared to at least 3 significant figures.

11.2.2.4 Add approximately 30 g muffled, granular Na2SO4 to the sample in the beaker, stir the mixture thoroughly with a scoopula until the mixture is dry and free flowing, and no material adheres to the beaker or scoopula. After initial mixing, it will be necessary to allow the mixture to sit at room temperature for at least 15 minutes (often longer), followed by further mixing, in order to ensure that the Na2SO4 has been allowed to dry the sample as effectively as possible.

11.2.2.5 Tune the sonic horn controller(s) in accordance with the manufacturer's instructions and record the action on the benchsheet.

11.2.2.6 Arrange the sample mixtures in a fume hood and keep them in the hood during the following steps (11.2.2.7 through 11.2.2.14) in order to minimize personal solvent exposure. Add working surrogate spike to all client and QC samples. Add working analyte (matrix) spike to all MS, DMS, LCS and DLCS samples.

Note: The AK103 methods require the use of the working analyte matrix spiking solution described in sec 7.2.3.4.

11.2.2.7 As soon as practicable, add approximately 100 mL of DCM to the mixture. Add more or less solvent as necessary, such that the solid mixture is immersed under at least 2 cm of solvent. Since DCM is quite volatile, it will be necessary to cover those sample mixtures (with Al foil or watch glasses, e.g.) which are not immediately taken to the next step in order to minimize evaporation and consequent exposure.

11.2.2.8 Place the sonic horn tip in the solvent layer above the top of the solid mixture and sonicate for 3 minutes at a 2 second pulse interval.

11.2.2.9 Decant as much of the extract as possible off the solid mixture into a KD apparatus.

11.2.2.10 Rinse the top of the Na_2SO_4 bed in the funnel (and whatever solid mixture was transferred with the extract) with approximately 20 mL of DCM.

11.2.2.11 Repeat the extraction (steps 11.2.2.8 through 11.2.2.10) twice more in sequence, starting each with a fresh portion of DCM sufficient to cover the solid mixture under at least 2 cm of solvent. Combine the 3 extracts in the same KD apparatus for concentration.

11.2.2.12 After the third decantation, and before rinsing the Na_2SO_4 bed, transfer the entire solid matrix (or as much as will fit) to the modified funnel (a stream of DCM squirted into the beaker as it is held over the funnel is usually sufficient to accomplish the transfer); then thoroughly rinse the mixture in the funnel with approximately 30 mL of DCM. A vacuum pump may be employed to maximize extract collection in the KD apparatus.

11.2.2.13 Remove the modified funnel from the KD and fit the KD with a Snyder column. Concentrate the extract by means of a hot water bath (S-Evap) at 70°C-75°C. Remove the KD apparatus from the bath when the apparent solvent level in the collector is approximately 5 mL. Do not allow the extract in the collector to evaporate to dryness.

11.2.2.14 Carefully remove the collector from the KD and take the extract to 10 mL in DCM in a disposable test tube. If the extract is particularly dark, viscous and/or obnoxious smelling, it may be advisable to take the extract to a higher final volume. If further concentration of the extract is necessary, use a gentle N2 stream and < 40°C water bath (N-Evap) or TurboVap. Again, and more importantly at this point, do not allow the extract to evaporate to dryness. A graduated, disposable pipet is used to measure the final volume.

11.2.2.15 Place 1 mL of extract in a 1.8 mL autosampler vial and cap tightly with a PTFE-lined, silicone septum (see note about natural rubber septa in 11.2.1.13). Cap and archive the test tube containing the balance of the extract.

11.2.2.16 Mark the extract meniscuses. Complete paperwork, taking care to note all problems, difficulties or deviations from the standard operating procedure.

11.2.3 Extract Cleanups (for removal of mostly biological interferences)

Note: If a sample cleanup is performed, a portion of each of all associated laboratory QC and common client QC must also undergo the same cleanup.

11.2.3.1 Sulfuric Acid/Silica Gel (for EPA 8015C and NWTPH methods):

11.2.3.2 Measure a volume (usually 1 or 2 mL) of the extract at final volume into a disposable test tube. Note: if the extract is not in DCM, it will be necessary to exchange the portion to be cleaned up by this method into DCM before proceeding further.

11.2.3.3 Add 2-3 mL of concentrated H2SO4. Tightly cap the test tube and vigorously but carefully vortex the mixture for approximately 15 seconds.

11.2.3.4 Centrifuge the mixture (at least 200 G for 5 minutes is recommended) in order to maximize separation of the layers; the extract will be the top layer.

11.2.3.5 Add 1-2 g activated silica gel to a separate test tube (cooling to room temperature, if directly from oven). Carefully (avoiding the H2SO4) draw off as much of the extract layer as practicable from the acid layer in the H2SO4-cleanup tube and transfer it to the silica gel-cleanup tube.

11.2.3.6 Tightly cap the test tube and gently swirl the contents. Allow the mixture to sit for at least 30 minutes at room temperature.

11.2.3.7 Carefully (avoiding the silica gel) transfer an aliquot of the extract to a 1.8 mL autosampler vial. Cap the vial tightly with a PTFE-lined, silicone septum (see note about latex septa in 11.2.1.13).

11.2.3.8 Mark the extract meniscus. Complete paperwork, taking care to note all problems, difficulties or deviations from standard operation procedure.

11.2.4 Silica Gel (ADEC Technical Memorandum 06-001 for AK102/AK103):

11.2.4.1 Place a pre-packed Restek silica gel column (0.5 g silica gel in a 3 mL total volume PP barrel with PE frits) in a disposable, 150 mm x 10 mm (length x diameter) test tube (the waste collection vessel). The lip at the top of the barrel will allow it to sit at the top of the test tube without falling in. Alternatively, a handmade column may be used.

11.2.4.2 Precondition the column with at least 12 mL DCM, draining eluent to waste: DO NOT FORCE THE SOLVENT THROUGH THE COLUMN; allow the solvent to drip under the force of gravity ONLY. After the last of the DCM has eluted (the PE frit atop silica gel can go dry; it takes about a minute for the silica gel itself to start drying), remove the column from the waste collection vessel and place it in an extract collection vessel (another 150 mm x 10 mm test tube).

Note: The entire silica gel bed at this point should have a translucent appearance, and it should also appear darker than when new.

11.2.4.3 Introduce exactly 0.50 mL extract to the column, collecting the eluent. Allow the PE frit to just go dry.

11.2.4.4 Add approximately 1.5 mL DCM to column, collecting the eluent. Allow the PE frit to just go dry.

11.2.4.5 Repeat step 11.2.4.1. Stop collecting eluent once it stops dripping from column: UNDER NO CIRCUMSTANCES SHOULD THE COLUMN BE FORCED TO DRYNESS by vacuum or pressure. Remove the column from the extract collection vessel: there will still be some solvent adhering to column exit. This is normal, and its presence does not imply the loss of any desired compounds of interest.

11.2.4.6 Gently concentrate the now cleaned-up (approximately 3.5 mL) extract, using a gentle N2 stream and < 40°C water bath (N-Evap), to 0.50 mL in DCM, and transfer to a 1.8 mL autosampler vial. Do not allow the extract to evaporate to dryness. Cap the vial tightly with a PTFE-lined, silicone septum (see note about natural rubber septa in 11.2.1.13).

11.2.4.7 Mark the extract meniscus. Complete paperwork, taking care to note all problems, difficulties or deviations from standard operation procedure.

11.2.5 TCLP leachates are prepared as aqueous samples. Prepare a leachate blank in addition to the laboratory blank.

11.3 Gas Chromatography

11.3.1 See Table 6 for suggested GC operating conditions. Samples are analyzed as part of an analytical sequence. Sequencing guidelines are described in section 3.2. Sequences are programmed in the GC operating software and run on user command. Prior to initiating an analytical sequence, check that gas supplies and autosampler rinse cup reservoirs are adequate to last the entire sequence.

11.3.2 Establish retention time windows for hydrocarbon ranges. For hydrocarbon range analytes, start and end times are established by analyzing a marker solution containing individual compounds that define the quantitation range. See Table 7 for guidance on establishing windows for the various method specified ranges. The aliphatic hydrocarbon marker mix should be analyzed at least once every 24 hours to verify no significant retention time shift has occurred with respect to start and end times for the quantitation ranges. The aromatic hydrocarbon marker mix should be analyzed at least once every 24 hours.

11.3.3 Method 8015C also describes background subtraction that should be performed, as follows: "Because the chromatographic conditions employed for DRO analysis can result in significant column bleed and a resulting rise in the baseline, it is appropriate to perform a subtraction of the column bleed from the area of the DRO chromatogram. In order to accomplish this subtraction, analyze a methylene chlorine blank during each 12-hour analytical shift which samples are analyzed for DRO. Measure the area of this chromatogram in the same fashion as is used for samples by projecting a horizontal baseline across the retention time range for DRO. Then subtract this area from the area measured for the sample and use the difference in areas to calculate the DRO concentration". Automated software functions (e.g. the instrument's column compensation feature) are typically used to perform this column bleed subtraction from a blank run. Perform column compensation between analytical batches once baseline has stabilized or as needed (CCB concentration approaching MDL).

11.3.4 Calibration

NOTE: The calibration procedure(s) and options chosen must follow the ALS protocols. Any exceptions to the calibration procedures detailed in SOP *Calibration of Instruments for Organics Chromatographic Analyses* (SOC-CAL) are described as follows:

11.3.4.1 Average of Response Factor: Calibration factors are calculated as the total area of the peak or integration range divided by the amount injected. If the percent relative standard deviation (%RSD) of all points is less than 20% (25% for Alaska Methods AK 102 and AK 103), linearity through the origin may be assumed and the average calibration factor may be used in quantifying sample data.

Note: For fuel analytes the integrated area for the surrogate compound is subtracted from the total area for the fuel pattern. This is done to eliminate the contribution of the surrogate area within the fuel range. The EnviroQuant software automatically performs this operation.

11.3.4.2 Calibration Curve, Linear Regression: This option may be used if the correlation coefficient is greater than or equal to 0.995 ($r > 0.995$, $r2 > 0.990$). This method of quantitation uses the equation of a line

(y=mx+b). It will generally provide an acceptable fit when %RSD is < 30% and is therefore a useful option in evaluating the acceptability of the calibration curve for generating quantitative values.

11.3.5 Evaluation of calibration curves for use in quantitative analysis should be performed in the following manner.

11.3.5.1 If %RSD for the compound is less than 20% (25% for Alaska Methods AK 102 and AK 103) use average of response factor from the calibration. If %RSD exceeds this value but is less than 30%, plot a linear regression.

11.3.5.2 For the NWTPH method: the measured result of every calibration standard employed in the initial calibration must be within 15% of its true value. For the 8015C method, the measured result of the lowest concentration calibration standards used must be within 30% of its true value; other calibration standards must be within 20% of the true value.

Note: For NWTPH-Dx analysis, when evaluating the initial calibration linearity the linear model is used, and when quantifying results the average response factor is used.

11.3.5.3 If these guidelines are exceeded, obtain a secondary opinion from a senior analyst or supervisor. The calibration may need to be reanalyzed.

11.3.6 Verify the calibration by analyzing an independent calibration verification standard. Acceptance criteria for the ICV are ±20% for 8015C, ±25% for Alaska Methods AK 102 and AK 103; and ±15 % for NWTPH.

11.3.6.1 Analyze a CCB to establish that the instrument is free of target analyte contamination.

11.3.6.2 The first set of 10 samples in an analytical sequence may be analyzed immediately following an acceptable calibration and analysis of a CCB. This set of samples is bracketed with continuing calibration verification (CCV) standard. This pattern is continued until the sequence is complete, or a CCV fails to meet acceptance criteria described below.

11.3.6.3 Verify the calibration each working day by the analysis of a CCV (mid-level concentration of the calibration curve). A mid-level standard (CCV) must be injected at the start of each sequence and after each set of sample extracts (every 10 samples or every 12 hours, whichever is first) in the analysis sequence. Table 8 lists method specific criteria for evaluating the acceptability of a CCV. See the SOP for *Calibration of Instruments for Organic Chromatographic Analyses* (SOC-CAL) for allowable exceptions.

11.3.7 Evaluation of the Analytical Sequence

11.3.7.1 Check that CCVs and CCBs were interspersed throughout the sequence at the proper frequency. Nonconformances must be documented and obtain supervisory approval before data is accepted.

11.3.7.2 Check retention times in the aliphatic hydrocarbon marker mix and aromatic hydrocarbon marker mix. Update retention times as needed

11.3.7.3 Check that CCVs and CCBs are acceptable. CCV acceptance criteria are described in Table 8. CCBs should be free of all target analyte contamination at a level less than the MRL.

11.3.7.4 Check Method Blanks for potential extraction batch contamination. Extraction Method Blanks should be free of all target analyte contamination at a level less than the MRL. In the event that a Method Blank contains target analyte greater than or equal to the MRL, samples testing positive for that compound at levels less than 20 times the level detected in the Method Blank should be reanalyzed or the associated data must be flagged to indicate potential laboratory contribution.

Note For DOD Projects: If the Method Blank contains target analyte(s) greater than or equal to ½ the MRL, the associated samples, where the analyte(s) are detected at a concentration greater than or equal to the LOD and is less than 10 times the amount detected in the Method Blank, require re-analysis or the data must be flagged with appropriate qualifiers. This project-specific requirement may apply to other projects as well.

11.3.7.5 Check Method Blank and LCS surrogate recoveries. Surrogate recoveries for MBs and LCSs are used to make inferences about associated environmental samples. Low recoveries in the MB and/or LCS constitute an out-of-control event for the entire batch of samples and all samples in the batch testing positive for any target analyte should be re-analyzed or the data must be flagged to indicate potential laboratory contribution. High recoveries in the MB and/or LCS show a potential high bias. However, if all target analyte concentrations (in associated samples) are below the MRL, no corrective action is required other than flagging the high surrogate recoveries in the analytical report.

11.3.7.6 In order to report the associated sample results, all LCS recoveries must be within acceptance criteria. If the LCS recovery is outside the criteria due to chromatographic problems (injection problem, etc.) take the necessary corrective action and reanalyze. If reanalysis does not correct the problem or if the LCS recovery is outside the criteria due to extraction/preparation failure, re-extract and reanalyze the associated samples.

11.3.7.7 If matrix spike recoveries are outside acceptance criteria, examine corresponding sample results for potential matrix interferences such as high levels of target analyte (spike levels should be a minimum of 4 times the background concentration). If no obvious matrix interferences are observed, evaluate the recovery of the laboratory control sample (LCS). Reanalyze if deemed appropriate. Do not re-extract the matrix spike unless required by the project.

11.3.7.8 Check RPD values on duplicate sample analyses (sample/duplicate or MS/DMS). If values exceed the criteria, examine the chromatograms, bench sheet comments and the actual sample for potential matrix

interferences. Examples include product layers on aqueous samples that may result in non-homogenous sub-sampling, severe emulsions, non-homogenous solid samples, inefficient extraction in one sample compared to another as indicated by significantly different surrogate recoveries. Flag RPD values that are outside of acceptance criteria and explain the anomalies in the form of a case narrative. Reanalysis and/or re-extraction and reanalysis may be required, see supervisor.

11.3.7.9 Check RPD values on duplicate LCS/DLCS analyses (when applicable). If values exceed criteria, examine the data for obvious problems. Reanalyze one or the other LCS if deemed appropriate. If reanalysis does not correct the problem, re-extract and reanalyze the associated samples.

11.3.8 Evaluation of Sample Analysis - ALS acceptance criteria are established based on either the method criteria or on control charting of analytical results. Unless specific criteria are required by the method, or stated in project specific data quality objectives, ALS-generated criteria will be used in evaluating acceptability of the analysis.

11.3.8.1 Calculate surrogate recovery as the amount detected in the sample (solution concentration) divided by the true value (solution concentration). If surrogate recovery is outside acceptance criteria examine the chromatogram for obvious matrix interferences such as high levels of target analyte or co-eluting peaks. If the problem is the result of obvious matrix interference, flag the recovery value as outside acceptance criteria due to matrix interference. If no obvious interference is observed, reanalyze (or re-extract and reanalyze if deemed more appropriate) the sample. If surrogate recovery is again outside criteria (or acceptable but within 5% of the primary analysis), report the original value and flag the recovery as being outside of acceptance criteria due to matrix interference. Otherwise, report the reanalysis.

11.3.8.2 Examine solution concentrations of target analytes in the samples. If the concentration is greater than the high calibration standard, reanalyze the sample at a dilution. Preferably, dilutions should be performed to keep target analytes in the top half of the calibration range.

11.3.8.3 Check for possible carryover. Pay particular attention to situations where samples containing low levels of target analyte were analyzed one or two runs after samples containing levels of target analyte near or above the high end of the calibration range. Reanalyze as needed.

11.3.8.4 Check integrations. Where possible, all integrations should be performed consistent with integration of the corresponding calibration standards. Hydrocarbon ranges are integrated baseline to baseline, dropped to the lowest point in the chromatographic window. The integration software currently in use is usually capable of performing the integrations (and subtracting any responses contributed by added surrogates) satisfactorily.

11.3.8.5 Perform qualitative evaluation of the chromatographic fingerprint. This is done by comparing the fingerprint of the sample to the two

marker mixes and the fingerprint library. The marker mixes are used to assess qualitatively the presence of common fuel components. The qualitative evaluation is used to report specific hydrocarbon products in the interpretive analyses (EPA Method 8015C) and to qualitatively flag the results for hydrocarbon range analyses. The following guidelines are provided:

- Gasoline is present if ethylbenzene, xylenes, and 1,2,3-trimethylbenzene, and a small hump of unresolved responses eluting about and after 1,2,3-trimethylbenzene are observed. Ethylbenzene and the xylenes should have the same pattern as the calibration standard (i.e. relative peak height to each other). Weathered gasoline is indicated by relative ethylbenzene and xylene responses that are different than in the calibration standard.
- Diesel is present if the fingerprint shows a recognizable n-alkane pattern that includes the isoprenes pristane and phytane, and a large hump of unresolved responses that elute from C10-C23 with its apex at about C15. 1,2,3-trimethylbenzene is usually distinguishable. Weathered diesel is indicated by (1) pristane/C17 and phytane/C18 response ratios greater than those in the calibration standard, (2) the later shifting of the unresolved response hump relative to that in the standard (yet pristane and phytane are observed) or (3) the presence of pristane and phytane with the lack of any recognizable n-alkanes. In the latter case, the unresolved hump is typically later eluting than in the standard, but should still be primarily within the diesel window; if most of the hump is outside the diesel window, the responses may be due to another fuel oil.
- Bunker C is present if the fingerprint shows a recognizable n-alkane pattern that includes pristane, phytane and contains fluoranthene and pyrene in approximately the correct ratio for Bunker C. Note that the aromatic compounds are more susceptible to weathering than the isoprenes, yet less susceptible to weathering than the n-alkanes.
- Lubricating range oil is present if the responses are chiefly a large hump of unresolved responses that resembles that of the 30-wt. Motor Oil calibration standard and usually elutes in approximately the same or heavier carbon range as that of the hump in the standard. The humps of lubricating oils of higher viscosity than the motor oil calibration standard elute later than that of the standard while those of lighter viscosity elute earlier, but still mostly within the motor oil range.
- Hydraulic fluids fingerprints resemble that of motor oil, and can indeed resemble 30-wt. motor oil in appearance and retention times, but often elute earlier, mostly in the latter half of the diesel range, and sometimes possess more than one, yet unresolved, hump (a large hump with a large shoulder).
- Transformer oil fingerprints also resemble that of motor oil, and are typically one simple hump, but elute earlier than hydraulic fluids, entirely within the early to middle regions of the diesel range.

- Turpentine is present if alpha pinene and beta pinene are observed in the chromatogram, in high concentrations (especially alpha pinene) relative to other extracted organics in the approximate elution range.
- Creosote is present if the chromatographic responses are due chiefly to PAHs, with many resolvable yet unidentifiable individual responses from C12-C36.
- Mineral spirits elute in a very short window approximately between C8-C14 with, in a relatively unweathered sample, the top of the hump centered between C10-C11, and the individual n-alkanes between C9-C12 especially prominent.
- Kerosene (and Jet A fuel; the two are chromatographically indistinguishable) is present if the fingerprint shows a recognizable n-alkane pattern that includes the isoprenes pristane and phytane (though all components eluting this late are at very low relative concentration and thus may not be observed if the concentration is relatively low), and a large hump of unresolved responses that elute from C8-C17 with its apex at about C12. 1,2,3-trimethylbenzene is more prominent than in diesel.
- Military jet fuels: Fingerprints can vary from site to site (and from supplier to supplier), and can have additives not present in kerosene, specifically ethylbenzene and xylenes. JP-5 tends to resemble kerosene, eluting at a slightly (extent can vary) earlier retention time; pristane/C17 and phytane/C18 are just distinguishable if the concentration is high enough. JP-4 resembles kerosene much less, and elutes at a much earlier retention time; pristane/C17 and phytane/C18 are not usually observed. JP-8 tends to resemble kerosene, eluting within the same window but with a flatter appearing hump; pristane/C17 can be observed but phytane/C18 usually cannot be observed.
- Weathered products: All fuels generally demonstrate weathering characteristics similar to those enumerated above for diesel. As a fuel is weathered, (1) its hump tends to shift later in the chromatogram, due to evaporation, (2) individual, semi-resolved, peaks in the hump become less resolved (the hump looks smoother) due to microbial weathering, (3) n-alkanes become much less prominent or disappear, also due to microbial action, and (4) individual marker compounds become more prominent relative to the total chromatographic responses due to their greater resistance to microbial weathering. For the purposes of this SOP (that is, among those marker compounds identifiable in this SOP), survivability of compounds increase in the following order:

  1 - n-alkanes < aromatics < branched alkanes
  2 - branched alkanes include the isoprenes pristane and phytane.

- Field samples often contain mixtures of many different petroleum products. Interpretation of petroleum product mixtures includes a

significant amount of analytical judgment and should be confirmed by an analyst experienced with interpretation or the supervisor.

Note: Alaska DEC requires both pre- and post-silica gel cleanup results be provided. The extract(s) must be analyzed per the standard AK102/AK103 methods, silica gel cleanup performed, re-analyzed and both results reported. Therefore, LIMS test codes AK_DRO_SGT or AK_RRO_SGT mean that both the non-SGT and SGT treated analyses will be reported.

12) QA/QC Requirements

12.1 Initial Precision and Recovery Validation

12.1.1 The accuracy and precision of the procedure must be validated before analysis of the samples begins, or whenever significant changes to the procedures have been made. IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

12.1.2 Perform IPR studies by preparing and analyzing four replicate laboratory control samples spiked at a level of 1 to 4 times the MRL. Calculate average percent recovery and relative standard deviation for the four replicate analyses. Unless specified by the method, acceptance criteria are the control limits used for LCS percent recovery and % RSD.

12.1.3 Calculate the average concentration found (x) in µg/mL, and the standard deviation of the concentrations (s) in µg/mL for each analyte. Calculate the MDL for each analyte. Refer to CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of detection and Quantification*. The MDL study must be verified annually.

12.2 Limits of Quantification (LOQ)

12.2.1 The laboratory establishes a LOQ for each analyte as the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements. Analysis of a standard or extract prepared at the lowest point calibration standard provides confirmation of the established sensitivity of the method. The LOQ recoveries must be within laboratory established acceptance range to verify the data reporting limit. Refer to CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of detection and Quantification*.

12.3 The Method Reporting Limits (MRLs) used at ALS are the routinely reported lower limits of quantitation which take into account day-to-day fluctuations in instrument sensitivity as well as other factors. These MRLs are the levels to which ALS routinely reports results in order to minimize false positive or false negative results. The MRL is normally two to ten times the method detection limit.

12.4 Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for *Sample Batches* (ADM-BATCH). Additional QC Samples may be

required in project specific quality assurance plans (QAPP). For example projects managed under the DoD ELAP must follow requirements defined in the DoD *Quality Systems Manual for Environmental Laboratories.* General QA requirements for DoD QSM are defined in the laboratory SOP, Department *of Defense Projects - Laboratory Practices and Project Management* (ADM-DOD/ADM-DOD5). General QC Samples are:

12.4.1 QA/QC Sample Analysis: Each sample preparation batch will contain a maximum of 20 field samples, one Method Blank, and one Laboratory Control Sample. Additional QC analyses are performed at a rate of one set per 20 field samples. Unless stated in project specific data quality objectives, the standard QA/QC sample set for aqueous and solid matrices is Matrix Spike/Duplicate Matrix Spike. A Duplicate client sample is required for the NWTPH method at a rate of no less than one per 10 field samples. A Duplicate Laboratory Control Sample is required for Alaska Methods AK 102 and AK 103, and when the appropriate Matrix Spike/Duplicate Matrix Spike or Duplicate client sample requirements cannot be met (due to limited sample volume). The standard QC sample set for miscible waste samples is a Duplicate client sample.

12.4.2 QC Analyses: The acceptance criteria for QC analyses are listed in Table 9. The acceptance criteria are established based on method-required criteria or from control charting of analytical results. Surrogate recoveries for new compounds or analyses not charted are set at 70-130%. Unless criteria are required by the method or stated in project specific data quality objectives, ALS criteria determined from historical results will be used in evaluating acceptability of the analysis.

12.5 Section 11 provides guidelines for corrective action when QC sample recoveries are outside acceptance criteria.

13) Data Reduction and Reporting

13.1 External Standard Calibration: The concentration of each compound in the sample is determined by calculating the amount of analyte injected using the calibration curve or the calibration factor. Calculations for determining specific analyte concentrations in the sample are described below.

Calibration Factors

For external calibration, the calibration factor is calculated as:

$$Cf = \frac{Rx}{Ax}$$

Where: Cf = Calibration factor
Rx = Response of the analyte (area)
Ax = Concentration of analyte injected (µg/mL).

13.2 Results based on average of response factor or a linear regression is calculated as:

Solution Concentration of Analyte Detected

$$Csol = \frac{Rcmpd}{Cf}$$

Where: $Csol$ = Solution concentration of the compound (mg/L)
$Rcmpd$ = Response of the compound (area)
Cf = Calibration factor

Sample Concentration of Analyte Detected

Aqueous Samples: $$Rsult(ug/L) = Csol \times \frac{(Fvol \times Df)}{Vsmp}$$

Solid and Waste Sample: $$Rsult(mg/Kg) = Csol \times \frac{(Fvol \times Df)}{Wsmp}$$

Where: $Rsult$ = Sample result (ppm)
$Csol$ = Solution concentration of the compound (mg/L)
$Fvol$ = Final volume of the extraction (mL)
Df = Dilution factor (if no dilution was performed Df = 1)
Cf = Calibration factor
$Vsmp$ = Volume of sample extracted (L)
W_{smp} = Weight of sample extracted (g), in dry weight or wet weight, depending on the reporting basis of the data.

13.3 Results of extracted solid and waste samples are reported in mg/Kg (ppm). Solid sample results are usually reported on a dry-weight basis.

13.4 Data Review

13.4.1 Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to SOP *Laboratory Data Review Process* (ADM-DREV) for details.

13.5 Reporting

13.5.1 Reports are generated in the ALS LIMS reporting software by compiling the SMO login, sample prep database, instrument date, and client-specified report requirements (when specified). The forms generated may be ALS standard reports, DOD, or client-specific reports. The compiled data from LIMS are also used to create EDDs.

13.5.2 Sample concentrations are reported when all QC criteria for the analysis have been met or the results are qualified with an appropriate footnote. Refer to the SOP for *Data Reporting and Report Generation* (ADM-RG) for specific procedures and guidelines used for reporting analytical results and generating analytical reports.

13.5.3 Data Qualifiers

13.5.3.1 Data should be qualified when significant information regarding the analysis needs to be communicated to assist the user in determining the usability of the results. Acceptable qualifiers should comply with standard ALS (Kelso) data qualifiers.

13.5.3.2 When it is requested that results for Gasoline or lighter analytes be reported, these results are to be reported as estimated values and appropriately narrated.

13.5.3.3 Always use data qualifiers to describe hydrocarbon products. Refer to *Data Reporting and Report Generation* (ADM-RG), Appendix B for the specific hydrocarbon qualifiers:

- F-The chromatographic fingerprint of the range in question approximately matches that of the calibration standard used to quantify the result.
- Y-The chromatographic fingerprint of the range in resembles a petroleum product that elutes approximately within the range of integration of the calibration standard used to quantify the result but does not match the chromatographic fingerprint of the standard. A sufficiently weathered product that would otherwise match the appropriate calibration standard also falls into this category.
- L,H-The chromatographic fingerprint of the range in question mostly matches that of a petroleum product, but is actually a partial contribution of a product which elutes mostly in a lighter or heavier range (e.g., motor oil partially eluting in the diesel range), respectively.
- O-The chromatographic fingerprint in a range later than the Diesel range resembles that of oil, but does not match that of the calibration standard used to quantify the result.
- Z-The hydrocarbon responses in the range in question are primarily not due to response from a petroleum product.

14) Contingencies for Handling Out-of-Control or Unacceptable Data

14.1 Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2 Refer to the SOP for *Non Conformance and Corrective Action* for documentation practices and proper action for *Out of Control* events.

15) Method Performance

15.1 This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2 The method detection limit (MDL) is established using the procedure described in the SOP *Performing and Documenting Method Detection Limit Studies and Establishing*

Limits of Detection and Quantification (CE-QA011/ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the ALS Quality Assurance Manual.

16) Pollution Prevention and Waste Management

16.1 The laboratory will comply with all Federal, State and local regulations governing waste restrictions as specified in the ALS Lab Waste Management Plan.

17) Training

17.1 Training is documented following the *Employee Training and New Employee Orientation* (ADM-TRAIN).

18) Method Modifications

18.1 There are no known modifications in this laboratory standard operating procedure from the reference methods.

19) References

19.1 EPA Method 3500C, SW-846 Update IV. *Organic Extraction and Sample Preparation*, USEPA, 02/2007.

19.2 EPA Method 3510C, SW-846 Update III. *Separatory Funnel Liquid-Liquid Extraction*, USEPA, December, 1996.

19.3 EPA Method 3540C, SW-846 Update III. *Soxhlet Extraction*, USEPA, December, 1996.

19.4 EPA Method 3550C, SW-846 Update IV. *Ultrasonic Extraction*, USEPA, December, 02/2007.

19.5 EPA Method 3580A, SW-846 Update I. *Waste Dilution*, USEPA, July 1992.

19.6 EPA Method 3600C, SW-846 Update III. *Cleanup*, USEPA, December, 1996.

19.7 EPA Method 3611B, SW-846 Update III. Alumina Column Cleanup and Separation of Petroleum Wastes, USEPA, December, 1996.

19.8 EPA Method 3630C, SW-846 Update III. *Silica Gel Cleanup*, USEPA, December, 1996.

19.9 EPA Method 3650B, SW-846 Update III. *Acid-Base Partition Cleanup*, USEPA, December, 1996.

19.10 EPA Method 8000C, SW-846 On-Line. *Gas Chromatography*, USEPA, 03/2003

19.11 EPA Method 8015C, SW-846 Update IV *Nonhalogenated Organics by Gas Chromatography*, USEPA, February, 2007.

19.12 Alaska DEC Method AK 102, Contaminated Sites Program, *Method AK102 for the Determination of Diesel Range Organics*, April 2002.

19.13 Alaska DEC Method AK 103, Underground Storage Tank/LUST Program, *Method for the Determination of Residual Range Organics*, April 2002.

19.14 Alaska DEC Technical Memorandum 06-001, Alaska Department of Environmental Conservation, Division of Spill Prevention and Response, Contaminated Sites Remediation Program, *Biogenic Interference and Silica Gel Cleanup*, May 2006.

19.15 NWTPH-Dx, Semi-Volatile Petroleum Products Method for Soil and

Water, Analytical Methods for Petroleum Hydrocarbons, Publication No. ECY 97-602, June 1997.

19.16 DOD Quality Systems Manual for Environmental Laboratories (QSM), Version 5.1.1, February 2018.

19.17 TNI Standard, Volume, 2009; 2016.

19.18 ISO/IEC 17025: 2017.

20) Changes Since the Last Revision

Summary of Revision Changes			
Revision Number	Effective Date	Document Editor	Description of Changes
17.0	12/2/2020	T Caron	Reformatted SOP to current ALS format. Updated signatories. Minor typographical changes to improve readability and consistency. Section 11: Numerous edits throughout the section including sample extraction, sample analysis. Updated References. Former Table 9 was removed (DQO, surrogate and MS recoveries, RPD for duplicate analysis).

21) Attachments, Tables, and Appendices

21.1 Table 1 - Applicable Method references, Target Analytes, Standard of Quantitation, Suggested Independent Calibration Verification Standards, and Calibration Techniques.

21.2 Table 2 - Method Reporting Limits.

21.3 Table 3 - Library of Fuel Fingerprints.

21.4 Table 4 - Maximum Recommended Analytical holding time and Preservatives.

21.5 Table 5 - Suggested Concentrations of Calibration Standards.

21.6 Table 6 - Suggested GC Operation Conditions.

21.7 Table 7 - Nominal Hydrocarbon Ranges for Petroleum Products.

21.8 Table 8 - Initial and Continuing Calibration Acceptance Criteria.

21.9 Table 9 - Summary of Calibration and QC Criteria with Corrective Actions.

21.10 Appendix A - Example Marker Chromatogram.

TABLE 1
Applicable Method References, Target Analytes, Standard of Quantitation, Suggested Independent Calibration Verification Standards, and Calibration Techniques

Method	Target Analytes	Standard of Quantitation	Suggested ICV	Calibration
EPA Method 8015C	C10-C28 Diesel Range Organics	Diesel #2 composite	Diesel #2	External
EPA Method 8015C	Diesel Range Organics (various ranges) Residual Range Organics (various ranges)	Diesel #2 composite 30 wt. Motor Oil	Diesel #2 30 wt. Motor Oil	External
Alaska DEC AK102	C10-C25 DRO (Diesel Range Organics)	Diesel #2 composite	Diesel #2	External
Alaska DEC AK103	C25-C36 RRO (Residual Range Organics)	30/40wt. Motor Oil Mix	30/40 wt. Motor Oil Mix	External
Northwest TPH-Dx	C12-C25 Diesel Range Organics C25-C36 Residual Range Organics	Diesel #2 composite 30wt. Motor Oil Mix	Diesel #2 30 wt. Motor Oil	External

TABLE 2
Method Reporting Limits

Compound	Aqueous Sample	Aqueous Sample	Solid/Waste Sample	Solid/Waste Sample
	MRL (µg/L)	MDL (µg/L)	MRL(mg/Kg)	MDL (mg/Kg)
8015C TPH-Diesel Range Hydrocarbons	50	15	25	1.6
NW TPH-Diesel Range Hydrocarbons	250	11	25	1.2
NW TPH-Residual Range Hydrocarbons	500	19	100	2.9
AK102 Diesel Range Hydrocarbons	800*	11	20*	1.3
AK103 Residual Range Hydrocarbons	500*	38	100*	2.9

* Required MRLs for Alaska fuels methods.

TABLE 3
Library of Fuel Fingerprints

Class	Product	Class	Product
Fuels and Oils	Unleaded Gasoline	**Fuels and Oils**	Hydraulic Oil (Standard Fluid)
	Jet A		Hydraulic Oil (Tractor Fluid)
	JP 4		Hydraulic Oil Composite
	JP 5		90 wt Gear Oil
	JP 8		Crude Oil
	White Gas	**Petroleum Solvents**	Petroleum Naphtha
	Kerosene		Mineral Spirits
	Diesel #1		Stoddard Solvent
	Diesel #2		410 Thinner
	#4 Fuel Oil		Commercial Paint Thinner
	#5 Fuel Oil		Turpentine
	#6 Fuel Oil (Bunker C)	**Waxes, Greases, Residuals**	Paraffin Wax
	Stove Oil		Vacuum Grease
	Transformer Oil		Petroleum Jelly
	Mineral Oil (Light Grade)		Bearing Grease
	Mineral Oil (Heavy Grade)		Pine Tar
	Machine Oil		Asphalt
	Cutting Oil		Creosote
	Chain Bar Oil		
	30 wt Motor Oil		
	40 wt Motor Oil		
	Multi-viscosity Motor Oils		

TABLE 4
Maximum Recommended Analytical Holding Time and Preservatives

Method	Matrix	Preservative	Extraction Holding Time	Analysis Holding Time
EPA Method 8015C	Aqueous	pH < 2 with HCL; 4°C	7 Days (collection)	40 Days (extraction)
	Solid	4°C	14 Days (collection)	40 Days (extraction)
	Waste	None	28 Days (collection)	40 Days (extraction)
Alaska DEC Method AK 102	Aqueous	pH < 2 with HCL; 4°C	14 Days (collection)	40 Days (extraction)
	Solid	4°C	14 Days (collection)	40 Days (extraction)
Alaska DEC Method AK 103	Solid	4°C	14 Days (collection)	40 Days (extraction)
Alaska DEC Method AK 103	Aqueous	pH < 2 with HCL, 4°C	14 Days (collection)	40 Days (extraction)
NWTPH-Dx	Aqueous	pH < 2 with HCL; 4°C	7 Days (collection) 14 Days (collection) if preserved	40 Days (extraction)
	Solid	4°C	14 Days (collection)	40 Days (extraction)

TABLE 5
Suggested Concentrations of Calibration Standards. Concentrations are in µg/ml (ppm)

Compound	Type	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	ICV
4-Bromofluorobenzene	Surrogate	1	2.5	10	25	100	250	NA	NA	NA
o-Terphenyl	Surrogate	1	2.5	10	25	100	250	NA	NA	NA
Triacontane	Surrogate	1	2.5	10	25	100	250	NA	NA	NA
Gasoline	Target Analyte	20	50	200	500	2000	5000	NA	NA	1000
Kerosene	Target Analyte	20	50	200	500	2000	5000	NA	NA	1000
Diesel #2	Target Analyte	20	50	200	500	2000	5000	20000	50000	1000
30 wt. Motor Oil	Target Analyte	20	50	200	500	2000	5000	NA	NA	1000
30wt./40wt. Motor Oil Mixture	Target Analyte	NA	50	200	500	2000	5000	NA	NA	1000

TABLE 6
Suggested GC Operating Conditions

Event	GC Setting
GC System	
Carrier Gas	Hydrogen
Injection Mode	Splitless
Septum Purge On Time (min)	0.10
Septum Purge Flow (ml/min)	50
Initial Temperature (°C)	60
Initial Hold Time (min)	1.5
Ramp Rate 1 (°C/min)	45
Temp Break 1 (°C)	280
Ramp Rate 2 (°C/min)	25
Temp Break 2 (°C)	315
Ramp Rate 3 (°C/min)	10
Final Temperature (°C)	330
Final Hold Time (min)	7.00
Inlet Temperature (°C)	280
Detector Temperature (°C)	340
Pressure Program	
Constant Flow (✓)	NA
Constant Pressure (✓)	NA
Initial Pressure (psi)	10
Pulse Rate (psi/min):	150
Pulse Maximum (psi)	30
Pulse Maximum Hold Time (min)	0
Pulse Return Rate (psi/min)	150
Pulse Minimum (psi)	3.0
Pressure Hold Time (min)	0.5
Pressure Rate (psi/min)	4.7
Pressure Maximum (psi)	75
Pressure Hold Time (min)	0
Approximate Cycle Time (min)	22

TABLE 7
Nominal Hydrocarbon Ranges for Petroleum Products

Method	Target Analyte	Starting Marker	Closing Marker
Nominal EPA Method 8015C	Diesel	Decane (inclusive)	Octacosane (inclusive)
8015C	Gasoline	Octane (inclusive)	Dodecane (exclusive)
8015C	Kerosene	Nonane (inclusive)	Octadecane (inclusive)
8015C when reported with separate heavy range	Diesel*	Dodecane (inclusive)	Pentacosane (exclusive)
8015C	Residual (or Heavy) Range Organics*	Pentacosane (inclusive)	Hexatriacontane (inclusive)
8015C	Motor Oil	Pentacosane (inclusive)	Hexatriacontane (inclusive)
8015C	#6 Fuel Oil (Bunker C)	Dodecane (inclusive)	Hexatriacontane (inclusive)
Alaska Method AK 102	Diesel Range Organics	Decane (inclusive)	Pentacosane (exclusive)
Alaska Method AK 103	Residual Range Organics	Pentacosane (inclusive)	Hexatriacontane (inclusive)
Washington NWTPH-Dx	Diesel Range Organics	Dodecane (inclusive)	Pentacosane (exclusive)
Washington NWTPH-Dx	Residual Range Organics	Pentacosane (inclusive)	Hexatriacontane (inclusive)

* These ranges may be defined slightly differently based on client requests/requirements

TABLE 8
Acceptance Criteria for Performance Tests

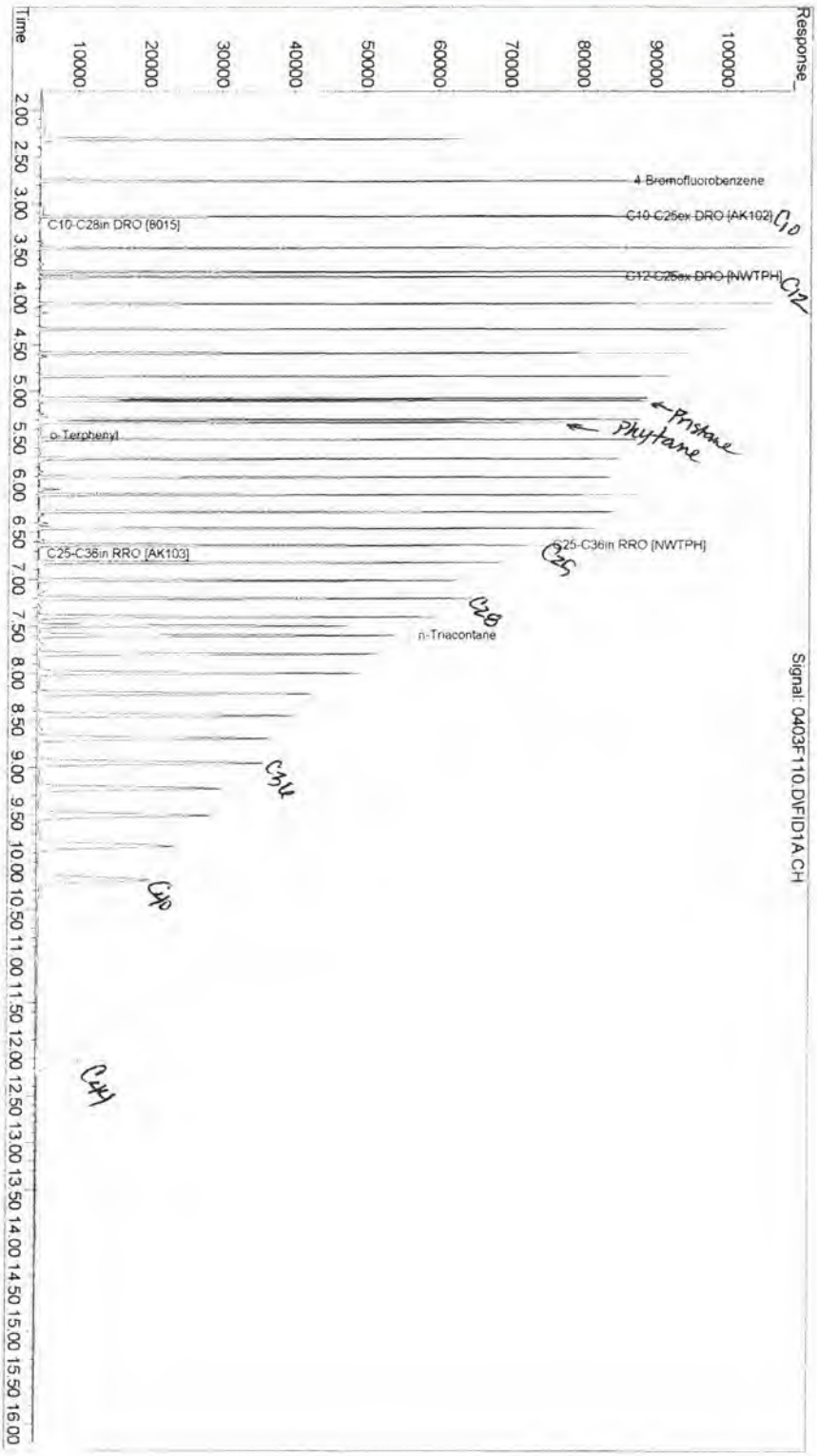
Method	Initial Calibration Criteria	CCV Criteria	Notes
EPA Method 8015C	<20 % RSD (or plot)	± 20 %	None
Alaska DEC Method AK 102	<25 % RSD (or plot)	± 25 %	Surrogate in CCV 60-120%
Alaska DEC Method AK 103	<25 % RSD (or plot)	± 25 %	Surrogate in CCV 60-120%
NWTPH-Dx	<20% RSD (or plot: $r \geq 0.990$)	± 15 %	Method sets criteria on linear regression. All points must be within 15% of true value.

TABLE 9

Summary of Calibration and QC Criteria with Corrective Actions

Method Reference	Control	Specification and Frequency	Acceptance Criteria	Corrective Action
EPA 8015C, NWTPH-Dx, AK102/103	ICAL	Prior to sample analysis	See Table 8	Correct problem then repeat ICAL
EPA 8015C, NWTPH-Dx, AK102/103	ICV	After ICAL	See Table 8	Correct problem and verify second source standard; rerun second source verification. If fails, correct problem and repeat initial calibration.
EPA 8015C, NWTPH-Dx, AK102/103	CCV	Prior to sample analysis	See Table 8	Correct problem then repeat CCV or repeat ICAL
EPA 8015C, NWTPH-Dx, AK102/103	Method Blank	Include with each analysis batch (up to 20 samples)	<MRL (unless project specified)	If target exceeds MRL, reanalyze to determine if instrument was cause. If still noncompliant then: Re-extract or reanalyze samples containing contaminate, unless samples contain > 20x amount in blank.
EPA 8015C, NWTPH-Dx, AK102/103	Laboratory Control Sample	Include with each analysis batch (up to 20 samples)	See Table 9	If exceeds limits, re-extract and re-analyze
EPA 8015C, NWTPH-Dx, AK102/103	Matrix Spike	Include with each analysis batch (up to 20 samples)	See Table 9	Evaluate data to determine if there is a matrix effect or analytical error
EPA 8015C, NWTPH-Dx, AK102/103	MS/Sample Duplicates	Include with each analysis batch (up to 20 samples)	See Table 9	Re-homogenize and re-analyze if result is > 5 X the MRL

Appendix A
Acceptance Criteria for Performance Tests



PCBs as Aroclors

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1) Scope & Applicability

1.1 This procedure is used to determine the concentrations of PCBs as Aroclors using EPA Method 8082A. This procedure is typically applied to water, sediment, and soil matrices but may also be applicable to tissue or various miscellaneous waste samples. Tables 1 and 2 lists the analytes that are determined by this procedure and lists the method reporting limits (MRLs) for each compound in water and soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL). Therefore, MRL=EQL. The reported MRL may be adjusted if required for specific project requirements; however, the capability of achieving other reported MRLs must be demonstrated. MDLs may change as repeat studies are conducted.

1.2 In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPPs supersede method specified requirements. An example of this are projects falling under DoD ELAP. QC requirements defined in the SOP *Department of Defense Projects - Laboratory Practices and Project Management* may supersede the requirements defined in this SOP.

2) Summary of Procedure

2.1 This procedure provides gas chromatographic conditions for the detection of parts-per-billion (ppb) levels of PCBs. The target PCBs are extracted from samples using the appropriate procedure for the sample matrix (see applicable SOP), analyzed, and reported as Aroclors.

2.2 An aliquot of the extract is injected into the gas chromatograph (GC). The compounds are separated on a fused silica capillary column. Compounds of interest are detected by an electron capture detector. Identification of the analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard and by comparison of elution patterns to those of Aroclor standards. Quantitative analysis is performed by using the authentic standard to produce a calibration factor or calibration curve, and using the calibration data to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume.

2.3 The sensitivity of this method usually depends on the level of interferences rather than on instrument limitations. If interferences prevent detection of the analytes, GPC, Florisil cleanup, sulfur cleanup, or concentrated sulfuric acid cleanups are used to eliminate interferences in the analysis. Refer to section 4.2 for cleanup procedure references.

3) Definitions

3.1 For laboratory definitions applicable to most analyses, refer to the SOP for *Sample Batches*.

4) Responsibilities

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

4.2 It is the responsibility of the department supervisor/manager to document analyst training and method proficiency, as described in Method 8082A and the SOP *Employee Training and New Employee Orientation* (ADM-TRAIN).

5) Interferences

5.1 Interferences by phthalate esters can pose a major problem in PCB determinations when using the electron capture detector. These compounds generally appear in the chromatogram as large, late-eluting peaks, especially in the 15% and 50% fractions from the Florisil cleanup. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Phthalate contamination is not usually a problem in our laboratory operation.

5.2 Co-extractables such as lipids, waxes, etc., can be removed via GPC cleanup (SOC-3640A). Certain fractionation cleanups can be used to selectively remove organochlorine pesticides, aiding in Aroclor determination (SOC-3665). The presence of elemental sulfur will result in interferences for most Aroclors. If GPC cleanup is insufficient, cleanup via Method 3660 (SOC-3660) may be used for the removal of sulfur. If interferences from polar compounds is evident, cleanup using Florisil (EXT-FLOR) can be utilized.

5.3 A standard of the DDT analogs must be injected with each initial calibration to determine which of the PCB or Aroclor peaks may be subject to interferences on the analytical columns used. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples.

6) Safety

6.1 Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in SDSs where available. Refer to the ALS Chemical Hygiene Plan and the appropriate SDSs prior to beginning this method.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Containers used to collect samples should be purchased pre-cleaned containers.

7.2 Samples should be tested for residual chlorine at the time of sampling. For aqueous samples with residual chlorine present, add 3-mL 10% sodium thiosulfate solution per gallon (0.008%).

7.3 Water and soil samples must be iced or refrigerated at <6°C from time of collection until extraction. Tissue samples should be stored in accordance with project requirements, typically refrigerated or frozen.

7.4 There are no holding time requirements for this method.

8) Standards, Reagents, and Consumable Materials

8.1 Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP *Reagent/Standards Login and Tracking* (ADM-RTL) for the complete procedure and documentation requirements.

8.2 Solvents: Hexane, acetone, methylene chloride, isooctane, and methanol. Pesticide grade or equivalent. Solvents must be of sufficient purity to permit usage without lessening the accuracy of the determination or introducing interferences.

8.3 Stock Standard Solutions

8.3.1 Stock standard solutions may be purchased from a number of vendors. All reference standards, where possible, must be traceable to SI units or NIST certified reference materials.

8.3.2 Stock standard solutions for each target Aroclor are purchased from AccuStandard at 1000 µg/mL. Other vendors may be used providing they meet the requirements in sec 8.3.1. Unopened stock standard solutions are stored as directed by manufacturer's recommendation. If no storage instructions are provided, the standards are stored at ambient and protected from light. Opened ampoules are stored in crimp-sealed vials at 4 ± 2°C. The expiration date for unopened ampoules is the manufacturer's assigned expiration date. If the manufacturer does not assign a date, an expiration date of 1 year from receipt is assigned. Check opened stock standards frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

8.3.2.1 Intermediate calibration standard solutions are made for the following Aroclor mixtures by diluting 1000 µg/mL stock standards 1:20 in hexane:

- Aroclor 1016/Aroclor 1260
- Aroclor 1221/Aroclor 1254
- Aroclor 1232/Aroclor 1262
- Aroclor 1242/Aroclor 1268
- Aroclor 1248

8.3.2.2 Prepare calibration standards at a minimum of five concentration levels for each Aroclor by dilution of the intermediate standards with hexane. One of the concentration levels should be at or below a concentration representing the method reporting limit (MRL). The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. See Table 4 for preparation and concentrations. A calibration standard of Aroclor 1016/Aroclor 1260 and the surrogates at mid-range concentration is used for the CCV.

8.3.2.3 Calibration standard solutions are stored at 4 ± 2°C and must be replaced after six months, or sooner, if comparison with check standards indicate a problem.

8.3.3 The independent calibration verification (ICV) standards for each target Aroclor are purchased from Ultra Scientific at 100 µg/mL. Other vendors may be used providing they meet the requirements in sec 7.3.1. Unopened stock standard solutions are stored as directed by manufacturer's recommendation. If no storage instructions are provided, the standards are stored at ambient and protected from light. Opened ampoules are stored in crimp-sealed vials at 4°C ± 2°C. The expiration date for unopened ampoules is the manufacturer's assigned expiration date. If the manufacturer does not assign a date, an expiration date of 1 year from receipt is assigned. Check opened stock standards frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

8.3.3.1 Working ICV standards at 1000 µg/L are prepared as described in Table 4. ICV Standards are stored at 4°C ± 2°C for up to six months.

8.3.4 Surrogate solutions are prepared from stock solutions of Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl (DCB) purchased from Ultra Scientific at 200 µg/mL. Other vendors may be used providing they meet the requirements in sec 7.3.1.

8.3.4.1 An intermediate surrogate standard is prepared with the Aroclor 1016/Aroclor 1260 intermediate standard by diluting the 200 µg/mL surrogate stock 1:40 with hexane.

8.3.4.2 A surrogate spiking solution is prepared at 2 µg/mL by making a 1:100 dilution of the surrogate stock standard in acetone. The surrogate solution is stored in the refrigerator for up to six months.

8.3.5 Matrix spike solution: Prepare a spiking solution at 40 µg/mL containing both Aroclors 1016 and 1260 by diluting the 1000 µg/mL stock standards 1:25 with acetone.

9) Apparatus and Equipment

9.1 Gas Chromatograph (GC)

9.1.1 Analytical system complete with gas chromatograph suitable for splitless or split automated injection into a wide bore capillary column with an electron capture detector (ECD). Use of Large Volume Injection (LVI) is optional. Helium is used as the carrier gas; argon/methane mixture is used for the detector makeup gas (auxiliary gas).

9.1.2 GC Autosampler: The GC system should be configured with a compatible autosampler for automated injection of standards, samples, and QC samples.

9.1.3 GC Columns - fused silica capillary columns

Column 1: DB-35MS 30 m x 0.32 mm ID, 0.25 µm df or equivalent*
Column 2: DB-XLB 30 m x 0.32 mm ID, 0.50 µm df or equivalent*

Note: Column diameter and film thickness may vary depending on instrument. Refer to the instrument maintenance logbook for the columns used for a specific instrument configuration.

9.2 Data System - A computer data system must be interfaced to the GC/ECD. The system must allow the continuous acquisition and storage on machine-readable media of all chromatographic data obtained throughout the duration of the chromatographic program. The computer must have software that includes automated calibration, identification, and quantitation routines. The software must also be capable of integrating the chromatographic peaks abundances.

10) Preventative Maintenance

10.1 All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. The entry in the log must include: date of event, the initials of who performed the work, and a reference to analytical control.

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

10.3 Gas Chromatograph

10.3.1 Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, o-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chromatographic performance. In some cases liners and seals may be cleaned and re-used. Additionally, disassembly and sonication of the septum-less head may be performed if indicated.

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

10.3.3 Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

11) Procedure

11.1 Sample Preparation

11.1.1 Water samples (1L or less) are extracted at a pH of 5-9 with methylene chloride, using either continuous Liquid-Liquid extraction (EPA Method 3510C or 3520C); with hexane by microextraction (SW-846 Method 3511). Refer to the applicable extraction SOP.

Note: Project-specific or regulation-specific extraction methods may apply. For projects originating from South Carolina and under the SC DHEC lab certification, use the EPA Method 3520C extraction method only.

11.1.2 Soil/sediment samples are extracted using either a automated Soxhlet extraction, (EPA Method 3541, EXT-3541) or by microwave extraction (SW-846 Method 3546, EXT-3546). Refer to the applicable extraction SOP.

11.1.3 Waste samples (organic liquids) may be prepared using the waste dilution method (EPA Method 3580A, EXT-3580). Refer to the extraction SOP for specific parameters.

11.1.4 Additional sample cleanup procedures may be employed as appropriate for the samples. Refer to the section on interferences and the appropriate ALS SOP.

11.2 Calibration

Note: The calibration procedure(s) and options chosen must follow the ALS protocols. Any exceptions to the calibration procedures detailed in *Calibration of Instruments for Organics Chromatographic Analyses* (SOC-CAL) are described as follows:

Note: Certain state or program protocols have specific procedures for calibration. The analyst must ensure that the correct procedures are used. Known exceptions are as follows:

- The use of quadratic regression calibration is not allowed for projects (samples) originating from South Carolina and under the SC DHEC lab certification.

Note: DOD QSM requires the quantitation for Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point.

11.2.1 For each target Aroclor, select three to six characteristic peaks to use for quantitation. Choose peaks that are at least 25% of the height of the largest Aroclor peak and do not coelute with any of the DDT analogs. For each Aroclor, select at least one quantitation peak that is unique to that Aroclor. Use at least five peaks for the Aroclor 1016 and Aroclor 1260 mixture, none of which should be found in both of these Aroclors. Establish the retention time window position using the mid-point of the ICAL range before processing the calibration curve.

11.2.2 Calibrate the system immediately prior to conducting any analyses. Refer to Table 3 for instrument conditions. Starting with the standard of lowest concentration, analyze each calibration standard and tabulate response (peak area) versus the concentration in the standard. Calculate the ratio of the response to the amount injected the (calibration factor) for each analyte at each standard concentration. The Relative Standard Deviation (RSD) must be less than 20% when average response factor is used.

11.2.3 The calibration of each Aroclor is verified by an independent source. Prepare an independent calibration verification standard (ICV) by dilution of a stock solution purchased from a different vendor and analyze immediately after each initial calibration. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. Evaluate the ICV as described in *Calibration of Instruments for Organics Chromatographic Analyses* (SOC-CAL).

11.3 Calibration Verification

11.3.1 The working calibration curve or calibration factor must be verified on each analytical sequence by the analysis of one or more mid-range calibration standards (CCV) containing Aroclors 1016/1260 and the surrogates. A standard (CCV) must be injected at the start of each sequence and after each set of sample extracts (every 10 samples or every 12 hours, whichever is first) in the analysis sequence. Evaluate the CCV as described in SOC-CAL. The %D for Aroclor 1016 is a control for Aroclors 1016, 1221, 1232, 1242, and 1248. The %D for Aroclor 1260 controls for Aroclors 1254, 1260, 1262, and 1268.

Note: DOD projects require a CCV analysis every 10 field samples.

11.4 Retention Time Windows

11.4.1 Pattern recognition/matching and retention times are used for the identification of PCBs as Aroclors.

11.4.2 Establish retention time windows for the peaks used for quantitation with the GC system in acceptable operating condition. Make three injections of all analytes throughout the course of a 72 hour period. Serial injections over less than a 72 hour period may result in retention time windows that are too tight. Using retention times from these analyses, calculate retention time windows. Refer to

EPA Method 8000C for detailed instructions.

11.4.3 Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. In those cases where the standard deviation for a particular standard is zero, the laboratory may use a default window of ± 0.03 minutes. If the peak width is > 0.06 minutes, use a default window of 0.1 minutes.

11.4.4 Calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. Retain this data in the method file.

11.5 Gas Chromatography

11.5.1 Set up an analytical sequence for the standards and samples to be analyzed. Calibrate the system as described in Section 11.2. Refer to Table 3 for typical instrument operating conditions. The same conditions must be used for samples as for calibration and QC analyses. Ensure that the instrument configuration is correct and that any necessary maintenance has been performed. Figure 1 shows a typical analysis sequence.

11.5.2 Evaluate the CCVs as indicated in Section 11.3. Use the standards interspersed throughout the sample analysis sequence to evaluate the qualitative performance of the GC system including positioning of the retention time window. If any retention time shift which would impede analyte identification is evident (as shown by Aroclor pattern irregularities or the surrogate falling outside of the retention time window), evaluate the chromatogram for possible causes such as carryover from a highly contaminated sample. If a problem related to GC system has been determined to be the cause of retention time shift, perform whatever maintenance is necessary before reanalyzing the CCV or recalibrating and proceeding with sample analysis. All samples that were injected after the sample exceeded the criteria must be reinjected if initial analysis indicated the presence of any analytes of interest.

FIGURE 1
Analysis Sequence

2,4'/4,4'-DDx Retention Time Marker	
Initial Calibration Blank	ICB
Standards 1-6	1016/1260/TCMX/DCB ICAL Standards
Standards 7-12	1221/1254 ICAL Standards
Standards 13-18	1232/1262 ICAL Standards
Standards 19-24	1242/1268 ICAL Standards
Standards 25-30	1248 ICAL Standards
Standards 31-39	ICVs for 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, 1268
Continuing Calibration Verification	CCV
CCB	
Method Blank	
Laboratory Control Sample	LCS
Sample 1 - 8	
Matrix Spike	
Duplicate Matrix Spike	
Continuing Calibration Verification	CCV

Continuing Calibration Blank	CCB
Sample 9 - 18	
Continuing Calibration Verification	CCV
Continuing Calibration Blank	CCB

Note: For DOD projects, the CCB must be analyzed following the CCV. Instrument blanks and CCBs may not be analyzed prior to QC samples or standards.

11.6. Troubleshooting

11.6.1. Initial calibration – If the initial calibration fails to meet the criteria, or the ICV fails (indicating a calibration problem), the following steps may be taken, depending on nature of the problem.

- Recheck the information entered into the software used for calibration and quantitation. Verify the standard values are correct and data files are correct. If incorrect, repeat the calibration with the correct information.
- Recheck standards preparation to ensure that standards are correct. Re-prepare and reanalyze if needed.
- Ensure that proper preventive maintenance was performed. Repeat the preventive maintenance if necessary and reanalyze the calibration.
- If calibration problems persist or more substantial calibration problems exist, corrective maintenance or repair may be needed. This includes such measures as column changes, detector maintenance, or GC repair. This will depend on the nature of the problem. Following any such maintenance, repeat the calibration.

11.6.2. Continuing calibration – If the CCV analysis fails to meet the criteria, the following steps may be taken, depending on nature of the problem.

- Recheck the information entered into the software used for calibration and quantitation. Verify the standard values are correct and data files are correct. If incorrect, repeat the calibration with the correct information.
- Recheck standards preparation to ensure that standards are correct and that the correct standard is used as the CCV. Re-prepare and reanalyze if needed. Note that NELAC and DOD requirements apply when multiple CCVs are analyzed.
- Ensure that proper preventive maintenance was performed. Repeat the preventive maintenance if necessary and reanalyze the CCV.
- If calibration problems persist or more substantial calibration problems exist, corrective maintenance or repair may be needed. This includes such measures as column changes, detector maintenance, or GC repair. This will depend on the nature of the problem. Following any such maintenance, repeat analysis of the CCV and necessary samples. Major maintenance will require recalibration. Note that some samples may quickly deteriorate the system to the point that closing CCVs will not pass. This should be verified through a second run of the samples and documented.

12) QA/QC Requirements

12.1 Initial Precision and Recovery Validation

12.1.1 The precision of the extraction procedure and the GC procedure must be validated before analysis of samples begins, or whenever significant changes to

the procedures have been made. To do this, four reagent water samples are spiked at a level near the midpoint of the calibration range (typically the LCS level), then extracting and proceeding with Section 11. The spiking solution may be prepared from pure standard materials, or purchased as certified solutions. If prepared by the laboratory, stock standards prepared independently from those used for calibration should be used. The concentration of the analytes to be spiked is 20x the MRL.

12.2 Method Detection Limits and Method Reporting Limits

12.2.1 A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike seven blank matrix (water or soil) samples with MDL spiking solution at a level below the MRL. Follow the analysis procedures in Section 11 to analyze the samples.

12.2.2 Calculate the average concentration found (x) in µg/mL, and the standard deviation of the concentrations (s) in µg/mL for each analyte. Calculate the MDL for each analyte. Refer to the SOP *Performing and Documenting Method Detection Limit Studies and Establishing Limits of Detection and Limits of Quantitation* (CE-QA011/ADM-MDL).

12.2.3 Limits of Quantification (LOQ)

12.2.3.1 The laboratory establishes a MRL/LOQ for each analyte as the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements. Analysis of a standard or extract prepared at the lowest point calibration standard provides confirmation of the established sensitivity of the method. The LOQ recoveries should be within 50-150% of the true values to verify the data reporting limit. Refer to the SOP *Performing and Documenting Method Detection Limit Studies and Establishing Limits of Detection and Limits of Quantitation* (CE-QA011/ADM-MDL).

12.2.4 The Method Reporting Limits (MRLs) used at ALS Kelso are the routinely reported lower limits of quantitation which take into account day-to-day fluctuations in instrument sensitivity as well as other factors. These MRLs are the levels to which ALS Kelso routinely reports results in order to minimize false positive or false negative results. The MRL is normally two to ten times the method detection limit.

12.3 Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project specific quality assurance plans (QAPP). General QA requirements for DoD QSM are defined in the laboratory SOP *Department of Defense Projects – Laboratory Practices and Project Management* (ADM-DOD) or *Department of Defense Projects – Laboratory Practices and Project Management – QSM 5.0* (ADM-DOD5). General QC Samples are:

12.3.1 Method Blank

12.3.1.1 A method blank is extracted and analyzed with every batch of 20 (or fewer) samples to demonstrate that there are no method interferences. If the method blank shows any hits above the reporting limit, corrective action must be taken. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and reanalysis. For some project

specific needs, exceptions may be noted and method blank results above the MRL may be reported for common lab contaminants (phthalate esters, etc.).

Note: DoD projects require that no analyte be detected > ½ the RL or 1/10 the regulatory limit, whichever is greater.

12.3.2 Laboratory control Sample

12.3.2.1 A lab control sample (LCS) must be extracted and analyzed with every batch of 20 samples. The water LCS is prepared by adding 50 µL of the matrix spike solution to 1L of reagent water, resulting in a concentration of 2.0 µg/L. The soil LCS is prepared by adding 100 µL of spike solution to 20 g of sand, resulting in a concentration of 200 µg/kg. For project-specific low-level extractions, spiking amounts can be adjusted accordingly. Calculate percent recovery (%R) as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

12.3.2.2 Current ALS QC acceptance criteria for lab control samples are listed in the current laboratory DQO tables. Project-specific or program-specific acceptance criteria may supersede ALS criteria. For example, for samples requiring South Carolina DHEC certification the acceptance criteria are 70-130 % recovery. If the lab control sample (LCS) fails acceptance limits for any of the compounds, corrective action must be taken. Corrective action includes recalculation, reanalysis, or re-extraction and reanalysis. Refer to the ALS-Kelso Quality Assurance Manual for guidance in evaluating recoveries that exceed LCS limits.

12.3.3 Matrix Spike

12.3.3.1 A matrix spike (MS) and duplicate matrix spike (DMS) must be prepared and analyzed with every batch of 20 (or fewer) samples. The MS/DMS is prepared by adding the same volume of the matrix spike solution to the sample as listed for the LCS, then proceeding the entire extraction and analysis as specified in Section 11. Calculate percent recovery (%R) as:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

12.3.3.2 Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{|R1 - R2|}{(R1 + R2)/2} \times 100$$

Where R1= % recovery of the MS
R2= % recovery of the DMS

12.3.3.3 Current QC acceptance criteria for MS/DMS are the same as the LCS as listed in the lab DQO tables. Project-specific acceptance criteria may supersede lab criteria. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken. Corrective action includes recalculation, reanalysis, or re-extraction and reanalysis.

12.3.3.4 Following analysis of the MS the percent recovery is calculated and compared to acceptance limits. If the recovery is within control limits the results may be reported. If not, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. It is verified that the spike level is at least five times the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as performing any additional cleanups, dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

12.3.4 Surrogate

12.3.4.1 Surrogate spike is added to every sample, blank and spike prior to extraction. Two surrogate standards (Tetrachloro-m-xylene and Decachlorobiphenyl) are added to each sample. For water, 100 µL of the surrogate spike is added to 1 L, resulting in 0.2 µg/L. For soil, 200 µL of the surrogate spike is added to 20 g, resulting in 20 µg/Kg. Calculate surrogate percent recovery (%R) as:

12.3.4.2 Calculate surrogate percent recovery (%R) as:

$$\%R = S/V \times 100$$

Where S = Amount of surrogate recovered
V = Amount spiked

12.3.4.3 Current QC acceptance criteria for surrogates are listed in the current laboratory DQO tables. Project specific acceptance criteria may supersede lab criteria. Both surrogate recoveries must be within the acceptance limits. If either (or both) surrogate is outside of acceptance limits for reasons other than matrix interferences, corrective action must be taken. Corrective actions include recalculation, reanalysis, or re-extraction and reanalysis. The acceptance criteria listed are current criteria, but are subject to change as control limits are updated.

12.3.5 QC results should be reviewed periodically for trends in results. Refer to the SOP *Trending, Control Limits, and Uncertainty* (ADM-TREND) for guidance. Control limits for QC analyses may be determined using the control charts or similar mechanism on an annual basis.

13) Data Reduction and Reporting

13.1 Identification of PCBs as Aroclors

13.1.1 To identify Aroclors, compare the chromatographic pattern of the sample to known Aroclor standards. Tentative identification of PCBs as Aroclors is made when the pattern of peaks in the sample chromatogram matches the pattern of

peaks in the Aroclor standard itself. There also needs to be agreement between the retention times and response ratios of the 3-6 selected quantitation peaks in the sample chromatogram and the Aroclor standard.

13.1.2 Tentative identification of analytes must be confirmed using a second GC column of dissimilar phase. Identify the Aroclor by comparing the chromatographic pattern of the sample to known Aroclor standards analyzed on the same column. Confirmation of the Aroclor is made when the sample chromatogram matches the pattern of peaks in the Aroclor being confirmed. Quantitation results for the 2 columns must agree (≤40% RPD) to confirm the identification. If interferences or other sample anomalies make the RPD value >40% but the analyst makes a positive identification, the basis of the identification must be documented and the data user notified of the discrepancy (see section 13.2).

13.2 Sample matrix difficulties

13.2.1 Weathering of PCBs in the environment and changes resulting from waste treatment processes may alter the pattern of a specific Aroclor so it does not closely match an Aroclor standard. The earlier eluting peaks will often diminish in comparison to the later eluting peaks. If this is observed, alternate peaks may be selected to aid identification to reduce quantitation bias.

13.2.2 Metabolism by organisms may also alter the pattern since individual PCB congeners are metabolized at different rates. When working with tissue samples, the 40% RPD criteria for confirmation may not be met.

13.2.3 Samples may also include mixtures of two or more Aroclors. To the extent possible, identify and quantify each Aroclor.

13.2.4 High amounts of organochlorine pesticides in the sample may interfere with identification. If this is observed, alternate peaks may be selected to aid identification and to reduce quantitation bias.

13.2.5 For all of these reasons a high level of analyst expertise is required to interpret complex chromatograms.

13.3 Quantitation of PCBs as Aroclors:

13.3.1 The quantitation of PCBs as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard or standards. All calibration acceptance criteria as described in section 11 must be met before reporting any results. Sample results should then be reported according to *Confirmation Procedures for GC and HPLC Analyses* (SOC-CONF). Results may be reported from either column if all calibration acceptance criteria as described in section 11 are met.

13.3.2 Once the Aroclor pattern has been identified, compare the responses of 3 to 6 major peaks in the calibration standard of that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each of the 3 to 6 peaks and the calibration model selected in section 11. The concentration is determined using the 3 to 6 characteristic peaks and then the concentrations are averaged to determine the concentration of the Aroclor. If there are interfering peaks with the 3 to 6 quantitation peaks that cause Aroclor average to be falsely overstated, then that interference peak is Q-deleted using the data system and the average is recalculated so that the average more truly represents the concentration in the sample. This often occurs when there are more than one Aroclor in a sample extract or if pesticides are

present. Quantitation of mixed Aroclors will require the selection of peaks that are not shared in common by both Aroclors.

13.3.3 For samples with severe matrix interferences, the quantitation may be performed by measuring the total area of the PCB pattern and quantifying on the basis of the Aroclor standard that is most similar to the sample. Any peaks that are not identifiable as PCBs should be subtracted from the total area. When the quantitation option is used, the sample problems should be described for the data user and quantification procedure documented.

13.3.4 Using the data system, calculate the concentration in the extract using the calibration model chosen for calibration from *Calibration of Instruments for Organics Chromatographic Analyses* (SOC-CAL).

13.3.4.1 Using the data system, calculate the concentration of each analyte in the sample extract (Cex) in µg/ml units using the calibration factor or calibration curve (Section 11). The sample concentration computed using the following equations:

Aqueous Samples: $$Concentration\ (\mu g/L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$$

Where	Cex	=	Concentration in extract in µg/mL
	Vf	=	Final volume of extract in mL
	D	=	Dilution factor
	Vs	=	Volume of sample extracted, liters

Non-aqueous Samples: $$Concentration\ (mg/Kg) = \frac{(Cex)\ (Vf)\ (D)\ x\ 1{,}000}{(W)\ x\ 1{,}000}$$

Where	Cex	=	Concentration in extract in µg/mL
	Vf	=	Final volume of extract in mL
	D	=	Dilution factor
	W	=	Weight of sample extracted in grams.

13.4 Sample concentrations are reported when all QC criteria for the analysis have been met or the results are qualified with a footnote.

13.5 Data Review

13.5.1 Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP *Laboratory Data Review Process* (ADM-DREV) for details. The person responsible for final review of the data report and/or data package should assess the overall validity and quality of the results and provide any appropriate comments and information to the Project Manager to inclusion in the report narrative.

13.6 Reporting

13.6.1 Reports are generated using the STEALTH Data Reporting System which compiles the SMO login information. This compilation is then transferred to a file, which STEALTH uses to generate a report. The forms generated may be ALS standard

reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.6.2 Sample concentrations are reported when all QC criteria for the analysis have been met or the results are qualified with an appropriate footnote. For Arizona projects the appropriate Arizona qualifier must be used.

14) Contingencies for Handling Out-of-Control or Unacceptable Data

14.1 Refer to the SOP for *Non Conformance and Corrective Action* (CE-QA008/ADM-NCAR) for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

15) Method Performance

15.1 This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available. The method detection limit (MDL) is established using the procedure described in the SOP *Performing and Documenting Method Detection Limit Studies and Establishing Limits of Detection and Quantification*). Method Reporting Limits are established for this method based on the low calibration point and the MDL study results.

16) Pollution Prevention and Waste Management

16.1 The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Lab Waste Management Plan.

17) Training

17.1 All analysts performing this analysis are required to read and understand this SOP.

17.2 Training is documented following the *Employee Training and New Employee Orientation* (ADM-TRAIN).

18) Method Modifications

18.1 There are no known modifications in this laboratory standard operating procedure from the reference method.

19) References

19.1 Polychlorinated Biphenyls (PCBs) as Aroclors, Method 8082A, Revision 1, February 2007, EPA Test Methods for Evaluating Solid Waste, SW-846, Update IV

19.2 Determinative Chromatographic Separations, EPA SW846, Test Methods For Evaluating Solid Waste, On-Line, Method 8000C, Revision 3, March 2003.

19.3 DoD Quality Systems Manual for Environmental Laboratories, current version .

19.4 8000C Method criteria, Arizona DHS, 2/13/2007. Available online at https://www.azdhs.gov/documents/preparedness/state-laboratory/lab-licensure-certification/technical-resources/additional-resources/method-criteria-8000.pdf.

19.5 TNI Quality Standards, 2009; 2016.

19.6 ISO/IEC 17025:2005/2017 American National Standard, General requirements for the competence of testing and calibration laboratories.

20) Changes Since the Last Revision

Summary of Revision Changes			
Revision Number	Effective Date	Document Editor	Description of Changes
19.0	12/2/20	T Caron	Updated Signatories Minor grammatical, typographical, and formatting corrections.

21) Attachments, Tables, and Appendices

21.1 Table 1 - Target Analytes and Method Reporting Limits* - Water.

21.2 Table 2 - Target Analytes and Method Reporting Limits* - Soil/Sediment.

21.3 Table 3 - Gas Chromatograph Operating Conditions*.

21.4 Table 4 - Calibration Standard Preparation.

21.5 Table 5 - Summary of Corrective Actions.

TABLE 1
Target Analytes and Method Reporting Limits* - Water

Analyte	Standard Level (µg/L)	Low-Level (µg/L)	Ultra Low-Level (µg/L)
Aroclor 1016	0.20	0.020	0.0050
Aroclor 1221	0.40	0.040	0.010
Aroclor 1232	0.20	0.020	0.0050
Aroclor 1242	0.20	0.020	0.0050
Aroclor 1248	0.20	0.020	0.0050
Aroclor 1254	0.20	0.020	0.0050
Aroclor 1260	0.20	0.020	0.0050
Aroclor 1262	0.20	0.020	0.0050
Aroclor 1268	0.20	0.020	0.0050

TABLE 2
Target Analytes and Method Reporting Limits* - Soil/Sediment

Analyte	Standard Level (mg/Kg)	Low-Level (µg/Kg)
Aroclor 1016	0.10	10
Aroclor 1221	0.20	20
Aroclor 1232	0.10	10
Aroclor 1242	0.10	10
Aroclor 1248	0.10	10
Aroclor 1254	0.10	10
Aroclor 1260	0.10	10
Aroclor 1262	0.10	10
Aroclor 1268	0.10	10

*For some analytes, LOQs may vary slightly from MRLs due to program/project requirements.

TABLE 3
Gas Chromatograph Operating Conditions*

Gas Chromatograph:	Agilent 6890 or equivalent with ECD
Injection Port Temperature:	Initial Temp 90°C for 0.5 min., 250°C/min ramp to 325°C for 5.0 min., 20°C/min ramp to 250°C for 5.0 min.
Oven Temperature Program:	90°C hold for 0.5min., 5°C/min ramp to 230°C for 0.5 min., 7°C/min ramp to 315°C, hold for 0.06 min.
Detector Temperature:	325°C
Injection Volume:	1 µL
Column 1:	DB-35MS, 30 m x 0.32 mm ID x 0.25 µm film thickness or equivalent.
Column 2:	DB-XLB, 30 m x 0.32 mm ID x 0.50 µm film thickness or equivalent
Carrier Gas:	Hydrogen
Auxiliary Gas:	Nitrogen
Data System:	HP EnviroQuant (acquisition), Target (data processing)

* The above instrument temperatures may be modified depending on the instrument used. The GC column diameter and phase thickness depend on the instrument used. All conditions must be the same for initial calibration, continuing calibration, sample, and QC analysis.

TABLE 4
Calibration Standard Preparation

1016/1260 Initial Calibration Standards (prepared in hexane)

Aroclor 1260 50 µg/mL Intermediate	Surrogate 5 µg/mL Intermediate	Final Volume	Final Concentration Aroclors	Final Concentration Surrogates
40 µL	40 µL	10 mL	200 µg/L	20 µg/L
20 µL	20 µL	10 mL	100 µg/L	10 µg/L
10 µL	10 µL	10 mL	50 µg/L	5 µg/L
	Aroclor1016-1260/Surg (20/200 ppb)			
1000 µL	1000 µL	10 mL	20 µg/L*	2 µg/L*
500 µL	500 µL	10 mL	10 µg/L	1 µg/L
250 µL	250 µL	10 mL	5 µg/L	0.5 µg/L
100 µL	100 µL	10 mL	2 µg/L	0.2 µg/L
50 µL	50 µL	10 mL	1 µg/L	0.1 µg/L

* CCV Standard

Single-Point Calibration Standards (1,000 µl/mL)

Aliquot	Final Volume	Solvent	Final Concentration
10 µL	10 mL	Hexane	1000 µg/L
10 µL	10 mL	↓	1000 µg/L
10 µL	10 mL	↓	1000 µg/L
10 µL	10 mL	↓	1000 µg/L
10 µL	10 mL	↓	1000 µg/L
10 µL	10 mL	↓	1000 µg/L
10 µL	10 mL	↓	1000 µg/L

ICV Standards (100 µl/mL)

Aliquot	Final Volume	Solvent	Final Concentration
100 µL	10 mL	Hexane	1000 µg/L
100 µL	10 mL	↓	1000 µg/L
100 µL	10 mL	↓	1000 µg/L
100 µL	10 mL	↓	1000 µg/L
100 µL	10 mL	↓	1000 µg/L
100 µL	10 mL	↓	1000 µg/L
100 µL	10 mL	↓	1000 µg/L
100 µL	10 mL	↓	1000 µg/L
100 µL	10 mL	↓	1000 µg/L

*As needed for projects requiring non-routine additional compounds, similar dilutions are prepared to obtain calibration standards for these compounds.

TABLE 5

Summary of Corrective Actions

Method Reference	Control	Specification and Frequency	Acceptance Criteria	Corrective Action
EPA 8000C EPA 8082A	ICAL	Prior to sample analysis	% RSD ≤ 20 R^2 COD ≥ 0.990	Correct problem then repeat ICAL
EPA 8000C EPA 8082A	ICV	After ICAL	± 20% Diff	Correct problem and verify second source standard; rerun second source verification. If fails, correct problem and repeat initial calibration.
EPA 8000C EPA 8082A	CCV	Prior to sample analysis, every 10 samples or 12 hours	± 20% Diff	Correct problem then repeat CCV or repeat ICAL
EPA 8000C EPA 8082A	Method Blank	Include with each analysis batch (up to 20 samples)	<MRL	If target exceeds MRL, reanalyze to determine if instrument was cause. If still noncompliant then: Re-extract or reanalyze samples containing contaminate, unless samples contain > 20x amount in blank.
EPA 8000C EPA 8082A	Laboratory Control Sample	Include with each analysis batch (up to 20 samples)	See DQO	If exceeds limits, re-extract and re-analyze
EPA 8000C EPA 8082A	Matrix Spike	Include with each analysis batch (up to 20 samples)	See DQO	Evaluate data to determine if the there is a matrix effect or analytical error
EPA 8000C EPA 8082A	Matrix Spike Duplicates	Include with each analysis batch (up to 20 samples)	See DQO	Evaluate data to determine if the there is a matrix effect or analytical error

Semi-Volatile Organic Compounds by GC/MS Low Level Procedure

DOCUMENT ID: SVM-8270L, REV. 11.0

Approved By: _____ Date: 12/2/2020
Inorganics Manager, Jonathon Walter

Approved By: Kurt Clarkson Date: 12/2/2020
Quality Assurance Manager, Kurt Clarkson

Approved By: _____ Date: 12/2/20
Laboratory Director, Charles(Pat) Byrne

1) Scope & Applicability

1.1. This procedure is used to determine low level concentrations of Semi-Volatile Organic Compounds in water and soil using EPA Method 8270D. This procedure may also be applicable to various miscellaneous waste samples. Table 1 indicates compounds that may be determined by this method and lists their method reporting limits (MRLs) in water and soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) or Low Level of Quantitation (LLOQ). The reported MRL may be adjusted if required for specific project requirements; however, the capability of achieving other reported MRLs must be demonstrated. The laboratory DQO Table lists Method Detection Limits (MDLs) that have been achieved. MDLs may change as studies are updated or new studies performed, and will vary depending on the preparation method.

1.2. This procedure can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone phase. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. Other compounds than those listed in Tables 4 and 6 may be analyzed. Refer to Section 1 of method 8270D.

1.3. In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DoD ELAP. QC requirements defined in the SOP *Department of Defense Projects - Laboratory Practices and Project Management - QSM 5.X* (ADM-DOD5) may supersede the requirements defined in this SOP.

2) Summary of Procedure

2.1 This method provides Gas Chromatography/Mass Spectrometry (GC/MS) conditions for the detection of Semi-volatile Organic Compounds. Prior to the use of this method, an appropriate sample preparation method must be used to recover the analytes of interest. A 1 µL aliquot of the extract is injected into the gas chromatograph (GC) using a Agilent Multi-Mode Injector (MMI). The compounds are separated on a fused silica capillary column. Compounds of interest are detected by a mass selective detector. Identification of the analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard, and by comparing mass spectra of analytes with mass spectra of reference materials. Quantitative analysis is performed by using the authentic standard to produce a response factor and calibration curve, and using the calibration data to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume.

2.2 The following compounds may require special treatment when being determined by this method. Hexachlorocyclopentadiene is subject to decomposition in the injection port of the gas chromatograph, to a chemical reaction in acetone, and can undergo photochemical decomposition. N-Nitroso-dimethylamine is difficult to separate from the solvent under the chromatographic conditions described. N-Nitroso-diphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Benzoic acid, pentachlorophenol, 2,4-dinitrophenol, 2-nitroaniline, 3-

nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

3) Definitions

3.1 For laboratory definitions applicable to most analyses, refer to the SOP for *Sample Batches*.

4) Responsibilities

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

4.2 It is the responsibility of the department supervisor/manager to document analyst training .and method proficiency, as described in: *Employee Training and Orientation* (ADM-TRAIN).

5) Interferences

5.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation of the samples. Corrective action should be taken to eliminate the interferences.

5.2 Accurate determination of phthalate esters can pose difficulties when using this methodology. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware may occur when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalates can best be minimized by avoiding contact with any plastic materials. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

5.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of a solvent blank to check for cross contamination.

6) Safety

6.1 Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in SDSs where available. Refer to the ALS Environmental, Health and Safety Manual and the appropriate SDS prior to beginning this method.

6.2 This method uses Dichloromethane, a known human carcinogen. Refer to the methylene chloride policy document, ENV-HSE-NA-EX-006-EN for proper handling.

G:\SAFETY\TRAINING\Methylene Chloride\Methylene Chloride - NA 031419.pdf.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Certified clean containers should be purchased for sample collection. The sample containers should be of glass or Teflon and have screw-top covers with Teflon liners. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

7.2 Water and soil samples should be iced or refrigerated at <6 C from time of collection until extraction.

7.3 Water samples must be extracted within 7 days and the extracts analyzed within 40 days following extraction. Soil samples must be extracted within 14 days and the extract analyzed within 40 days following extraction. Extracts are stored at ≤10°C.

8) Standards, Reagents, and Consumable Materials

8.1 Solvents: Acetone, methylene chloride, methanol, and other appropriate solvents.

8.2 Stock Standard Solutions (See Table 2)

8.2.1 Commercially prepared stock standards are typically used when available at a concentration of 1000 µg/mL or more. They must be obtained from an A2LA or ISO9000 certified vendor. Standard concentrations can be verified by comparison versus an independently prepared standard. Alternatively, prepare stock standard solutions at a concentration of 1000 µg/ml by dissolving 0.0100 g of reference material in methylene chloride or other suitable solvent and diluting to volume in a 10 mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Store according to vendors recommendations.

8.2.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at ≤10°C and protect from light. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

8.2.3 Stock standard solutions must be replaced after one year, or sooner, if comparison with check standards or samples indicates a problem.

8.3 Internal Standard Solutions (See Table 4) - The internal standards are 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} (See Table 4 for corresponding compounds). The nominal concentration of the standard is 100 ng/µL. Each 1 mL of sample extract undergoing analysis should be spiked with 10 µL of the internal standard solution, resulting in a concentration of 1.0 ng/µL of each internal standard. Store at -10°C or less when not being used. When using premixed certified solutions, store according to the vendors recommendations.

8.4 GC/MS Tuning Standard - For a 20 µL injection, use a methylene chloride solution containing 2.5 ng/µL of decafluorotriphenylphosphine (DFTPP). This will result in 50 ng on column injection of DFTPP. The standard should also contain 2.5 ng/µL of benzidine, DDT, and pentachlorophenol, to verify injection port inertness and GC column performance. Store at -10°C or less when not being used, or store according to the manufacturer's recommendations. For smaller injection volumes, use a DFTPP standard with a concentration that will result in a 10-50 ng on column injection.

8.5 Calibration Standards (See Table 2)

8.5.1 A minimum of five initial calibration standards should be prepared from stock solutions. One of the calibration standards should be at a concentration at or below the method reporting limit; the others should correspond to the range of concentrations found in real samples, but should not exceed the working range of the GC/MS system. At least one calibration standard must be at a concentration corresponding to a sample concentration meeting project-specific data quality objectives. Each standard should contain each analyte for detection by this method. Each 1 ml aliquot of calibration standards should be spiked with 10 µL of the internal standard solution prior to analysis. All calibration standards should be stored at -10°C or less and should be freshly prepared once a year, or sooner if check standards indicate a problem.

8.5.2 The daily calibration standard (CCV) is prepared from stock solutions at a concentration at the midpoint of the calibration curve (typically 2-4 ng/µL). The CCV is prepared weekly and can be stored at 4 ± 2°C, or as recommended by the manufacturer. The DFTPP standard may be combined with this standard (maintaining a 1-50 ng on column injection) providing tuning verification and calibration verification can be done without interferences.

8.6 QC Standards (See Table 3)

8.6.1 Surrogates: Prepare a working solution in methanol containing 2-fluorophenol, phenol-d_6, and 2,4,6-tribromophenol at 150 ng/µL and nitrobenzene-d_5, 2-fluorobiphenyl, and terphenyl-d_{14} at 100 ng/µL. Aliquots of the solution are spiked into all extracted samples, blanks, and QC samples according to the extraction SOP used.

8.6.2 Matrix Spike Standards: Prepare a working solution in methanol containing all analytes of interest (full list spike) at 100 ng/µL (Pyridine is at 200 ng/µL). Aliquots of the solution are spiked into the selected QC aliquots according to the extraction SOP used.

Note: The spiking level of surrogate and spike may need to be adjusted according to project requirements, if dilutions are expected due to high levels of extracted components, or if a lower calibration range is used

9) Apparatus and Equipment

9.1 Gas Chromatograph/Mass Spectrometer System

9.1.1 Gas Chromatograph - HP 5890, Agilent 6890, or Agilent 7890 -An analytical system complete with a temperature-programmable gas chromatograph suitable for large volume injection with cryogenic cooling, or for small volume injection without cryogenic cooling.

9.1.1.1 Atas Optic 2 or 3 large volume injector units - This allows for injection of up to 100 µL of solvent for each analysis. After injection into the cold injector, the solvent is vented while the analytes are retained within a specially packed liner. The injector is then flash heated and the analytes are transferred into the GC. The unit also controls gas flow rates.

9.1.1.2 Agilent Split/Splitless Injector - The injector is held at a constant temperature allowing for the direct transfer of analytes onto the analytical column. The unit also controls the gas flow rates.

9.1.1.3 Agilent MMI injector - This allows for injection of up to 1000 µL of extract. The injector can be temperature programmed and is equipped with cryogenic cooling. The unit also controls gas flow rates.

9.1.1.4 Autosampler - HP 7673, Agilent 7683, Agilent 7693, or equivalent with programmable operation.

9.1.1.5 All other required accessories, syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

9.1.2 Column: ZB-5MS, 30 m x 0.25 mm ID x 0.25 µm film thickness silicone-coated fused-silica capillary column with 5 m guard column (or equivalent). Recommended part number 7HG-6010-11-GGA.

9.1.3 Mass Spectrometer - HP 5972, Agilent 5973, or Agilent 5975 - Capable of scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 1A or 1B when a 10-50 ng tune standard is injected onto the GC

9.1.4 GC/MS Interface - Any GC-to-MS interface that gives acceptable calibration points for each compound of interest and achieves acceptable tuning performance criteria may be used.

9.1.5 Data System - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. NIST98 Mass Spectral Library is used for spectral comparisons.

9.2 Appropriate analytical balance (0.0001 g), volumetric flasks, syringes, vials, and bottles for standards preparation.

10) Preventative Maintenance

10.1 All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. Maintenance entries should include date, symptom of problem, corrective actions, and description of maintenance, date, and name. The log should contain a reference to return to analytical control.

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

10.3 Gas Chromatograph

10.3.1 Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, O-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chromatographic performance. In some cases liners and seals may be cleaned and re-used.

10.3.2 Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool. The column head pressure must be adjusted to maintain proper flow rates.

10.3.3 Over time, the column will exhibit poorer overall performance, as indicated by poor peak shape and reduced responses. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in performance is evident, more thorough maintenance is necessary. Some steps are to solvent rinse the split vent and septum lines with a mix of 20% methanol in DCM. When these and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

10.4 Mass Spectrometer

10.4.1 For units under service contract, certain maintenance is performed by instrument service staff, including pump oil changed, vacuuming boards, etc., as recommended by the manufacturer.

10.4.2 MS source cleaning should be performed as needed, depending on the performance of the unit. This may be done by the analyst or by instrument service staff.

10.4.3 Tune the MS as needed to result in consistent and acceptable performance while meeting the required ion abundance criteria.

11) Procedure

11.1 Sample Preparation

11.1.1 Water samples

11.1.1.1 Water samples are prepared using continuous liquid-liquid extraction and EPA method 3520C. Refer to EXT-3520 or samples may be prepared using separatory funnel procedures (EPA 3510C). Refer to EXT-3510.

11.1.1.2 Perform the extraction on a 1000mL or less aliquot of sample.

11.1.2 Soil, sediment, and solid samples are prepared using automated soxhlet extraction EXT-3541. The nominal sample size is 20g. Sample amounts may be decreased in the case of high-concentration waste samples. GPC cleanup may be used (SOP SOC-3640A).

11.1.3 Extracts should be screened by GC/FID (SOC-SCR).

11.1.4 Following sample preparation, sample extracts are then transferred to the extract cold storage unit. Extracts must be analyzed within 40 days of extraction.

11.2 The recommended GC/MS operating conditions are listed below. The GC conditions may be modified to accommodate specific instrument models and configurations.

Mass range:	35-500 amu
Scan Time:	1 sec/scan
Initial temperature:	45°C, hold for 3.5 minutes
Temperature program:	45-100°C at 15°C/min, 100-270°C at 10°C/min, 270-320°C at 6°C/min, hold until 3 minutes after benzo[g,h,i]perylene has eluted.
Detector interface temp:	300°C

Injector: Atas Optic 2, or Optic 3 or equivalent
Final time: 32.7 min.
Initialization time: 0.00 min.

Temperature profile: Initial temperature - 5°C

RAMP	RATE	FINAL TEMP	ISOTHERMAL TEMP
1		5°C	2.20 MIN
2	6°C/sec	305°C	30.5 min

Pressure ramps:

RAMP	START PRESS	FINAL TEMP	TARGET PRESS
1	5.00 PSI	0.7 min	**5.00 psi**
2	10.00 PSI	3.30 min	10.00 psi
3*	*psi	19 min	*psi
4*	*psi	8.0 min	*psi

* Will vary to give 1.0 ml/min. @ 45°C and the target pressure is set to give 1.5 ml/min @ 320°C.

Split state:

Time	Split State
Initial	Vent
0.75 min	Closed/splitless*
1.90 min	Open/split

* Splitless time may be adjusted to optimize GC performance.

Vent Flow	85 ml/min. @ 5 psi, 50°C
Split Flow	2-5 ml/min. @ 10 psi, 45°C
Sample Volume	20 µL (5972/5973)
	µL (5975)
Carrier Gas	Helium as 30-45 cm/sec

11.3 Initial Calibration

NOTE The calibration procedure(s) and options chosen must follow the ALS protocols. Any exceptions to the calibration procedures detailed in SOC-CAL, *Calibration of Instruments for Organics Chromatographic Analyses* are described as follows:

11.3.1 Prior to calibration, analyze the GC/MS tuning standard using instrument conditions used for calibration. Obtain the spectrum for evaluation using one of the following options:

- Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or part of any other closely eluting peak.
- Use one scan at the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or part of any other closely eluting peak.
- Use one scan either directly preceding or following the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or part of any other closely eluting peak.
- Use the average across the entire peak up to a total of 5 scans. If the peak is wider than 5 scans, the tune will consist of the peak apex scan and the two scans immediately preceding and following the apex. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or part of any other closely eluting peak.

11.3.2 Evaluate the spectrum obtained for DFTPP against the tuning criteria. Criteria are given in Tables 1and 1A. The GC/MS must meet the DFTPP ion abundance criteria prior to further analyses. To assess column performance and injection port inertness, pentachlorophenol and benzidine should be present at an acceptable level and peak tailing should not exceed a tailing factor of 2. DDT degradation should not exceed 20%. If excessive tailing, poor chromatography, or degradation of >20% is noted, the injection port may require cleaning. It may also be necessary to remove the first 15-30 cm of the GC column. If hardware tuning criteria cannot be met, the source may need cleaning, filaments replaced or other maintenance.

11.3.3 The internal standards should permit most of the components of interest in the chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Refer to Table 4 for internal standards and corresponding analytes assigned for quantitation. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1 in EPA method 8270D). If interferences are noted, use the next most intense ion as the quantitation ion (i.e. for 1,4-dichlorobenzene-d_4, use 152 m/z for quantitation).

11.3.4 Analyze 20 µL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound (see Table 1 in EPA method 8270D). Calculate response factors (RFs) for each compound relative to one of the internal standards as follows:

$$RF = (A_x C_{is})/(A_{is} C_x)$$

Where:

A_x = Area of the characteristic ion for compound being measured.
A_{is} = Area of the characteristic ion for specific internal standard.
C_{is} = Concentration of the specific internal standard (ng/µL).
C_x = Concentration of the compound being measured (ng/µL).

11.3.5 The percent relative standard deviation (%RSD) should be less than or equal to 20% for each compound. It is also recommended that a minimum response factor for the most common target analytes, as noted in Table 5, be demonstrated as a means to ensure that these compounds are performing as expected.

$$\%RSD = \frac{SD}{\overline{RF}} \, x \, 100$$

where:

RSD = relative standard deviation.
$\overline{RF}$ = mean of initial RFs for a compound.
SD = standard deviation of average RFs for a compound.

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(RF_i - \overline{RF})^2}{N - 1}}$$

where:

RF_i = RF for each of the calibration levels
N = Number of RF values (e.g., 6)

11.3.6 The relative retention times (RRT) of each compound in each calibration run should agree within 0.06 relative retention time units.

$$RRT = \frac{\text{Retention time of the analyte}}{\text{Retention time of the internal standard}}$$

11.3.7 Linearity - If the % RSD of any compound is 20% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

11.3.8 If the %RSD for a compound is >20%, then alternative calibration models should be used. Refer to SOC-CAL, Calibration of Instruments for Organics Chromatographic Analysis for alternative fit guidance.

11.3.9 If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too reactive

for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure.

11.3.10 When calculating the calibration curves using the alternative curve fits, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve (see Method 8000C for additional details). It is not necessary to re-analyze a low concentration standard; rather the data system can recalculate the concentrations as if it were an unknown sample. The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration.

11.3.11 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution should be verified on the mid-point concentration of the initial calibration as well as the laboratory designated continuing calibration verification level if closely eluting isomers are to be reported

11.4 Independent Calibration Verification

11.4.1 Following initial calibration, analyze an ICV standard. The ICV solution must contain all analytes in the calibration standards at a concentration in the middle of the range of the initial calibration. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. The maximum allowed % Difference or % Drift is ±30%.

11.4.2 If a second source standard is not available or is cost prohibitive (such as certain non-routine analytes), then a second lot number may be used as the ICV and must meet the criteria above.

11.4.3 After the multi-point calibration has passed all of the above criteria, and the Independent Calibration Verification has been performed, samples can be analyzed. The calibration curve mid-point standard may serve as the CCV for the opening set of samples within the same 12-hour window as the initial calibration.

11.5 Continuing Calibration

11.5.1 A calibration standard, or standards, at mid-concentration (See Table 2) containing all semivolatile analytes, DFTPP, and all required surrogates, must be analyzed every 12 hours during analysis. The DFTPP must result in a mass spectrum which meets the criteria given in Tables1A or 1B. These criteria must be demonstrated during each 12 hour shift. Obtain the DFTPP spectrum as described.

11.5.2 The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as required. If the EICP area for any of the internal standards changes by a factor of two (50% to 200%) from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as appropriate. When corrective action is taken, reanalysis of samples analyzed while the system

was malfunctioning is required. Update the reference spectra and retention times in the quantitation database for the instrument method or ID file. The initial calibration average RF or calibration curve is then used in the quantitation of subsequent analyses.

11.5.3 If the percent difference or percent drift for each compound is less than or equal to 20%, the initial calibration is assumed to be valid and the analysis of samples may begin. Calculate the percent drift using:

$$\%\ Drift = \frac{C_I - C_c}{C_I} \times 100$$

where: C_I = Compound standard concentration.
C_c = Measured concentration using selected quantitation method.

11.5.4 Due to the large number of compounds that may be analyzed by this method, some compounds may fail to meet the ≤ 20% criteria. If no more than 20% of the compounds, included in the initial calibration, differ from their true concentration by 40%, the initial calibration is valid and no corrective action is necessary.

11.5.5 In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. This is done by analyzing a MRL check standard for the failing compounds, where the resulting peaks for the failing compounds must pass 3:1 signal to noise.

11.5.6 Non-detected analytes can be reported from analyses when a CCV exhibit a positive bias (i.e., outside the upper control limit), no further documentation is required.

11.5.7 For situations when the CCV fails to meet the criterion in section 11.5.4, and a confirmed detection exceeds the MRL, the sample must be reanalyzed to ensure accurate quantification. If it is not possible to reanalyze the sample, the result must be reported as an estimated value.

11.5.8 A blank (method blank, GPC blank, or solvent blank) should be analyzed after the CCV, or at any other time during the analytical shift, to prove the system is free of contaminants. If contaminants are found in a method blank or GPC blank, then a solvent blank should be analyzed to help isolate the source of contamination.

11.5.9 Each of the most common target analytes in the calibration verification standard should meet the minimum response factors noted in Table 5.

11.5.10 If the minimum response factors are not met, the system should be evaluated, and corrective action should be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination of the front end of the analytical column, and active sites in the column or chromatographic system.

11.6 GCMS Analysis

11.6.1 Evaluate FID screen and make proper dilution (See FID screening SOP)

11.6.2 Spike the 1 mL extract obtained from sample preparation with 10 µL of the internal standard solution just prior to analysis. Use the same operating conditions as were used for initial calibration.

11.6.3 If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required 1.0 ng/µL of each internal standard in the extracted volume. The diluted extract must be reanalyzed.

11.6.4 Store the extracts at -10°C or less, protected from light in vials equipped with unpierced Teflon lined septa. Archive the extract in freezer for 3 months after analysis in the instrument/date specific storage boxes.

12) QA/QC Requirements

12.1 In addition to instrument criteria for calibration, the ability of each analyst/instrument to generate acceptable accuracy and precision must be documented prior to sample analysis (IPR study). This must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four tap water samples are spiked with each target analyte, extracted, and analyzed.

12.2 Method Detection Limits

12.2.1 A method detection limit (MDL) study, or verification of an existing LOD from an equivalent instrument must be completed prior to analysis of samples. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution (at a level below the MRL) for each target analyte, extract, and analyze. The MDL studies should be done for each matrix, prep method, and instrument. Refer to the SOP Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification..

12.2.2 Calculate the average concentration found (x) in the sample concentration, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study must be verified annually.

12.3 Limits of Quantification (LOQ)

12.3.1 The laboratory establishes a LOQ for each analyte as the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements. Analysis of a standard or extract prepared at the lowest point calibration standard provides confirmation of the established sensitivity of the method. The LOQ recoveries should be within the laboratories LCS acceptance limits to verify the data reporting limit. Refer to the SOP Performing and Documenting Method Detection Limit Studies and Establishing Limits of Detection and Quantification (CE-QA011/ADM-MDL).

12.4 The Method Reporting Limits (MRLs) used at ALS are the routinely reported lower limits of quantitation which take into account day-to-day fluctuations in instrument sensitivity as well as other factors. These MRLs are the levels to which ALS routinely reports results in order to minimize false positive or false negative results. The MRL is normally two to ten times the method detection limit.

12.5 Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project specific quality assurance plans (QAPP). General QA requirements for DoD QSM are defined in the laboratory SOP, Department *of Defense Projects - Laboratory Practices and Project Management (ADM-DOD5).* General QC Samples are:

12.5.1 Method blank - A method blank is extracted and analyzed with every batch of 20 or fewer samples to demonstrate that there are no method interferences. The method blank must demonstrate that interferences from the analytical and preparation steps are minimized. No target analytes should be detected above the MRL in the method blank. For some project specific needs, exceptions may be noted and method blank results above the MRL may be reported for common lab contaminants (phthalate esters, etc.).

12.5.2 A lab control sample (LCS) must be extracted and analyzed with every batch of 20 or fewer samples. The LCS is prepared by spiking a blank with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

12.5.2.1 Acceptance criteria for lab control samples can be found in the laboratory Data Quality Objectives (DQO) tables. The accuracy of the analysis is controlled on a subset of target analytes. If the project analyte list is fewer than 20 analytes, all are considered control analytes. Analytes which are used for control analytes are listed in Table 6. For DoD projects all project target analytes are considered control analytes. If the LCS recovery for any control analyte fails acceptance limits, the cause is evaluated and corrective action taken as necessary. If instrument corrective action is not applicable or ineffective, re-extraction of the associated samples may be required. If any other analyte fails the advisory acceptance limits, the analyst must evaluate the impact on data quality and take any necessary corrective action, which may include re-extraction of the associated samples. Project-specific requirements may require all compounds to be treated as control analytes, or dictate use of project acceptance criteria.

12.5.3 A matrix spike/duplicate matrix spike (MS/DMS) must be extracted and analyzed with every batch of 20 or fewer samples. The MS is prepared by spiking a sample aliquot with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$\%RPD = \frac{R1 - R2}{(R1 + R2)/2} x\ 100$$

Where R1 = %recovery of the MS
R2= %recovery of the DMS

12.5.3.1 The acceptance limits for the MS/DMS recovery can be found in the laboratory Data Quality Objectives (DQO) tables. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken. (See Quality Assurance Manual section 11) The RPD acceptance limits are 30% for water and 40% for soils, sediments, and solids. Project-specific requirements may dictate the use of project acceptance criteria.

12.5.4 The acceptance limits for the surrogates can be found in the laboratory Data Quality Objectives (DQO) tables. If any surrogate recovery is outside acceptance criteria, the sample data must be closely evaluated for possible matrix interferences. If none are present, then corrective action must be taken. The sample should be re-analyzed if instrument factors (calibration, injection port) are suspected. If not, re-extraction and re-analysis is required, except in cases of high recovery and no positive hits in the sample for the analyte class represented by the particular surrogate.

12.5.5 The acceptance criteria listed in the laboratory Data Quality Objective (DQO) Tables are current criteria, but are subject to change as control limits are updated.

12.6 Additional QA/QC measures include control charting of QC sample results

13) Data Reduction and Reporting

13.1 Qualitative Analysis - The qualitative identification of compounds determined by this procedure is based on retention time, and comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the instrument and conditions used for the sample analysis. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds should be identified as present when the criteria below are met.

13.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

13.1.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

13.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

13.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two

isomer peaks is <25% of the sum of the 2 peak heights. Otherwise, structural isomers are identified as isomeric pairs.

13.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks appear to represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification. When analytes coelute, the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

13.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. Refer to method 8270D for guidance on tentatively identified compound (TIC) identification and quantification.

13.3 Quantitation and Calculations

13.3.1 The GC/MS data stations, in current use, all use the H-P RTE Integrator to generate the raw data used to calculate the standards $\overline{RF_x}$ values, the sample amounts, and the spike values. The software does three passes through each data file. The first two identify and integrate each internal standard and surrogate. The third pass uses the time-drift information from the first two passes to search for all method analytes in the proper retention times and with the proper characteristic quantitation ions

When $\overline{RF_x}$ is used, calculate the extract concentration as follows:

$$C_{ex} = \frac{(Resp_x)(Amt_{ISTD})}{(Resp_{ISTD})(\overline{RF_x})}$$

Where:

C_{ex} = the concentration in the sample extract (ppb);
$Resp_x$ = the peak area of the analytes of interest;
$Resp_{ISTD}$ = the peak area of the associated internal standard;
Amt_{ISTD} = the amount, in ppb, of internal standard added
$\overline{RF_x}$ = the average response from the initial calibration.

13.3.2 The concentration of analytes in the original sample is computed using the following equations:

Aqueous Samples: $Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$

Where Cex = Concentration in extract in ng/mL
Vf = Final volume of extract in mL

D = Dilution factor
Vs = Volume of sample extracted, liters

Non-aqueous Samples: $$Concentration\,(ug/Kg) = \frac{(Cex)\,(Vf)\,(D)}{(W)}$$

Where Cex = Concentration in extract in ng/mL
Vf = Final volume of extract in mL
D = Dilution factor
W = Weight of sample extracted in grams.

13.4 Data Review and Assessment

13.4.1 Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP *Laboratory Data Review Process (ADM-DREV)* for details. The person responsible for final review of the data report and/or data package should assess the overall validity and quality of the results and provide any appropriate comments and information to the Project Chemist to inclusion in the report narrative

13.5 Reporting

13.5.1 Refer to the SOP for Data Reporting and Report Generation for reporting guidelines.

13.5.2 Reports are generated in the ALS LIMS by compiling the SMO login, sample prep database, instrument, date, and client-specified report requirements (when specified). This compilation is then transferred to a file which the Stealth reporting system uses to generate a report. The forms generated may be ALS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

14) Contingencies for Handling Our-of-Control or Unacceptable Data

14.1 Refer to the SOP for *Nonconformance and Corrective Action Procedures* (ADM-NCAR) for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

15) Method Performance

15.1 This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2 The method detection limit (MDL) is and related method reporting limit(s) were established using the procedure described in SOP *Performing and Documenting Method Detection Limit Studies and Establishing Limits of Detection and Quantification* (Ce-QA011/ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the ALS Quality Assurance Manual.

16) Pollution Prevention and Waste Management

16.1 The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Lab Waste Management Plan.

16.2 This method uses Methylene Chloride and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and recycled off site.

16.3 This method uses non-halogenated solvents and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

17) Training

17.1 Training is documented following ADM-TRAIN, ALS-Kelso Training Procedure.

18) Method Modifications

18.1 Section 11.5.4 - No limit defined in reference method, so lab assigned a limit of 40% based on CLP protocols.

18.2 Lower reporting levels are achieved in this procedure through injection of larger volumes and a lower calibration range. Lower limits of quantitation for soils are > 10 µg/Kg for soils and > 0.2 µg/L for waters.

18.3 Minimum Response factors listed in Table 5 were determined from representative initial calibrations performed after system maintenance. Recommended minimum response factors listed in Table 4 of EPA 8270D are not applicable due the differences in quantitation levels from the reference method.

19) References

19.1 *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry*, Method 8270D, EPA Test Methods for Evaluating Solid Waste, SW-846, Final Update IV, February 2007.

19.2 *Determinative Chromatographic Separations*, Method 8000C, EPA Test Methods for Evaluating Solid Waste, SW-846, On-Line March 2003.

19.3 DoD Quality Systems Manual for Environmental Laboratories current version.

19.4 TNI Standard, Volume 1- 2009, 2016.

19.5 ISO/IEC 17025: 2017

20) Changes Since the Last Revision

Summary of Revision Changes			
Rev. #	Effective Date	Document Editor	Description of Changes
11.0	12/2/2020	K Clarkson	Section 6.1: updated chemical handling and storage references. Sections 7.1 & 7.2: updated container and sample storage requirements. Section 11.1.1.1 & 11.1.1.2: Updated applicable water extraction methods and volume requirements

			Section 11.1.2: updated applicable soil extraction methods and mass requirements. Section 17.1: updated training documentation

21) Attachments, Tables, and Appendices

21.1 Table 1A - DFTPP Key Ions and Ion Abundance Criteria.

21.2 Table 1B - DFTPP Key Ions and Ion Abundance Criteria for 5973 and newer GC/MS Systems.

21.3 Table 2 - 8270 LL Standards.

21.4 Table 3 - QC Standards.

21.5 Table 4 - Semivolatile Internal Standards With Corresponding Analytes Assigned for Quantitation.

21.6 Table 5 - Recommended Minimum Response Factors.

21.7 Table 6 - Control Analyties for Non- DoD Projects.

21.8 Table 7 - Summary of Corrective Actions.

Table 1A
DFTPP Key Ions and Ion Abundance Criteria

Method 8270D Ion Abundance Criteria		CLP OLM04.2 Ion Abundance Criteria	
Mass	Ion Abundance Criteria	Mass	Ion Abundance Criteria
51	30-60% of mass 198	51	30-80% of mass 198
68	< 2% of mass 69	68	<2% of mass 69
70	< 2% of mass 69	69	Present
127	40-60% of mass 198	70	<2% of mass 69
197	< 1% of mass 198	127	25-75% of mass 198
198	Base peak,100% relative abundance	197	<1% of mass 198
199	5-9% of mass 198	198	Base peak,100% relative abundance
275	10-30% of mass 198	199	5-9% of mass 198
365	> 1% of mass 198	275	10-30% of mass 198
441	Present but less than mass 443	365	>0.75% of mass 198
442	> 40% of mass 198	441	Present, but less than mass 443
443	17-23% of mass 442	442	40-110% of mass 198
		443	15-24% of mass 442

Table 1B
DFTPP Key Ions and Ion Abundance Criteria
FOR **5973** and newer GC/MS SYSTEMS

Mass	Ion Abundance Criteria
51	10-80% of mass 198
68	0-2% of mass 69
70	0-2% of mass 69
127	10-80% of 198
197	0-2% of 198
198	30-100% of 442 (alternate base)
199	5-9% of 198
275	10-60% of 198
365	1-50% of 442
441	0.01-100% of 443
442	30-100% of 198 (alternate base)
443	15-24% of 442

Alternate tuning criteria (from Method 525.2 or CLP OLM04.2) may be used provided that method performance is not adversely affected and that method performance criterion is met. The criteria used must be the same for **all** ion abundance criteria checks associated with a given analysis. For example, initial calibration, continuing calibration(s), QC, and sample analyses for a given sample must all use the same criteria.

Table 2
8270-LL Standards

CALIBRATION

The calibration curve is prepared from the following Supelco stock standards (or equivalent from other vendors*):8270 CLP Mix, Equity 8270 Mix 4, Equity Benzidines Mix, Equity N-Nitrosodiphenylamine, 1-Methylnaphthalene, Acetophenone and 8270 Surrogates Mix.

The mix of standards to prepare the ICAL intermediate may be changed to meet project requirements.

Calibration curve is prepared from a 100 ppm intermediate solution and ranges from 50 - 10000ppb depending upon project requirements. Add internal standard when curve is prepared.

Place in amber autosampler vial, cap.

Store at -10°C. Expiration is 1 year from date prepared or expiration of parent standard(s), whichever is earlier.

ICV
Recommended: AccuStandard catalog # (or equivalent from other vendors*):

CLP-HC-BN-R	2000 ppm BN mix
CLP-HC-A-PAK	2000 ppm Acid composite mix
Z-014E-R3	2000 ppm Composite 3 mix
M-8270-SS-PAK	4000 ppm Surrogates mix
Z-014J	4000 ppm Internal standards mix
M-625C	25000 ppm DFTPP
Z-014F	2000 ppm Benzidines mix

Add 10 µl internal standard for each 1 ml of ICV prepared.
Place in autosampler vial, analyze, recap, and refrigerate.
Expiration is one year after ICV was prepared or the expiration date from the manufacturer, which ever is earlier.

CCV & TUNE

Prepare 1 ml 8270_LL CCV standard that falls near the mid point of the calibration curve. DFTPP is added to the CCV to give a final concentration of 2.5 µg/ml. The CCV is stored in an amber autosampler vial and is good for one week.

RECAP AND STORE IMMEDIATELY AFTER INJECTING

* Vendor must be A2LA and/or ISO9000 certified.

Table 3
QC Standards

Surrogate Spiking Solution					
Parent	Initial Concentration	Aliquot	Final Volume	Final Concentration	Solvent
8-61377*	5000 µg/mL	20 mL	1000 mL **	100 µg/mL	Methanol
8-61376*	10000 µg/mL	15 mL		150 µg/mL	
S-8522*	5000 µg/mL	20 mL		100 µg/mL	
Expiration - Unopened = 6 months from prep date					
* 8-61377 - Supelco BN Surrogate Standard (custom mix) - reorder 4 at a time					
* 8-61376 - Supelco Acids Surrogate Standard (custom mix) - reorder 3 at a time					
* S-8522 - Accustandard custom - reorder 4 at a time.					
** Split into 4 - 250 mL bottles.					
LCS/MS Spiking Solution					
Parent	Initial Concentration (ppm)	Aliquot (mL)	Final Volume	Final Concentration	Solvent
50608	1000	10	100 mL	100 ppm	MeOH
4-8467	2000	5			
46702-u	5000	2			
86-1148	2000	5			
79131	1000	10			
48462	2000	5			
App9186-20x	2000	5			
Expiration -6 months from prep date.					

TABLE 4
Semivolatile Internal Standards with corresponding Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d4 Internal Standard		
N-Nitrosodimethylamine	2-Chlorophenol	N-Nitrosodi-n-propylamine
Aniline	Benzyl Alcohol	Hexachloroethane
2-Fluorophenol (surrogate)	Pyridine	2-Methylphenol
Bis(2-chloroethyl) Ether	1,2-Dichlorobenzene	3- and 4-Methylphenol (coeluting cpds)
Phenol-d_6 (surrogate)	1,3-Dichlorobenzene	Bis(2-chloroisopropyl) Ether
Phenol	1,4-Dichlorobenzene	Nitrobenzene
Nitrobenzene-d5 (surrogate)		
Naphthalene-d8 Internal Standard		
Naphthalene	2,4-Dimethylphenol	4-Chloroaniline
Benzoic Acid	Isophorone	Hexachlorobutadiene
2,4-Dichlorophenol	Bis(2-chloroethoxy)methane	2-Methylnaphthalene
1-Methylnaphthalene	4-Chloro-3-methylphenol	1,2,4-Trichlorobenzene
2-Nitrophenol		
Acenaphthene-d10 Internal Standard		
2-Fluorobiphenyl (surrogate)	Acenaphthylene	Fluorene
Hexachlorocyclopentadiene	Acenaphthene	4-Nitrophenol
2-Chloronaphthalene	Dibenzofuran	2,4,6-Trichlorophenol
2-Nitroaniline	2,4-Dinitrotoluene	2,4,5-Trichlorophenol
3-Nitroaniline	2,6-Dinitrotoluene	2,4-Dinitrophenol
4-Nitroaniline	Diethyl Phthalate	Azobenzene
Dimethyl Phthalate	4-Chlorophenyl Phenyl Ether	2,2,4,6-Tetrachlorophenol
2-Methyl-4,6-dinitrophenol		

Table 4 (continued)

Phenanthrene-d10 Internal Standard		
Phenanthrene	Anthracene	Pentachlorophenol
4-Bromophenyl Phenyl Ether	Di-n-butyl Phthalate	Carbazole
Hexachlorobenzene	Fluoranthene	
Chrysene-d12 Internal Standard		
Pyrene	Benz(a)anthracene	Terphenyl-d14 (surrogate)
Butylbenzyl Phthalate	Bis(2-ethylhexyl) Phthalate	
3,3'-Dichlorobenzidine	Chrysene	
Perylene-d12 Internal Standard		
Di-n-octyl Phthalate	Benzo(a)pyrene	Benzo(g,h,i)perylene
Benzo(b)fluoranthene	Indeno(1,2,3-c,d)pyrene	
Benzo(k)fluoranthene	Dibenz(a,h)anthracene	

Table 5
Recommended Minimum Response Factors

Compound	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl) Ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.500
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitrosodi-n-propylamine	0.500
Hexachloroethane	0.300
Isophorone	0.300
Nitrobenzene	0.200
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.100
Bis(2-chloroethoxy)methane	0.200
2,4-Dichlorophenol	0.100
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
2-Methylnaphthalene	0.300
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.700
2,-Nitroaniline	0.010
Dimethyl Phthalate	0.010

Table 5 (continued)

Compound	Minimum Response Factor (RF)
2,6-Dinitrotoluene	0.100
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.700
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.800
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.600
Anthracene	0.600
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.600

Table 5 (continued)

Compound	Minimum Response Factor (RF)
Chrysene	0.600
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.600
Benzo(k)fluoranthene	0.600
Benzo(a)pyrene	0.600
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

Table 6
Control Analytes for Non-DoD Projects

1,2,4-Trichlorobenzene
1,4-Dichlorobenzene
2,4-Dinitrotoluene
2-Chloronaphthalene
2-Chlorophenol
4-Bromophenyl Phenyl Ether
4-Chloro-3-methylphenol
4-Nitrophenol
Acenaphthene
Benzo(a)pyrene
Diethyl Phthalate
Hexachloroethane
N-Nitrosodi-n-propylamine
Pentachlorophenol
Phenol
Pyrene

Table 7

Summary of Corrective Actions

Method Reference	Control	Specification and Frequency	Acceptance Criteria	Corrective Action
EPA 8000C, EPA 8270D	ICAL	Prior to sample analysis	% RSD ≤ 20 COD ≥ 0.990	Correct problem then repeat ICAL
EPA 8000C,	ICV	After ICAL	± 30% Diff	Correct problem and verify second source standard; rerun second source verification. If fails, correct problem and repeat initial calibration.
EPA 8270D	CCV	Prior to sample analysis	± 20% Diff	Correct problem then repeat CCV or repeat ICAL
EPA 8000C,	Method Blank	Include with each analysis batch (up to 20 samples)	<MRL	If target exceeds MRL, reanalyze to determine if instrument was cause. If still noncompliant then: Re-extract or reanalyze samples containing contaminate, unless samples contain > 20x amount in blank.
EPA 8270D	Laboratory Control Sample	Include with each analysis batch (up to 20 samples)	See DQO Tables	If exceeds limits, re-extract and re-analyze
EPA 8000C,	Matrix Spike	Include with each analysis batch (up to 20 samples)	See DQO Tables	Evaluate data to determine if the there is a matrix effect or analytical error
EPA 8270D	Sample Duplicates	Include with each analysis batch (up to 20 samples)	W: RPD ≤ 30 S: RPD ≤ 40	Re-homogenize and re-analyze if result is > 5 X the MRL

Semi-Volatile Organic Compounds by GC/MS Selective Ion Monitoring

DOCUMENT ID: SVM-8270S, REV 9.0

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1) Scope & Applicability

1.1. This procedure is used to determine the concentrations of Semi-Volatile Organic Compounds in water, soil, and tissue matrices using EPA Method 8270D and Selected Ion Monitoring (SIM). This procedure may also be applicable to various miscellaneous waste samples. Table 1 lists compounds that may be determined by this method and lists nominal method reporting limits (MRLs) in water, soil, and tissue. Other compounds may be analyzed to meet project requirements. Due to the nature of this analysis and its applications, MRLs may vary due to project specifications. Table 1 lists Method Detection Limits (MDLs) that have been achieved, however, MDLs may change as MDL studies are performed, and may vary depending on the instrument used and preparation method.

1.2. The procedure is intended for samples containing trace-level amounts of target compounds. Samples containing high concentrations of target analyte will not be analyzed undiluted. Extracts may be screened using GC/FID to estimate the hydrocarbon content and concentrations of individual polynuclear aromatic hydrocarbons (PAHs). Samples containing PAHs are diluted prior to analysis. All MRLs will be adjusted in accordance with this dilution. Therefore, samples containing high levels of PAHs will not be analyzed to achieve the optimum MRLs for the analysis.

1.3. This procedure can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone phase. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons, phthalate esters, nitrosamines, haloethers, ketones, anilines, aromatic nitro compounds, and phenols, including nitrophenols. Most acidic compounds are derivatized in tissue samples prior to analysis to aid sensitivity and chromatographic resolution.

1.4. Other compounds than those listed in Table 1 may be analyzed. However, analytes not summarized in Table 1 may not been validated with a method detection limit study. Therefore, the lab will not use this procedure to analyze for non-routine analytes unless a similar analyte has been validated with a MDL study. As a general rule, the MRL for these compounds will equal the MRL of a similar compound in the routine analyte list. Results will not be reported below this estimated MRL.

1.5. In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DoD ELAP. QC requirements defined in the SOP *Department of Defense Projects - Laboratory Practices and Project Management* (ADM-DOD) may supersede the requirements defined in this SOP.

2) Summary of Procedure

2.1 This method provides Gas Chromatography/Mass Spectrometry (GC/MS) conditions for the detection of Semi-volatile Organic Compounds. Prior to the use of this method, an appropriate sample preparation method must be used to recover the analytes of interest. Soil, tissue, and colored water extracts may be cleaned using EPA Method 3640 (gel permeation chromatography) prior to analysis.

2.2 A 0.5-20 µL aliquot of the extract is injected into the gas chromatograph (GC). The compounds are separated on a fused silica capillary column. Compounds of interest are detected by a mass selective detector in the selective ion mode. Identification of the

analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard, and by comparing mass spectra of analytes with mass spectra of reference materials. Quantitative analysis is performed by using the authentic standard to produce a response factor and calibration curve, and using the calibration data to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume. To achieve MRLs listed in Table 1 the initial sample amount, final extract volume, and injection aliquot are specified for each product.

2.3 The following compounds may require special treatment when being determined by this method. Benzidine, 3, 3'-Dichlorobenzidine, nitrophenols, Hexachlorocyclopentadiene, Pentachlorophenol, Benzoic Acid and Benzyl Alcohol are subject to poor extraction efficiency or erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

3) Definitions

3.1 For laboratory definitions applicable to most analyses, refer to the SOP for *Sample Batches*.

4) Responsibilities

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

4.2 It is the responsibility of the department supervisor/manager to document analyst training .and method proficiency, as described in: *Employee Training and Orientation* (ADM-TRAIN).

5) Interferences

5.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation of the samples. Corrective action should be taken to eliminate the interferences.

5.2 Accurate determination of phthalate esters can pose difficulties when using this methodology. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware may occur when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalates can best be minimized by avoiding contact with any plastic materials. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

5.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of an instrument blank to check for cross contamination.

6) Safety

6.1 Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in SDSs where available. Refer to the ALS Chemical Hygiene Plan and the appropriate SDSs prior to beginning this method.

6.2 This method uses Dichloromethane, a known human carcinogen. Refer to the methylene chloride policy document, ENV-HSE-NA-EX-006-EN for proper handling.

G:\SAFETY\TRAINING\Methylene Chloride\Methylene Chloride - NA 031419.pdf.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Certified clean containers should be purchased for sample collection. The sample containers should be of glass or Teflon and have screw-top covers with Teflon liners. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

7.2 Water and soil samples should be iced or refrigerated at 4 ± 2□C from time of collection until extraction. Tissue samples are stored frozen until extraction.

7.3 Water and soil samples should be iced or refrigerated at <6 C from time of collection until extraction. Tissue samples are stored frozen until extraction.

8) Standards, Reagents, and Consumable Materials

8.1 Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP *Reagent/Standards Login and Tracking* (ADM-RTL) for the complete procedure and documentation requirements.

8.2 BSTFA + TMCS 99:1, Supelco 33154-U: This is used to derivatize the phenols in extracts.

8.3 Standards

8.3.1 Stock standard solutions may be purchased from a number of vendors. All reference standards, where possible, must be traceable to SI units or NIST certified reference materials. Commercially prepared stock standards are typically used when available at a concentration of 100 µg/mL or more. Standard concentrations can be verified by comparison versus an independently prepared standard. Alternatively, prepare stock standard solutions at a concentration of 1000 µg/mL by dissolving 0.0100 g of reference material in methylene chloride or other suitable solvent and diluting to volume in a 10 mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard.

8.3.2 Transfer the stock standard solutions into amber Teflon-sealed crimp top autosampler vials at -10°C and protect from light, or store as

recommended by the manufacturer. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. The expiration date is 1 year.

8.3.3 Unopened stock standards and neat materials have an expiration date equal to the manufacturer's recommendation. Neat material that does not have a manufacturer's recommended expiration date should be replaced after five years. Stock standard solutions received in sealed ampules with manufacture expiration dates in excess of 1 year have an expiration date of 1 year from the date of opening the sealed ampoule.

8.4 Internal Standard Solutions - The internal standards are 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} (See Table 3 for corresponding compounds). The nominal concentration of the standard is 100 ppb. For ultralow-level analyses, use 10 ppb. Each 100 µL of sample extract undergoing analysis should be spiked with 1 µL of the internal standard solution. Store at -10°C or less when not being used. When using premixed certified solutions, store according to the manufacturer's recommendations. The expiration date is 1 year

8.5 GC/MS Tuning/Standard, A methylene chloride solution containing decafluorotriphenylphosphine (DFTPP) and pentachlorophenol is prepared. The concentration used (ng injected) will vary depending on the instrument model and/or sensitivity, but generally is 10-50ng. Store at -10°C or less when not being used, or store according to the manufacturer's recommendations. The expiration date is 1 year.

8.6 Calibration Standards

8.6.1 A minimum of five initial calibration standards must be prepared from stock solutions (note that a seven point calibration is recommended). One of the calibration standards must be at a concentration at or below the method reporting limit. The others should correspond to the range of concentrations found in samples, but should not exceed the working range of the GC/MS system. Each 1 mL aliquot of calibration standards should be spiked with 10 µL of the internal standard solution prior to analysis. All calibration standards should be stored at -10°C or less and should be freshly prepared from stocks every 1 year, or sooner if check standards indicate a problem.

8.6.2 A calibration range of 10-5000 ng/mL is recommended. Higher concentrations may be prepared for poorer performing analytes. Calibration levels and injection amount may be adjusted to meet project-specific requirements.

8.6.3 The daily calibration standard (CCV) is prepared at a mid-level concentration from stock solutions. The CCV is prepared weekly and can be stored at 4 ± 2°C, or as recommended by the manufacturer.

8.7 QC Standards

8.7.1 Surrogates: Prepare a working solution in methanol containing 2-

fluorophenol, phenol-d_6, and 2,4,6-tribromophenol at 150 ng/µL and nitrobenzene-d5, 2-fluorobiphenyl, and terphenyl-d_{14} at 100 ng/µL. This solution may be combined with the surrogate solution used to monitor analyses for PAHs only (Fluorene-d_{10}, and Fluoranthene-d_{10}). Custom project-specific surrogate solutions may also be used. Aliquots of the solution are spiked into all extracted samples, blanks, and QC samples according to the extraction SOP used.

8.7.2 Matrix Spike Standards: Prepare a working solution in methanol containing all analytes of interest ("full list spike"). Base-Neutral analytes and acid analytes are prepared at a concentration of 100 ng/µL. Aliquots of the solution are spiked into the selected QC aliquots according to the extraction SOP used

9) Apparatus and Equipment

9.1 Gas Chromatograph/Mass Spectrometer System

9.1.1 Gas Chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless, split, or large-volume injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

9.1.2 Column: 5% Diphenyl, 95% Dimethyl Polysiloxane - 30 m x 0.25 mm ID x 0.25 µm film thickness silicone-coated fused-silica capillary column or equivalent (recommended: Phenomenex Zebron ZB-5MS, catalog # 7HG-G010-11-GGA.) Some projects may require a similar column, but with a different phase, to achieve improved separation.

9.1.3 Mass Spectrometer - Capable of scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode, and capable of operating in the SIM mode.

9.1.4 GC/MS Interface - Any GC-to-MS interface that gives acceptable calibration points for each compound of interest and achieves acceptable tuning performance criteria may be used.

9.1.5 Data System - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits.

9.1.6 Appropriate analytical balance (0.0001 g), volumetric flasks, syringes, vials, and bottles for standards preparation

10) Preventative Maintenance

10.1 All maintenance activities are recorded in a maintenance logbook kept for each

instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. Maintenance entries should include date, symptom of problem, corrective actions, and description of maintenance, date, and name. The log should contain a reference to return to analytical control.

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

10.3 Gas Chromatograph

10.3.1 Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance may include swabbing out the port, changing the injection port liner, seal, washer, O-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chromatographic performance. In some cases liners and seals may be cleaned and re-used.

10.3.2 Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting too.

10.3.3 Over time, the column will exhibit poorer overall performance, as indicated by poor peak shape and reduced responses. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in performance is evident, more thorough maintenance is necessary. Some steps are to solvent rinse the split vent and septum lines with a mix of 20% methanol in DCM. When these and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties

10.4 Mass Spectrometer

10.4.1 For units under service contract, certain maintenance is performed by instrument service staff, including pump oil changed, vacuuming boards, etc., as recommended by the manufacturer.

10.4.2 MS source cleaning should be performed as needed, depending on the performance of the unit. This may be done by the analyst or by instrument service staff.

10.4.3 Tune the MS as needed to result in consistent and acceptable performance while meeting the required ion abundance criteria given in section 11.

11) Procedure

11.1 Sample Preparation

11.1.1 Water, soil, tissue and waste samples are prepared using the appropriate extraction and cleanup methods (refer to SOPs) and may be screened by GC/FID (see screening SOP). Soil, tissue, waste, and colored water extracts may be cleaned using EPA Method 3640 (GPC).

11.1.2 Tissue extracts and standards that are to be analyzed for phenols are derivatized by the following procedure: 100 µL of BSTFA+TMCS is added to 1.0 mL of the extract or standard in 2 mL autosampler vial. The vial is then sealed with a cap and heated to 60 C for 1.0 hour.

11.1.3 Following sample preparation, sample extracts are then transferred to the extract cold storage unit. Extracts must be analyzed within 40 days of extraction.

11.2 Typical GC/MS operating conditions are listed below, but should be optimized for specific instruments and projects to achieve optimal performance for the application.

Ion dwell time:	10 - 50 msec per ion
Initial temperature:	40°C, hold for 1 minutes
Temperature program:	40-150°C at 10°C/min hold for 2 minutes
Temperature program:	150-270°C at 15°C/min hold for 2 minutes
Final temperature:	270-300°C at 30°/min, hold for 4.33 minutes
Injector temperature:	275°C
Detector interface temp:	300°C
Injector:	Atas Optic, Agilent PTV or equivalent (parameters are method-specific)
Sample volume:	0.5-20 µL
Carrier gas:	helium at 1.3mL/min (constant flow)

11.3 Selected Ion Acquisition

11.3.1 Determine the ions to be monitored for the compounds of interest. Refer to Table 2 for characteristic ions. At a minimum, 2 ions (preferably 3 or more) are monitored for each compound, and a minimum of 3 monitored for halogenated compounds and compounds with more complex fragmentation patterns. Set the SIM windows in order to monitor the correct ions at the correct time, based on chromatographic elution of the compounds. This can be setup by analyzing a standard using a full scan analysis and using the GC conditions of the SIM analysis. This analysis will give retention time and spectral information for determining the location of start times for the SIM groups or windows. This is often referred to as a "locator" analysis.

11.3.2 Select the dwell times to be used for each group of ions to be monitored. Dwell times should be selected in order to provide a sufficient number of measurements across the chromatographic peak to accurately define the peak shape. Too few measurements across the peak will result in poor definition of the peak and subsequently result in poor accuracy and precision of results. Too

many measurements across the peak may result in inconsistent detector behavior over the calibration range. Significant differences in dwell times may also affect sensitivity.

11.4 Initial Calibration

NOTE: The calibration procedure(s) and options chosen must follow the ALS protocols. Any exceptions to the calibration procedures detailed in the ALS SOP for *Calibration of Instruments for Organics Chromatographic Analyses* (SOC-CAL) are described as follows:

11.4.1 Prior to calibration, analyze the GC/MS tuning standard using instrument conditions used for calibration. The DFTPP solution is analyzed to verify GC/MS tuning, injection port inertness, and GC column performance. This injection is acquired in full scan mode and evaluated in accordance with the method specified criteria.

11.4.2 Evaluate the spectrum obtained for DFTPP against the tuning criteria in Table 4 and 4A. The GC/MS must meet the DFTPP ion abundance criteria prior to further analyses. Once the instrument is tuned, all subsequent analyses of standards, samples, and QA/QC samples must be analyzed using the identical mass spectrometer operating conditions. If hardware tuning criteria cannot be met, the source may need cleaning, filaments replaced or other maintenance. The following tune verification options can be used.

- Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.

- Use one scan at the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.

- Use one scan either directly preceding or following the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.

- Use the average across the entire peak up to a total of 5 scans. Peak integration must be consistent with standard operating procedure. If the peak is wider than 5 scans, the tune will consist of the peak apex scan and the two scans immediately preceding and following the apex.

Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.

11.4.3 To assess column performance and injection port inertness, pentachlorophenol should be present at an acceptable level and peak tailing should not be excessive. Tailing is evaluated using a tailing factor. The tailing factor is calculated by the processing software as follows (also refer to EPA 8270D, Figure1):

Tailing Factor = *PeakBack/PeakFront*

Where:

PeakBack is the time from Peak Apex to Peak End measured at 10% of the peak height
PeakFront is the time from Peak Start to Peak APEX measured at 10% of the peak height.

The tailing factor should not exceed 2. If excessive tailing or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to remove the first 15-30 cm of the GC column.

11.4.4 The internal standards should permit most of the components of interest in the chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Refer to Table 3 for internal standards and corresponding analytes assigned for quantitation. Use the base peak ion from the specific internal standard as the primary ion for quantitation (See Table 2). If interferences are noted, use the next most intense ion as the quantitaion ion (i.e. for 1,4-dichlorobenzene-d_4, use 152 m/z for quantitation).

11.4.5 Analyze each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound (as indicated in Table 2). Calculate response factors (RFs) for each compound relative to one of the internal standards as follows:

$$RF = (A_x C_{is})/(A_{is} C_x)$$

where:

A_x = Area of the characteristic ion for compound being measured.
A_{is} = Area of the characteristic ion for specific internal standard.
C_{is} = Concentration of the specific internal standard (ng/µL).
C_x = Concentration of the compound being measured (ng/µL).

11.4.6 The recommended minimum acceptable average RF for target analytes is given in Table 5. If the RF does not meet these recommendations the GC system may require maintenance (see section 9) and recalibration.

11.4.7 The percent relative standard deviation (%RSD) should be less than 20% for each compound. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units.

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

Where:

RSD = relative standard deviation.

$\overline{RF}$ = mean of 5 initial RFs for a compound.
SD = standard deviation of average RFs for a compound.

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(RF_i - RF)^2}{N - 1}}$$

where:

RF_i = RF for each of the 5 calibration levels
N = Number of RF values (i.e., 5)

11.4.8 Linearity - If the % RSD of any compound is 20% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

11.4.9 If the %RSD for a compound is >20%, then alternative calibration models should be used. See the SOP (SOC-CAL) *Calibration of Instruments for Organics Chromatographic Analysis* for further guidance.

11.5 Review of calibration curve

11.5.1 The calibration curve must be reviewed to ensure it represents the calibration data. This is done by re-fitting each calibration level against the true concentration of each calibration standard. See the SOP (SOC-CAL) *Calibration of Instruments for Organics Chromatographic Analysis* for further guidance.

11.5.2 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution should be verified on the mid-point concentration of the initial calibration as well as the laboratory designated continuing calibration verification level if closely eluting isomers are to be reported

11.6 Independent Calibration Verification

11.6.1 Following initial calibration, analyze an ICV standard. The ICV solution must contain all analytes in the calibration standards. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. For each compound of interest, the calculated value must be ± 30% of the true value for the initial calibration to be valid.

11.7 Continuing Calibration

11.7.1 Following an acceptable tune, a calibration standard, or standards, at mid-concentration containing all semivolatile analytes, and all required surrogates, must be analyzed every 12 hours during analysis.

11.7.2 For each compound in the continuing calibration standard, a minimum response factor given in Table 5 should be met. If the minimum response factors are not met, the system should be evaluated, and corrective action taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

11.7.3 If the percent difference or percent drift for each compound is less than or equal to 20%, the initial calibration is assumed to be valid and the analysis of samples may begin.

Calculate the percent drift using:

$$\%\ Drift = \frac{C_i - C_c}{C_i} \times 100$$

Where:

C_i = Compound standard concentration.
C_c = Measured concentration using selected quantitation method.

11.7.4 If the percent difference or percent drift for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large number of compounds that may be analyzed by this method, some compounds may fail to meet the ≤ 20% criteria. If no more than 20% of the compounds, included in the initial calibration, differ from their true concentration by 40%, the initial calibration is valid and no corrective action is necessary.

11.7.5 In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit.

11.7.6 Non-detected analytes can be reported from analyses when a CCV exhibit a positive bias (i.e., outside the upper control limit), no further documentation is required.

11.7.7 For situations when the CCV fails to meet the criterion in section 11.7.3, and a confirmed detection exceeds the MRL, the sample must be reanalyzed to ensure accurate quantification. If it is not possible to reanalyze the sample, the result must be reported as an estimated value.

11.7.8 The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as required. If the EICP area for any of the internal standards changes by a factor of two (50% to 200%) from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as appropriate. When corrective action is taken, reanalysis of samples analyzed while the system was malfunctioning is required. Update the reference spectra and retention times in the quantitation database for the instrument method or ID file. The initial calibration average RF or calibration curve is then used in the quantitation of subsequent analyses.

11.7.9 A blank (method blank, GPC blank, or solvent blank) should be analyzed after the CCV, or at any other time during the analytical shift, to prove the system is free of contaminants. If contaminants are found in a method blank or GPC blank, then a solvent blank should be analyzed to help isolate the source of contamination.

11.8 GC/MS Analysis

11.8.1 Evaluate FID screen, if performed, and make proper dilution (See FID screening SOP).

11.8.2 Spike the 1 mL extract obtained from sample preparation with 10 µL of the internal standard solution just prior to analysis. Use the same operating conditions as were used for initial calibration.

11.8.3 If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required concentration of each internal standard in the extracted volume. The diluted extract must be reanalyzed.

11.8.4 Store the extracts at -10°C or less, protected from light in vials equipped with unpierced Teflon lined septa. Extracts are archived in a freezer for 3 months after analysis in the instrument/date specific storage boxes.

12) Data Reduction and Reporting

12.1 Qualitative Analysis - The qualitative identification of compounds determined by this Qualitative Analysis - The qualitative identification of compounds determined by this procedure is based on retention time, and comparison of the sample mass spectrum with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the instrument and conditions used for the sample analysis. The characteristic ions from the reference mass spectrum are defined to be the ions monitored in the SIM mode and typically are the two or three ions of greatest relative intensity. Compounds are identified as present when the criteria below are met.

12.2 Qualitative Analysis - The qualitative identification of compounds determined by this procedure is based on retention time, and comparison of the sample mass spectrum with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the instrument and conditions used for the sample analysis. The characteristic ions from the reference mass spectrum are defined to be the ions monitored in the SIM mode and typically are the two or three ions of greatest relative intensity. Compounds are identified as present when the criteria below are met.

12.2.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

12.2.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

12.2.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

12.2.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the sum of the 2 peak heights. Otherwise, structural isomers are identified as isomeric pairs.

12.2.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When the ion relative intensities do not agree within 30% due to these types of matrix interferences, the analyst uses professional judgement in determining whether the analyte is or is not present. The analyst will take into account other knowledge of the sample, presence of other compounds, and other presumptive evidence available for review. Difficulties will be noted in the case narrative.

12.2.6 Evaluate internal standard areas in each sample. If the area in the sample is less than 50% or greater than 200% the area of in the CCV,

corrective action must be taken. Depending on the analysis, this corrective action may include reinjection or dilution of the extract followed by reinjection.

12.3 Tentatively identified compound (TIC) cannot be reported using this method.

12.4 Quantitation and Calculations

12.4.1 The GC/MS data stations, in current use, all use the H-P RTE Integrator to generate the raw data used to calculate the standards $\overline{RF_x}$ values, the sample amounts, and the spike values. The software does three passes through each data file. The first two identify and integrate each internal standard and surrogate. The third pass uses the time-drift information from the first two passes to search for all method analytes in the proper retention times and with the proper characteristic quantitation ions. When $\overline{RF_x}$ is used, calculate the extract concentration as follows:

$$C_{ex} = \frac{(Resp_x)(Amt_{ISTD})}{(Resp_{ISTD})(\overline{RF_x})}$$

Where: C_{ex} = the concentration in the sample extract (ppm);
$Resp_x$ = the peak area of the analytes of interest;
$Resp_{ISTD}$ = the peak area of the associated internal standard;
Amt_{ISTD} = the amount, in ppm, of internal standard added
$\overline{RF_x}$ = the average response from the initial calibration.

12.4.2 The concentration of analytes in the original sample is computed using the following equations:

Aqueous Samples: $Concentration\ (\mu g/L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$

Where Cex =Concentration in extract in µg/mL
Vf = Final volume of extract in mL
D = Dilution factor
Vs = Volume of sample extracted, liters

Non-aqueous Samples: $Concentration\ (mg/Kg) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$

Where Cex = Concentration in extract in µg/mL
Vf = Final volume of extract in mL
D = Dilution factor
W = Weight of sample extracted in grams.

12.5 Data Review

12.5.1 Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for *Laboratory Data Review Proocess* (ADM-DREV) for details.

12.6 Reporting

12.6.1 Reports are generated in the ALS LIMS by compiling the SMO login, sample prep database, instrument, date, and client-specified report requirements (when specified). This compilation is then transferred to a file which the Stealth reporting system uses to generate a report. The forms generated may be ALS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13) Contingencies for Handling Our-of-Control or Unacceptable Data

13.1 Refer to the SOP for *Non Conformance and Corrective Action Procedure* (ADM-NCAR) for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14) Method Performance

14.1 This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

14.2 The method detection limit (MDL) is and related method reporting limit(s) were established using the procedure described in SOP *Performing and Documenting Method Detection Limit Studies and Establishing Limits of Detection and Quantification*. Method Reporting Limits are established for this method based on MDL studies and as specified in the ALS Quality Assurance Manual.

15) Pollution Prevention and Waste Management

15.1 The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Lab Waste Management Plan.

16) Training

16.1 All analysts performing this analysis are required to read and understand this SOP.

16.2 Training is documented following the *Employee Training and New Employee Orientation* (ADM-TRAIN).

17) Method Modifications

17.1 There are no known modifications from the reference method.

18) References

18.1 *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry*, Method

8270D Revision 4, EPA Test Methods for Evaluating Solid Waste, SW-846, Update IV, February 2007..

18.2 DoD Quality Systems Manual for Environmental Laboratories current version.

18.3 TNI Standard, Volume 1- 2009, 2016.

18.4 ANSI/ISO/IEC 17005:2005/2017 American National Standard, General requirements for the competence of testing and calibration laboratories.

18.5 ALS-Kelso SOPS

18.5.1 Continuous Liquid-Liquid Extraction, EXT-3520
18.5.2 Solid Phase Extraction, EXT-3535
18.5.3 Automated Soxhlet Extraction, EXT-3541
18.5.4 Silica Gel Cleanup, EXT-3630
18.5.5 Gel Permeation Chromatography, EXT-3640

19) Changes Since the Last Revision

Summary of Revision Changes			
Rev. #	Effective Date	Document Editor	Description of Changes
9.0	12/2/2020	T CARON	Reformatted SOP to current ALS brand. Minor typographical, grammatical and format changes to improve readability not affecting content. Section1: Removed obsolete reference to the MDL found in Table 1. Section 6: Added Methylene Chloride exposure control plan. Updated safety references, where applicable throughout the document. Section 11.1: Subsampling and compositing performed by the Soil Prep Department, replacing General Chemistry. Section: 11.2.1.9 Removed obsolete Stealth reference Section 21: Added updated extraction benchsheet associated with SVM-AQ Revision 1.0. Section 7.1 & 7.2 updated collection and storage requirements

20) Attachments, Tables, and Appendices

20.1 Table 1A -Water and Soil Target Analytes.

20.2 Table 1B -Tissue Target Analytes.

20.3 Table 2 - Target Compounds and Corresponding Primary and secondary Quantitation Ions.

20.4 Table 3- Semi-Volatile Internal Standards with Corresponding Analytes assigned for Quantitation.

20.5 Table 4 - DFTPP Key Ions and Ion Abundance Criteria.

20.6 Table 4A - DFTPP Key Ions and Ion Abundance Criteria for 5973 and newer GC/MS.

20.7 Table 5 - Target Compounds and Recommended Minimum RT.

Table 1A
Water and Soil Target Analytes

ANALYTE	CAS No.	MATRIX	MRL	UNITS
Pentachlorophenol	87-86-5	Soil	200	µg/kg
1,4-Dioxane	123-91-1	Soil	5.0	µg/Kg
PBDE 100	189084-64-8	Soil	0.1	µg/Kg
PBDE 128	CASID30336	Soil	0.1	µg/Kg
PBDE 138	182677-30-1	Soil	0.1	µg/Kg
PBDE 153	68631-49-2	Soil	0.1	µg/Kg
PBDE 154	207122-15-4	Soil	0.1	µg/Kg
PBDE 17	147217-75-2	Soil	0.1	µg/Kg
PBDE 183	207122-16-5	Soil	0.1	µg/Kg
PBDE 190	CASID30338	Soil	0.1	µg/Kg
PBDE 203	CASID30339	Soil	0.1	µg/Kg
PBDE 206	63936-56-1	Soil	1.0	µg/Kg
PBDE 209	1163-19-5	Soil	1.0	µg/Kg
PBDE 28	41318-75-6	Soil	0.1	µg/Kg
PBDE 47	5436-43-1	Soil	0.1	µg/Kg
PBDE 66	189084-61-5	Soil	0.1	µg/Kg
PBDE 71	189084-62-6	Soil	0.1	µg/Kg
PBDE 85	182346-21-0	Soil	0.1	µg/Kg
PBDE 99	60348-60-9	Soil	0.1	µg/Kg
PBDE 47C13 (surr.)	CASID30341	Soil	NA	%
PBDE 99C13 (surr.)	CASID30342	Soil	NA	%
1,4-Dioxane-d_8 (surr.)	17647-74-4	Soil	NA	%
Pentachlorophenol	87-86-5	Water	1.0	µg/L
1,4-Dioxane	123-91-1	Water	1.0	µg/L
Di(proplyene glycol) butyl ether	29911-28-2	Water	0.25	µg/L
PBDE 100	189084-64-8	Water	1.0	ng/L
PBDE 128	CASID30336	Water	1.0	ng/L
PBDE 138	182677-30-1	Water	1.0	ng/L
PBDE 153	68631-49-2	Water	1.0	ng/L
PBDE 154	207122-15-4	Water	1.0	ng/L
PBDE 17	147217-75-2	Water	1.0	ng/L
PBDE 183	207122-16-5	Water	1.0	ng/L
PBDE 190	CASID30338	Water	1.0	ng/L
PBDE 203	CASID30339	Water	1.0	ng/L
PBDE 206	63936-56-1	Water	10	ng/L
PBDE 209	1163-19-5	Water	10	ng/L
PBDE 28	41318-75-6	Water	1.0	ng/L
PBDE 47	5436-43-1	Water	1.0	ng/L
PBDE 66	189084-61-5	Water	1.0	ng/L
PBDE 71	189084-62-6	Water	1.0	ng/L

Table 1A Continued

ANALYTE	CAS No.	MATRIX	MRL	UNITS
PBDE 85	182346-21-0	Water	1.0	ng/L
PBDE 99	60348-60-9	Water	1.0	ng/L
PBDE 99C13 (Surr.)	CASID30342	Water	NA	%
PBDE 47C13 (Surr.)	CASID30341	Water	NA	%
1,4-Dioxane-d_8 (Surr.)	17647-74-4	Water	NA	%

TABLE 1B
Tissue Target Analytes

ANALYTE	CAS No.	MATRIX	MRL	UNITS
1,2,4,5-Tetrachlorobenzene	95-94-3	Tissue	40	µg/Kg
1,2,4-Trichlorobenzene	120-82-1	Tissue	40	µg/Kg
1,2-Dichlorobenzene	95-50-1	Tissue	40	µg/Kg
1,3-Dichlorobenzene	541-73-1	Tissue	40	µg/Kg
1,4-Dichlorobenzene	106-46-7	Tissue	40	µg/Kg
2,3,4,6-Tetrachlorophenol	58-90-2	Tissue	40	µg/Kg
2,4,5-Trichlorophenol	95-95-4	Tissue	40	µg/Kg
2,4,6-Trichlorophenol	88-06-2	Tissue	40	µg/Kg
2,4-Dichlorophenol	120-83-2	Tissue	40	µg/Kg
2,4-Dimethylphenol	105-67-9	Tissue	40	µg/Kg
2,4-Dinitrophenol	51-28-5	Tissue	500	µg/Kg
2,4-Dinitrotoluene	121-14-2	Tissue	80	µg/Kg
2,6-Dinitrotoluene	606-20-2	Tissue	40	µg/Kg
2-Chloronaphthalene	91-58-7	Tissue	40	µg/Kg
2-Chlorophenol	95-57-8	Tissue	40	µg/Kg
2-Methyl-4,6-dinitrophenol	534-52-1	Tissue	500	µg/Kg
2-Methylnaphthalene	91-57-6	Tissue	40	µg/Kg
2-Methylphenol	95-48-7	Tissue	40	µg/Kg
2-Nitroaniline	88-74-4	Tissue	200	µg/Kg
2-Nitrophenol	88-75-5	Tissue	40	µg/Kg
3-Nitroaniline	99-09-2	Tissue	400	µg/Kg
4-Bromophenyl Phenyl Ether	101-55-3	Tissue	40	µg/Kg
4-Chloro-3-methylphenol	59-50-7	Tissue	40	µg/Kg
4-Chlorophenyl Phenyl Ether	7005-72-3	Tissue	40	µg/Kg
4-Methylphenol	106-44-5	Tissue	40	µg/Kg
4-Nitroaniline	100-01-6	Tissue	400	µg/Kg
4-Nitrophenol	100-02-7	Tissue	40	µg/Kg
Acenaphthene	83-32-9	Tissue	40	µg/Kg
Acenaphthylene	208-96-8	Tissue	40	µg/Kg
Acetophenone	98-86-2	Tissue	80	µg/Kg
Anthracene	120-12-7	Tissue	40	µg/Kg
Azobenzene	103-33-3	Tissue	40	µg/Kg
Benz(a)anthracene	56-55-3	Tissue	40	µg/Kg
Benzo(a)pyrene	50-32-8	Tissue	40	µg/Kg
Benzo(b)fluoranthene	205-99-2	Tissue	40	µg/Kg
Benzo(g,h,i)perylene	191-24-2	Tissue	40	µg/Kg
Benzo(k)fluoranthene	207-08-9	Tissue	40	µg/Kg
Benzyl Alcohol	100-51-6	Tissue	40	µg/Kg
Biphenyl	92-52-4	Tissue	40	µg/Kg
Bis(2-chloroethoxy)methane	111-91-1	Tissue	40	µg/Kg
Bis(2-chloroethyl) Ether	111-44-4	Tissue	40	µg/Kg
Bis(2-chloroisopropyl) Ether	39638-32-9	Tissue	40	µg/Kg
Bis(2-ethylhexyl) Phthalate	117-81-7	Tissue	200	µg/Kg
Butyl Benzyl Phthalate	85-68-7	Tissue	40	µg/Kg

TABLE 1B
Tissue Target Analytes (continued)

Caprolactam	105-60-2	Tissue	200	µg/Kg
Carbazole	86-74-8	Tissue	40	µg/Kg
Chrysene	218-01-9	Tissue	40	µg/Kg
Dibenz(a,h)anthracene	53-70-3	Tissue	40	µg/Kg
Dibenzofuran	132-64-9	Tissue	40	µg/Kg
Diethyl Phthalate	84-66-2	Tissue	40	µg/Kg
Dimethyl Phthalate	131-11-3	Tissue	40	µg/Kg
Di-n-butyl Phthalate	84-74-2	Tissue	100	µg/Kg
Di-n-octyl Phthalate	117-84-0	Tissue	40	µg/Kg
Fluoranthene	206-44-0	Tissue	40	µg/Kg
Fluorene	86-73-7	Tissue	40	µg/Kg
Hexachlorobenzene	118-74-1	Tissue	40	µg/Kg
Hexachlorobutadiene	87-68-3	Tissue	40	µg/Kg
Hexachloroethane	67-72-1	Tissue	40	µg/Kg
Indeno(1,2,3-cd)pyrene	193-39-5	Tissue	40	µg/Kg
Isophorone	78-59-1	Tissue	40	µg/Kg
Naphthalene	91-20-3	Tissue	40	µg/Kg
Nitrobenzene	98-95-3	Tissue	40	µg/Kg
N-Nitrosodimethylamine	62-75-9	Tissue	200	µg/Kg
N-Nitrosodi-n-propylamine	621-64-7	Tissue	40	µg/Kg
N-Nitrosodiphenylamine	86-30-6	Tissue	40	µg/Kg
Pentachlorophenol	87-86-5	Tissue	100	µg/Kg
Phenanthrene	85-01-8	Tissue	40	µg/Kg
Phenol	108-95-2	Tissue	100	µg/Kg
Pyrene	129-00-0	Tissue	40	µg/Kg
2,4,6-Tribromophenol (Surr.)	118-79-6	Tissue	NA	%
2-Fluorobiphenyl (Surr.)	321-60-8	Tissue	NA	%
2-Fluorophenol (Surr.)	367-12-4	Tissue	NA	%
Nitrobenzene-d_5 (Surr.)	4165-60-0	Tissue	NA	%
Phenol-d_6 (Surr.)	13127-88-3	Tissue	NA	%
Terphenyl-d_{14} (Surr.)	1718-51-0	Tissue	NA	%

TABLE 2
TARGET COMPOUNDS AND CORRESPONDING PRIMARY AND SECONDARY QUANTITATION IONS

Compound	Primary Ion	Secondary Ion
Phenol	94	66
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene-d_4 (I.S.)	152	150
1,4-Dichlorobenzene	146	148,111
1,2-Dichlorobenzene	146	79148,111
Benzyl alcohol	79	108,77
2-Methylphenol	108	77
Hexachloroethane	117	119,121
3-and 4-Methylphenol (coelute)	108	77
2,4-Dimethylphenol	122	107
1,2,4-Trichlorobenzene	180	182,145
Naphthalene-d_8 (I.S.)	136	68
Naphthalene	128	127
Benzoic acid	122	105
Hexachlorobutadiene	225	227,223
2-Methylnaphthalene	141	142
Acenaphthylene	152	153
Dimethyl phthalate	163	164,194
Acenaphthlene-d_{10} (I.S.)	164	162
Acenaphthene	154	153
Dibenzofuran	168	139
Fluorene	166	165
Diethyl phthalate	149	177
N-Nitrosodiphenylamine	169	168,167
Hexachlorobenzene	284	249,142
Pentachlorophenol	266	268
Phenanthrene-d_{10} (I.S.)	188	94
Phenanthrene	178	179
Anthracene	178	176
Di-n-butyl phthalate	149	150
Fluoranthene	202	203
Pyrene	202	203
Butyl Benzyl Phthalate	149	91,206
Benz (a) anthracene	228	226
Chrysene-d_{12} (I.S.)	240	236
Chrysene	228	226

TABLE 2
TARGET COMPOUNDS AND CORRESPONDING PRIMARY AND SECONDARY QUANTITATION IONS
(continued)

Bis (2-ethylhexyl) phthalate	149	167,279
Di-n-octyl phthalate	149	150,279
Benzo(b)fluoranthene	252	126
Benzo(k)fluoranthene	252	126
Benzo(a)pyrene	252	126
Perylene-d_{12} (I.S.)	264	260
Indeno (1,2,3-cd) pyrene	276	277
Dibenz (a, h) anthracene	278	279
Benzo(g,h,i)perylene	276	277
2-Fluorophenol (surr.)	112	64
Phenol-d_6 (surr.)	99	71
Nitrobenzene-d_5 (surr.)	82	128
2-Fluorobiphenyl (surr.)	172	171
2,4,6-Tribromophenol (surr.)	330	332
Terphenyl-d_{14} (surr.)	244	122
1,4-Dioxane	88	58,43
1,4-Dioxane-d_8	96	64,48
2-Methylphenol	151	166,152
4-Methylphenol	165	180,90
2-Chlorophenol	185	200,93
2,4-Dimethylphenol	194	179,105
Benzoic Acid	179	105,135,194
4-Chloro-3-methylphenol	199	214,201
2,4-Dichlorophenol	219	234,183
2-Nitrophenol	196	151,197
2,4,6-Trichlorophenol	253	255,270
2,4,5-Trichlorophenol	253	255,270
4-Nitrophenol	196	211,150
2,3,4,6-Tetrachlorophenol	289	287,304
Pentachlorophenol	323	325,338
2-Fluorophenol (surr.)	91	184
Phenol-d_5 (surr.)	156	171
2,4,6-Tribromophenol (surr.)	389	404,227

TABLE 3

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANITATION*

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Phenol	Benzoic Acid	Acenaphthene
Benzyl Alcohol	2,4-Dimethylphenol	Acenaphthylene
1,3-Dichlorobenzene	Hexachlorobutadiene	Dibenzofuran
1,4-Dichlorobenzene	Naphthalene	Diethyl phthalate
1,2-Dichlorobenzene	1,2,4-Trichlorobenzene	Dimethyl phthalate
2-Fluorophenol (surr.)	2-Methylnaphthalene	Fluorene
Hexachloroethane	2-Methylphenol	2-Fluorobiphenyl (surr.)
Phenol-d_6 (surr.)	4-Methylphenol	Hexachlorocyclopentadiene
Nitrobenzene-d_5 (surr.)		N-Nitrosodiphenylamine

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
Anthracene	Benzo(a) anthracene	Benzo(b)fluoranthene
Di-n-butyl phthalate	Bis (2-ethylhexyl)phthalate	Benzo(k)fluoranthene
Hexachlorobenzene	Butyl benzyl phthalate	Benzo(g,h,i)perylene
Pentachlorophenol	Chrysene	Dibenz(a,h)anthracene
Phenanthrene	Pyrene	Benzo(a)pyrene
2,4,6-Tribromophenol (surr.)	Terphenyl-d_{14} (surr.)	Indeno (1,2,3-ccd)pyrene
Fluoranthene		Di-n-octyl-phthalate

* Example list only

TABLE 4
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

TABLE 4A
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA
FOR **5973** and newer GC/MS Systems

Mass	Ion Abundance Criteria
51	10-80% of mass 198
68	0-2% of mass 69
70	0-2% of mass 69
127	10-80% of 198
197	0-2% of 198
198	30-100% of 442 (alternate base)
199	5-9% of 198
275	10-60% of 198
365	1-50% of 442
441	0.01-100% of 443
442	30-100% of 198 (alternate base)
443	15-24% of 442

Alternate tuning criteria (from Method 525.2 or CLP OLM04.2) may be used provided that method performance is not adversely affected and that method performance criteria is met. The criteria used must be the same for **all** ion abundance criteria checks associated with a given analysis. For example, initial calibration, continuing calibration(s), QC, and sample analyses for a given sample must all use the same criteria.

TABLE 5
TARGET COMPOUNDS AND RECOMMENDED MINIMUM RF

Compound	Min RF
Phenol	0.800
1,3-Dichlorobenzene	0.050
1,4-Dichlorobenzene-d_4 (I.S.)	-
1,4-Dichlorobenzene	0.050
1,2-Dichlorobenzene	0.050
Benzyl alcohol	0.050
2-Methylphenol	0.700
Hexachloroethane	0.050
3-and 4-Methylphenol (coelute)	0.600
2,4-Dimethylphenol	0.200
1,2,4-Trichlorobenzene	0.050
Naphthalene-d_8 (I.S.)	-
Naphthalene	0.700
Benzoic acid	0.050
Hexachlorobutadiene	0.010
2-Methylnaphthalene	0.400
Acenaphthylene	0.900
Dimethyl phthalate	0.01
Acenaphthlene-d_{10} (I.S.)	-
Acenaphthene	0.900
Dibenzofuran	0.800
Fluorene	0.900
Diethyl phthalate	0.010
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Pentachlorophenol	0.050
Phenanthrene-d_{10} (I.S.)	-
Phenanthrene	0.700
Anthracene	0.700
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl Benzyl Phthalate	0.010
Benz (a) anthracene	0.800
Chrysene-d_{12} (I.S.)	-
Chrysene	0.700

TABLE 5
TARGET COMPOUNDS AND RECOMMENDED MINIMUM RF
(continued)

Compound	Min RF
Bis (2-ethylhexyl) phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Perylene-d_{12} (I.S.)	-
Indeno (1,2,3-cd) pyrene	0.500
Dibenz (a, h) anthracene	0.400
Benzo(g,h,i)perylene	0.500
2-Fluorophenol (surr.)	-
Phenol-d_6 (surr.)	-
Nitrobenzene-d_5 (surr.)	-
2-Fluorobiphenyl (surr.)	-
2,4,6-Tribromophenol (surr.)	-
Terphenyl-d_{14} (surr.)	-
1,4-Dioxane	0.010
1,4-Dioxane-d_8 (surr.)	0.010
Derivatized Phenols	
2-Methylphenol	0.05
4-Methylphenol	0.05
2-Chlorophenol	0.05
2,4-Dimethylphenol	0.05
Benzoic Acid	0.05
4-Chloro-3-methylphenol	0.05
2,4-Dichlorophenol	0.05
2-Nitrophenol	0.05
2,4,6-Trichlorophenol	0.05
2,4,5-Trichlorophenol	0.05
4-Nitrophenol	0.05
2,3,4,6-Tetrachlorophenol	0.05
Pentachlorophenol	0.05
2-Fluorophenol (surr.)	0.05
Phenol-d_6 (surr.)	0.05
2,4,6-Tribromophenol (surr.)	0.05

Chlorinated Pesticides by GC/MS/MS

DOCUMENT ID: SVM-PESTMS2, REV 7.0

Approved By: Organics Manager, Jonathon Walter
Signature on File

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Signature on File

1) Scope & Applicability

1.1. This Standard Operating Procedure (SOP) describes the method used for trace analysis of various chlorinated pesticides by high performance gas chromatography/tandem mass spectrometry (HPGC/MS/MS). This SOP describes both the extraction and chromatographic procedures used to determine the target analytes. Table 1 indicates compounds that may be determined by this method and lists their method reporting limits (MRLs) in water and soil. The reported MRL may be adjusted to meet specific project requirements; however, the capability of achieving other reported MRLs must be demonstrated. Method Detection Limits (MDLs) which have been achieved are also given. The Method Detection Limits (MDLs) will vary depending on the instrument used and preparation method.

1.2. This procedure is used to determine the concentrations of chlorinated pesticides in water, soil, and tissue using HPGC/MS/MS. The procedure may be applied to other miscellaneous sample matrices providing that the analyst demonstrates the ability of the procedure to give data of acceptable quality in that matrix.

2) Summary of Procedure

2.1 This SOP describes HPGC/MS/MS conditions for the detection chlorinated pesticides. Prior to the use of this method, an appropriate sample preparation method must be used to recover the analytes of interest. Stable, isotopically labeled analogs of the analytes of interest are spiked into the samples before they are extracted. They are used to both assess the efficiency of the extraction and to quantitate the analytes of interest.

2.2 A 5.0 µL aliquot of the extract is injected into the gas chromatograph (GC). The compounds are separated on a fused silica capillary column. Compounds of interest are detected by a mass selective detector. Identification of the analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard, and by comparing the ratio of the two parent/daughter transitions acquired for each compound with the same ratio in the CCV Standard. Quantitative analysis is performed by using the authentic standard to produce a response factor and calibration curve, and using the calibration data to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume.

2.3 The following compounds may require special treatment when being determined by this method. DDT and Endrin are subject to thermal decomposition in the inlet of the gas chromatograph. Endosulfan I, Endosulfan II, Dieldrin, Endrin, Endrin aldehyde, and Endrin ketone produce relatively low response areas due to their fragmentation patterns.

3) Definitions

3.1 For environmental laboratory quality definitions, guidance on analytical calibration and sample batches, refer to the SOP for Sample Batches, ADM-BATCH.

4) Responsibilities

4.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in

accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

4.2 It is the responsibility of the department supervisor/manager to document analyst training .and method proficiency, as described in: *Employee Training and Orientation* (ADM-TRAIN).

5) Interferences

5.1 The GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation of the samples. Corrective action should be taken to eliminate the interferences.

5.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

5.3 The reporting limits for these analytes are in the low ppb to ppt level. Therefore, low level background contamination is a concern when conducting this method. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences.

6) Safety

6.1 Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in SDSs where available. Refer to the ALS Chemical Hygiene Plan and the appropriate SDSs prior to beginning this method.

6.2 This method uses Methylene Chloride, a known human carcinogen. Viton brand gloves should be used while rinsing, pouring or transferring the solvent.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Containers used to collect samples should be purchased pre-cleaned containers. Alternatively, containers used to collect samples for the determination of pesticides may be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or Teflon and have screw-top covers with Teflon liners. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

7.2 Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the

sample comes in contact with the sampler (e.g., if an automatic sampler is used), run reagent water through the sampler and use the rinsate as a field blank.

7.3 Water and soil samples must be iced or refrigerated at 0-6ºC from time of collection until extraction.

7.4 Tissue samples should be kept frozen until they are extracted to minimize decomposition.

7.5 Water samples must be extracted within 7 days and the extracts analyzed within 40 days following extraction. Solid, mixed-phase, semi-solid, tissue, and oily samples – Extract and analyze within 1 year of collection. If a sample is to be stored for more than 14 days, and results are to be reported in solids units, either hermetically seal the sample container or determine the moisture content upon receipt and immediately prior to analysis. Adjust the final concentration based on the original moisture content. Extracts are stored at <-10ºC and analyzed within 40 days of extraction.

8) Standards, Reagents, and Consumable Materials

8.1 Solvents: Acetone, methylene chloride, methanol, hexane, and other appropriate solvents. Solvents must be of sufficient purity to permit usage without lessening the accuracy of the determination or introducing interferences.

8.2 Stock Standard Solutions

8.2.1 Commercially prepared stock standards are typically used, and are available from several vendors. They must be A2LA or ISO9000 certified by the manufacturer. Standard concentrations can be verified by comparison versus an independently prepared standard. Alternatively, prepare stock standard solutions at a concentration of 1000 µg/ml by dissolving 0.0100 g of reference material in hexane, methylene chloride or other suitable solvent and diluting to volume in a 10mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard.

8.2.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at -10˚C and protect from light, or store as recommended by the manufacturer. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

8.2.3 Stock standard solutions must be replaced after one year, or sooner, if comparison with check standards or samples indicates a problem.

8.3 Instrument Internal Standard – 10 ul of a 10ug/ml Pyrene-d10 solution is added to each 1 ml sample extract undergoing analysis. The resulting concentration of the standard is 100ng/mL. Store at 0-6˚C when not being used. When using premixed certified solutions, store according to the manufacturer's recommendations. Other internal standards can be used as long as they do not interfere with the target analytes, and have similar retention times.

8.4 DDT/Endrin calibration standard. A hexane solution containing 20ng/mL of 4,4'-DDT, 4,4'-DDE, 4,4'-DDD, Endrin, Endrin Ketone, Endrin aldehyde, and 100ng/mL of the instrument internal standard.

8.5 DDT/ Endrin Standard - A hexane solution containing 20ng/ml of 4,4'-DDT, and Endrin, and 100ng/ml of the instrument internal standard. This is used to verify injection port inertness and GC column performance. Store at 0-6˚C when not being used, or store according to the manufacturer's recommendations.

8.6 Calibration Standards

8.6.1 A minimum of eight initial calibration standards should be prepared from stock solutions. One of the calibration standards should be at a concentration at or below the method reporting limit; the others should correspond to the range of concentrations found in real samples, but should not exceed the working range of the GC/MS system. At least one calibration standard must be at a concentration corresponding to a sample concentration meeting project-specific data quality objectives. Each standard should contain each analyte for detection by this method. Each 1 ml aliquot of calibration standards should be spiked with 10 µL of the internal standard solution prior to analysis. All calibration standards should be stored at 0-6˚C or less and should be freshly prepared once a year, or sooner if check standards indicate a problem.

8.6.2 The daily calibration standard (CCV) is prepared at a nominal 20 ng/ml concentration from stock solutions. The CCV is prepared weekly and can be stored at 0-6˚C, or as recommended by the manufacturer.

8.7 QC Standards

8.7.1 Labeled internal standards: These are isotopically labeled of the pesticides being analyzed. Prepare a working solution in acetone containing the following: Hexachlorobenzene13c6, Chlorpyrifos-d10, gamma.BHC-d6, Aldrin13c12, Heptachlor13c10, Endrin13c12, cis-Heptachlorepoxide13c10,Oxychlordane13c10, Octachlorostyrene13c8, Isodrin13c12, Methoxychlor-d6, Endrin ketone13c12, 4,4'-DDD-d4, 4,4'DDT-d4, and Mirex13c10 at the concentrations listed in Table 4. Aliquots of the solution are spiked into all extracted samples, blanks, and QC samples according to the extraction SOP used. All labeled internal standards should be stored at 4 ±2˚C or less. General expiration guidelines do not apply to labeled isotopes, but standard should be freshly prepared once a year, or sooner if problems are identified. To certify that the labeled internal standards are not degraded or concentrated from storage, and still suitable for use: prepare and analyze a standard containing the labeled standards and the unlabeled target analytes at 100 ng/ml. The ratio of: Labeled Compound Area/ Unlabeled Compound Area should be between 0.5-1.5.

8.7.2 Matrix Spike Standards: Prepare a working solution in methanol containing all analytes of interest ("full list spike") from the standard

analyte list (Table1) at 200ng/mL. Aliquots of the solution are spiked into the selected QC aliquots according to the extraction SOP used.

Note: The spiking level of Labeled internal standards and spike may need to be adjusted according to project requirements, if dilutions are expected due to high levels of extracted components, or if a lower calibration range is used.

9) Apparatus and Equipment

9.1 Gas Chromatograph/Mass Spectrometer System

9.1.1 Gas Chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for large volume cool splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source. Agilent 6890N equipped with the Agilent PTV injector recommended.

9.1.2 Column: ZB-Multiresidue-1 30 m x 0.25 mm ID x 0.25 µm film thickness silicone-coated fused-silica capillary column or equivalent. Recommended: Phenomenex #7HG-G016-11.

9.1.3 MS/MS System – Waters Micromass Quattro Micro GC, tandem mass spectrometer or equivalent.

9.1.4 Data System - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. (Waters MassLynx or equivalent) This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

9.1.5 Appropriate analytical balance (0.0001 g), volumetric flasks, syringes, vials, and bottles for standards preparation.

10) Preventative Maintenance

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

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

10.3 Gas Chromatograph

10.3.1 Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, O-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chromatographic performance. In some cases liners and seals may be cleaned and re-used.

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

10.3.3 Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

10.4 Mass Spectrometer

10.4.1 For units under service contract, certain maintenance is performed by instrument service staff, including pump oil changed, vacuuming boards, etc., as recommended by the manufacturer.

10.4.2 MS source cleaning should be performed as needed, depending on the performance of the unit. This may be done by the analyst or by instrument service staff.

11) Procedure

11.1 Sample Preparation

11.1.1 Water samples

11.1.1.1 Water samples are prepared using solid phase extraction (SPE) and EPA method 3510. Refer to the ALS SOP EXT-3510. Alternative extraction procedures such as EPA 3520 may be used for aqueous samples not suitable for 3510.

11.1.1.2 Perform the extraction on a 1000mL aliquot of sample. For heavily contaminated samples use a smaller volume or dilute the sample before extraction.

11.1.2 Soil, sediment, and solid samples are prepared using automated soxhlet extraction (SOP EXT-3541). The nominal sample size is 10g. Sample amounts may be decreased in the case of high-concentration waste samples.

11.1.3 Tissue samples are prepared using automated soxhlet extraction (SOP EXT-3541). The nominal sample size is 2.0g.

11.1.4 Product samples may not be analyzed by this method.

11.1.5 Extracts should be screened by GC/FID (SOP SOC-SCR). Cleanup by GPC, and carbon columns is mandatory for soil and tissue samples, and may be needed for contaminated water samples. All colored extracts must be cleaned with carbon columns. GPC is used to remove lipids from tissue extracts.

11.1.6 Extracts are solvent exchanged to hexane before they are taken to final volume. The final volume for all extracts is 1.0ml.

11.1.7 Following sample preparation, sample extracts are then transferred to the extract cold storage unit. Extracts must be analyzed within 40 days of extraction.

11.2 Analysis

11.2.1 The recommended GC/MS operating conditions are listed below. The GC conditions may be modified to accommodate specific instrument models and configurations.

GC Conditions

Injector	Agilent PTV
Mode	Solvent Vent, Ramped pressure
Injector liner:	Baffled, Restek Siltek Deactivated #21704-214.10
Injection volume:	5uL
Injection speed:	Slow
Post injection dwell:	1.0min
Carrier gas:	Helium @ 0.7 ml/min
Injector temperature:	20 – 320˚C, temperature programmed

GC Temperature program:

Initial temperature	65˚C for 2 min
Initial ramp	65 – 240˚C @ 20˚C per min
Second ramp	240 – 280˚C @ 6.1˚C per min
Third ramp	280 – 310˚C @ 15˚C per min
Final Hold	0.7 min or until Mirex or the last analyte elutes

Mass Spectrometer Conditions

Source temperature	225˚C
Detector interface temp:	290˚C
Trap Current	200
Collision Gas	Argon @ 2-3 × 10-3 torr
Tune file	dtrscan
Photomultiplier	650

11.2.2 Analysis sequence – Samples are analyzed in a set referred to as an

analysis sequence. The sequence begins with injection of a DFTPP Standard, followed by a DDT/Endrin standard and then the initial calibration standards. Once calibrated, a CCV is evaluated and extracts can be run. The sequence ends after 12 hours based on the DFTPP injection time

11.2.3 Tune the MS as needed to maximize sensitivity for MS/MS analysis. For the Micromass Quattro micro GC in EI mode the parameters will be similar to these:

Electron Energy	70
Trap	200
Repeller	0.5
Extraction Lens	5.0
Focus lens 1	40
Focus Lens 3	45
LM1 Resolution	10
HM1 Resolution	10
Ion Energy 1	0.9
Entrance	-1
Collision	2
Exit	+1
LM 2 Resolution	12
HM 2 Resolution	12
Ion Energy 2	1.0
Multiplier	650

11.2.4 Daily DFTPP Tune Verification

11.2.4.1 5ul of a 1.0ug/ml DFTPP standard is injected and analyzed in MRM mode to verify the MS/MS functioning of the instrument. The 442>198, 443>198, 198>110, 255>186, and the 127>77 transitions are monitored. Acceptance criteria are listed in table 8.

11.2.4.2 The analysis time for DFTPP is used to define the start of the 12-hour window in which all analyses must be performed. Once the instrument is tuned, all subsequent analyses of standards, samples, and QA/QC samples within the same 12-hour window must be analyzed using the identical mass spectrometer operating conditions.

11.2.5 Initial Calibration

NOTE: The calibration procedure(s) and options chosen must follow the ALS protocols. Any exceptions to the calibration procedures detailed in the ALS SOP for Calibration of Instruments for Organics Chromatographic Analyses are described as follows:

11.2.5.1 To assess column performance and injection port inertness; first analyze 5uL of the DDT/Endrin calibration standard by MRM and update

the calibration file. Then analyze the DDT/Endrin standard and quantitate it against the DDT/Endrin calibration.

11.2.5.2 Calculate the DDT/Endrin breakdown as follows:

%DDT = (4,4'-DDE + 4,4'-DDD)*100/(4,4'-DDE + 4,4'-DDD + 4,4'-DDT)

%Endrin = (endrin aldehye + endrin ketone)*100/(endrin aldehyde + endrin ketone + endrin)

11.2.5.3 DDT and Endrin degradation should not exceed 20%. If degradation of >20% is noted, the injection port may require cleaning. It may also be necessary to remove the first 15-30 cm of the GC column

11.2.5.4 The internal standards should permit most of the components of interest in the chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Refer to Table 6 for internal standards and corresponding analytes assigned for quantitation.

11.2.5.5 Analyze 5.0 µL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound (as indicated in Table 1). Calculate response factors (RFs) for each compound relative to one of the internal standards as follows:

RF = (AxCis)/(AisCx)

where:
Ax = Area of the characteristic ion for compound being measured.
Ais = Area of the characteristic ion for specific internal standard.
Cis = Concentration of the specific internal standard (ng/µL).
Cx = Concentration of the compound being measured (ng/µL).

11.2.5.6 The minimum RF for analytes and surrogates is 0.01

11.2.5.7 Calculate the %RSD for each analyte and internal standard.

$$\%RSD = \frac{SD}{\overline{RF}} \, x \, 100$$

where:
RSD = relative standard deviation.
$\overline{RF}$ = mean of initial RFs for a compound.
SD = standard deviation of average RFs for a compound.

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(RF_i - RF)^2}{N - 1}}$$

where:

RFi = RF for each of the calibration levels
N = Number of RF values (i.e., 8)

11.2.5.8 The % RSD should be less than 20% for each compound.

11.2.5.9 If the % RSD for any compound is ≤ 20%, linearity can be assumed over the calibration range, and the relative response factor for each analyte and surrogate is used.

11.2.5.10 If the %RSD for a compound is >20%, then alternative calibration models should be used. See the SOP (SOC-CAL) Calibration of Instruments for Organics Chromatographic Analysis for further guidance.

11.2.6 Independent Calibration Verification

11.2.6.1 Following initial calibration, analyze an ICV standard. The ICV solution must contain all analytes in the calibration standards. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. The ICV should quantitate within 75-125 % of the target concentration.

11.3 Continuing Calibration

11.3.1 First analyze the DDT/Endrin breakdown standard as detailed in 11.3.1 to 11.3.3.

11.3.2 A mid-level calibration verification standard, containing all pesticides, labeled, and recovery internal standards must be analyzed every 12 hours during analysis. Calculate the percent drift using:

$$\%\ Drift = \frac{C_t - C_m}{C_t} x\ 100$$

where:

Ct = Analyte standard concentration.
Cm = Measured concentration using selected quantitation method.

11.3.3 If the percent drift for each analyte is less than or equal to 25%, the initial calibration is assumed to be valid. If no source of the problem can be determined after corrective action has been taken, a new multipoint calibration must be generated. This criterion must be met

before sample analysis begins. If the % RSD for any analyte exceeds 25%, the analyst must determine if the response is sufficient to attain MRL for that analyte and any hits for that analyte must be rerun for quantitation.

11.3.4 The internal standard responses and retention times in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for the instrument internal standard changes by more than 30 seconds from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as required. If the EICP area for the PCB-52 13C12 internal standard changes by a factor of two (50% to 200%) from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as appropriate. When corrective action is taken, reanalysis of samples analyzed while the system was malfunctioning is required. Update retention times and transition ratios in the quantitation database for the instrument method or ID file. The initial calibration average RF or calibration curve is then used in the quantitation of subsequent analyses.

11.3.5 A blank (method blank, GPC blank, or solvent blank) should be analyzed after the CCV to prove the system is free of contaminants. If contaminants are found in a method blank or GPC blank, then a solvent blank should be ran to help isolate the source of contamination.

11.4 GC/MS Analysis

11.4.1 Evaluate FID screen and make proper dilution (See FID screening SOP).

11.4.2 Spike the 1 mL extract obtained from sample preparation with 10 µL of the instrument internal standard solution just prior to analysis. Use the same operating conditions as were used for initial calibration.

11.4.3 If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required 100ng/ml of the instrument internal standard in the extract volume. The diluted extract must be reanalyzed.

11.4.4 If a dilution of more than 10X is required; re-extract the sample using a smaller aliquot or re-fortify the sample for further dilution with internal standard/labeled compounds.

11.4.5 Store the extracts at -10°C or less, protected from light in vials equipped with un-pierced Teflon lined septa. Archive the extract in freezer for 3 months after analysis in the instrument/date specific storage boxes.

12) QA/QC Requirements

12.1 Initial Precision and Recovery Validation

12.1.1 The accuracy and precision of the procedure must be validated before analyses of samples begin, or whenever significant changes to the procedures have been made. To do this, four laboratory generated samples are spiked with the LCS spike solution, then prepared and analyzed. The acceptance criterion is the same as LCS criteria.

12.2 Method Detection Limits, Limits of Detection and Limits of Quantitation

12.2.1 Method detection limit (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ) must be determined before analysis of samples can begin. Refer to, Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification.

12.2.2 Calculate the average concentration found (x) in ng/L, and the standard deviation of the concentrations (s) in ng/L for each analyte. Calculate the MDL for each analyte.

12.3 Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. In general, these include:

12.3.1 Method blank - A method blank is extracted and analyzed with every batch of 20 or fewer samples to demonstrate that there are no method interferences. The method blank must demonstrate that interferences from the analytical and preparation steps minimized. No target analytes should be detected above the MRL in the method blank. For some project specific needs, exceptions may be noted and method blank results above the MRL may be reported.

12.3.2 An LCS or Laboratory Control Sample must be extracted and analyzed with every batch of 20 or fewer samples. The LCS is prepared by spiking a blank with the matrix spike solution, and going through the entire extraction and analysis.

12.3.2.1 Calculate percent recovery (%R) as follows:

$$\%R = X/TV \times 100$$

Where:

X = Concentration of the analyte recovered
TV = True value of amount spiked

12.3.2.2 If the LCS recovery for any control analyte fails acceptance limits, corrective action is required. If instrument corrective action is not applicable or ineffective, re-extraction of the associated samples is required. If any other analyte fails the advisory acceptance limits, the analyst must evaluate the impact on data quality and take any necessary corrective action, which may include re-extraction of the associated

samples. Project-specific requirements may require all compounds to be treated as control analytes, or dictate use of project acceptance criteria.

12.3.3 A matrix spike/duplicate matrix spike (MS/DMS) must be extracted and analyzed with every batch of 20 or fewer samples. The MS is prepared by spiking a sample aliquot with the matrix spike solution, and going through the entire extraction and analysis.

12.3.3.1 Calculate percent recovery (%R) as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where:

X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

12.3.3.2 Calculate Relative Percent Difference (RPD) as:

$$\%RPD = \frac{|R1 - R2|}{(R1 + R2)/2} \times 100$$

Where:

R1 = recovered concentration in the MS
R2 = recovered concentration in the DMS

12.3.4 If any internal standard recovery is outside acceptance criteria, the sample data must be closely evaluated for possible matrix interferences. If none are present, then corrective action must be taken. The sample should be re-analyzed if instrument factors (calibration, injection port) are suspected. If not, re-extraction and re-analysis is required, except in cases of high recovery and no positive hits in the sample for the analyte class represented by the particular surrogate.

12.4 Additional QA/QC measures include control charting of QC sample results.

13) Data Reduction and Reporting

13.1 Qualitative Analysis - The qualitative identification of compounds determined by this procedure is based on retention time, and the ratio of the quantitation transition and the qualifier transition. Compounds should be identified as present when the criteria below are met.

13.1.1 The intensities of the characteristic transitions of a compound maximize in the same scan or within one scan of each other. Selection of a peak

by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing transitions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

13.1.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

13.1.3 The ratio of the quantitation transition/qualifier transition agrees within 30% of the ratio of these transitions in the reference spectrum.

13.1.4 Structural isomers that produce very similar transitions should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the sum of the 2 peak heights. Otherwise, structural isomers are identified as isomeric pairs.

13.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing transitions contributed by more than one analyte. When gas chromatographic peaks appear to represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted transition current profiles of appropriate transitions can aid in the selection of spectra, and in qualitative identification. When analytes coelute, the identification criteria can be met, but each analyte spectrum will contain extraneous transitions contributed by the coeluting compound.

13.2 Quantitation and Calculations

13.2.1 The GC/MS/MS data stations, in current use, use the Waters MassLynx Integrator to generate the raw data used to calculate the standards $\overline{RF_x}$ values, the sample amounts, and the spike values. The software does three passes through each data file. The first two identify and integrate each internal standard and surrogate. The third pass uses the time-drift information from the first two passes to search for all method analytes in the proper retention times and with the proper characteristic quantitation transitions.

When $\overline{RF_x}$ is used, calculate the extract concentration as follows:

$$C_{ex} = \frac{(Resp_x)(Amt_{ISTD})}{(Resp_{ISTD})(\overline{RF_x})}$$

Where:

Cex = the concentration in the sample extract (ppm);
Respx = the peak area of the analytes of interest;

RespISTD = the peak area of the associated internal standard;
AmtISTD = the amount, in ppm, of internal standard added
= the average response from the initial calibration.

13.2.2 The concentration of analytes in the original sample is computed using the following equations:

Aqueous Samples:

When $\overline{RF_x}$ is used, calculate the extract concentration as follows:

$$C_{ex} = \frac{(Resp_x)(Amt_{ISTD})}{(Resp_{ISTD})(\overline{RF_x})}$$

Where:
Cex = Concentration in extract in µg/mL
Vf = Final volume of extract in mL
D = Dilution factor
Vs = Volume of sample extracted, liters

Non-aqueous Samples:

$$Concentration\ (mg/Kg) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$$

Where Cex = Concentration in extract in µg/mL
Vf = Final volume of extract in mL
D = Dilution factor
W = Weight of sample extracted in grams.

13.2.2.1 If the area of the quantitation transition for any of the pesticides exceeds the calibration range, dilute and reanalyze the extract. Add additional recovery internal standard to maintain its concentration at 100ng/ml.

13.2.2.2 If a dilution greater than 10X is required; re-extract the sample using a smaller aliquot.

13.2.2.3 If the recovery of any of the labeled internal standards is outside of the limits, extract and analyze a smaller aliquot of the sample or refortify if due to dilution. Adjust the MRL/MDL to account for this dilution.

13.3 Data Review

13.3.1 Following primary data interpretation and calculations, all data is

reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for Laboratory Data Review Process for details.

13.4 Reporting

13.4.1 Reports are generated using the STEALTH Data Reporting System which compiles the SMO login information and Enviroquant data. This compilation is then transferred to a file, which STEALTH uses to generate a report. The forms generated may be ALS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.4.2 As an alternative, reports are generated using Excel© templates located in R:\SVM\forms. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The detected analytes, surrogate and matrix spikes are then transferred, by hand or electronically, to the templates.

14) Contingencies for Handling Our-of-Control or Unacceptable Data

14.1 Refer to the SOP for *Nonconformance and Corrective Action Procedures* (ADM-NCAR) for procedures for corrective action.

14.2 Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

15) Method Performance

15.1 This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2 The method detection limit (MDL) is and related method reporting limit(s) were established using the procedure described in SOP *Performing and Documenting Method Detection Limit Studies and Establishing Limits of Detection and Quantification*). Method Reporting Limits are established for this method based on MDL studies and as specified in the ALS Quality Assurance Manual.

16) Pollution Prevention and Waste Management

16.1 The laboratory will comply with all Federal, State and local regulations governing waste restrictions as specified in the ALS Lab Waste Management Plan.

17) Training

17.1 All analysts performing this analysis are required to read and understand this SOP.

17.2 Training is documented following the Employee Training and New Employee Orientation (ADM-TRAIN).

18) Method Modifications

18.1 N/A.

19) References

19.1 *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry, Method 8270C*, EPA Test Methods for Evaluating Solid Waste, SW-846, Final Update III, December 1996.

19.2 *Method 1699*, USEPA: *Pesticides in Water, Soil, Biosolids, and Tissue by HRGC/HRMS,* Office of Water, Office of Science and Technology, Engineering and Analysis Division. December 2007.

19.3 DoD Quality Systems Manual for Environmental Laboratories, current version.

19.4 TNI Standard, Volume 1, 2009 & 2016.

19.5 ISO/IEC 17025: 2017 American National Standard, General requirements for the competence of testing and calibration laboratories.

20) Changes Since the Last Revision

Rev. #	Effective Date	Document Editor	Description of Changes
7.0	4/22/2021	T.Caron	Admin Changes not affecting technical content. Documented date of annual SOP Review, updated SOP signatories; boiler plate standard paragraphs and references. Section 7.5: New content added regarding solid, mixed-phase, semi-solid, tissue and oily samples. Section 12.3.2.2: LCS Recovery/acceptance has been updated. Section 12.3.3.3: MS/DMS acceptance/recovery has been removed. Sections 13.2.2.3&12.3.4: Internal Standard Recovery/acceptance has been updated. Table 5: QC Acceptance Criteria has been removed. • Procedural Change Request submitted by J. Walter, on 4/16/2021. • Procedural Change Request submitted by J. Walter, on 4/21/2021.

21) Attachments, Tables, and Appendices

21.1 Table 1: ORGANOCHLORINE PESTICIDES- STANDARD ANALYTE LIST.

21.2 Table 2 – Labeled Compound Spiking Solution.

21.3 Table 3; Recovery Internal Standard Spiking Solution.

21.4 Table 4: Concentration of Calibration Solutions (ng/ml).

21.5 Table 5: Quantitation References.

21.6 Table 6: Target Compound Transitions and Collision Energy.

21.7 Table 7: DFTPP Transitions, Collision Energy, Acceptance Criteria.

Table 1

ORGANOCHLORINE PESTICIDES- STANDARD ANALYTE LIST

	Water		Soil		Tissue	
Compound	**MDL (ng/L)**	**MRL (ng/L)**	**MDL(µg/Kg)**	**MRL (µg/Kg)**	**MDL(µg/Kg)**	**MRL (µg/Kg)**
alpha-BHC	0.27	0.50	0.0069	0.050	0.26	1.0
gamma-BHC	0.15	0.50	0.011	0.050	17	0.50
beta-BHC	0.31	0.50	0.012	0.050	0.40	1.0
delta-BHC	0.19	0.50	0.011	0.050	0.28	1.0
Hexachlorobenzene	0.13	0.50	0.015	0.050	0.70	2.5
Heptachlor	0.17	1.0	0.0091	0.10	0.090	0.50
Chlorpyrifos	0.17	0.50	0.0072	0.050	0.13	0.5
Aldrin	0.47	1.0	0.012	0.10	0.22	1.0
Octachlorostyrene	0.28	1.0	0.017	0.10	0.16	0.50
Isodrin	1.1	2.0	0.029	0.20	0.23	1.0
Oxychlordane	0.72	2.0	0.092	0.20	0.77	2.50
Heptachlor epoxide	0.47	1.0	0.030	0.10	0.061	0.50
2,4'-DDE	0.19	0.50	0.0069	0.050	0.42	1.0
gamma-Chlordane	0.20	0.5	0.0081	0.050	0.13	0.50
alpha-Chlordane	0.15	0.50	0.0066	0.050	0.12	0.50
trans-Nonachlor	0.24	0.50	0.014	0.050	0.094	0.50
Endosulfan I	1.2	2.0	0.050	0.20	0.42	1.0
4,4'-DDE	0.13	0.50	0.0055	0.050	0.70	2.5
2,4'-DDD	0.11	0.50	0.0094	0.050	0.31	1.0
Dieldrin	2.3	5.0	0.098	0.50	0.22	1.0
Endrin	0.84	2.0	0.045	0.20	0.45	1.0
2,4'-DDT	0.13	0.50	0.0060	0.050	0.46	1.0
cis-Nonachlor	0.31	0.5	0.015	0.20	0.13	0.50
4,4'-DDD	0.20	0.50	0.015	0.050	013	1.0
Endosulfan II	0.51	1.0	0.043	0.10	0.35	1.0
Endrin aldehyde	0.67	1.0	0.051	0.10	56	1.0
4,4'-DDT	0.23	0.50	0.014	0.050	0.35	1.0

	Water		Soil		Tissue	
Compound	**MDL (ng/L)**	**MRL (ng/L)**	**MDL(µg/Kg)**	**MRL (µg/Kg)**	**MDL(µg/Kg)**	**MRL (µg/Kg)**
Endosulfan sulfate	0.12	0.50	0.017	0.050	0.22	1.0
Methoxychlor	0.16	0.50	0.018	0.050	0.15	0.50
Endrin Ketone	0.70	1.0	0.036	0.10	0.16	0.50
Mirex	0.12	1.0	0.029	0.10	0.11	0.50

Table 2:
Labeled Compound Spiking Solution

Compound Name	**Spiking solution conc. (ng/ml)**	**Spike amount (ng) 100ul spike volume**
g-BHC-d6	200	20
Hexachlorobenzene13C6	200	20
Heptachlor-13C10	200	20
Chlorpyrifos-d10	200	20
Aldrin-13C12	200	20
Octachlorostyrene-13C8	200	20
Isodrin-13C12	200	20
Oxychlordane-13C10	200	20
Heptachlorepox13C10	200	20
Endrin-13C12	200	20
4,4'-DDD-d4	50	5
4,4'-DDT-d4	50	5
Methoxychlor-d6	200	20
Endrin ketone-13C12	200	20
Mirex-13C10	50	5

Table 3
: Recovery Internal Standard Spiking Solution

Compound Name	**Spiking solution conc. (ug/ml)**	**Spike amount (ng) 100ul spike volume**
Pyrene-d10	10	100

Table 4
Concentration of Calibration Solutions (ng/ml)

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6 CCV	CS-7	CS-8	CS-9
Pyrene-d10	100	100	100	100	100	100	100	100	100
g-BHC-d6	20	20	20	20	20	20	20	20	20
Hexachlorobenzene13C6	20	20	20	20	20	20	20	20	20
Heptachlor-13C10	20	20	20	20	20	20	20	20	20
Chlorpyrifos-d10	20	20	20	20	20	20	20	20	20
Aldrin-13C12	20	20	20	20	20	20	20	20	20
Octachlorostyrene-13C8	20	20	20	20	20	20	20	20	20
Isodrin-13C12	20	20	20	20	20	20	20	20	20
Oxychlordane-13C10	20	20	20	20	20	20	20	20	20
Heptachlorepox13C10	20	20	20	20	20	20	20	20	20
Endrin-13C12	20	20	20	20	20	20	20	20	20
4,4'-DDD-d4	5	5	5	5	5	5	5	5	5
4,4'-DDT-d4	5	5	5	5	5	5	5	5	5
Methoxychlor-d6	20	20	20	20	20	20	20	20	20
Endrin ketone-13C12	20	20	20	20	20	20	20	20	20
Mirex-13C10	5	5	5	5	5	5	5	5	5
alpha-BHC	0.5	1	2	5	10	20	50	70	100
Gamma-BHC	0.5	1	2	5	10	20	50	70	100
Beta-BHC	0.5	1	2	5	10	20	50	70	100
Delta-BHC	0.5	1	2	5	10	20	50	70	100
Hexachlorobenzene	0.5	1	2	5	10	20	50	70	100
Heptachlor	0.5	1	2	5	10	20	50	70	100
Chlorpyrifos	0.5	1	2	5	10	20	50	70	100
Aldrin	0.5	1	2	5	10	20	50	70	100
Octachlorostyrene	0.5	1	2	5	10	20	50	70	100
Isodrin	0.5	1	2	5	10	20	50	70	100
Oxychlordane	0.5	1	2	5	10	20	50	70	100
Heptachlor epoxide	0.5	1	2	5	10	20	50	70	100
2,4'-DDE	0.5	1	2	5	10	20	50	70	100
Gamma-Chlordane	0.5	1	2	5	10	20	50	70	100
Alpha-Chlordane	0.5	1	2	5	10	20	50	70	100
Trans-Nonachlor	0.5	1	2	5	10	20	50	70	100
EndosulfanI	0.5	1	2	5	10	20	50	70	100

Table 4
Concentration of Calibration Solutions (ng/ml) continued

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6 CCV	CS-7	CS-8	CS-9
4,4'-DDE	0.5	1	2	5	10	20	50	70	100
2,4'-DDD	0.5	1	2	5	10	20	50	70	100
Dieldrin	0.5	1	2	5	10	20	50	70	100
Endrin	0.5	1	2	5	10	20	50	70	100
2,4'-DDT	0.5	1	2	5	10	20	50	70	100
Cis-Nonachlor	0.5	1	2	5	10	20	50	70	100
4,4'-DDD	0.5	1	2	5	10	20	50	70	100
EndosulfanII	0.5	1	2	5	10	20	50	70	100
Endrin aldehyde	0.5	1	2	5	10	20	50	70	100
4,4'-DDT	0.5	1	2	5	10	20	50	70	100
Endosulfan sulfate	0.5	1	2	5	10	20	50	70	100
Methoxychlor	0.5	1	2	5	10	20	50	70	100
Endrin ketone	0.5	1	2	5	10	20	50	70	100
Mirex	0.5	1	2	5	10	20	50	70	100

LABELED PESTICIDE STANDARDS

Isotopically labeled pesticide and PCB standards are obtained from Cambridge Isotope Laboratories, C/D/N Isotopes, and Dr Erhenstorfer Laboratories.

CALIBRATION

The multipoint calibration is prepared from: AccuStandard catalog # M-8081-SC, M-8080-OP, P-297S, P-184S, P-331S-H, P-066S, P-094S, P-471S, APP-9-112 (or equivalent from other vendors*)

ICV

The ICV standard is prepared from the following: Ultra Scientific catalog # PPM-808C-1, PPM-828-1, PST-480C100A01, PST-590C100A01, PST-120C100A01, PST-3185C100A01, PP-430-1, PST-2705M100A01, PST-1200C100A01, PST-720C100A01

CCV

Use the same solutions that were used for the calibration curve
Prepare 1 ml of 20 ng/ml CCV standard, place in autosampler vial and cap. Expiration date is 1 week after CCV was prepared.

RECAP AND STORE IMMEDIATELY AFTER INJECTING

Store remaining stock solutions in 1 ml amber vial. Expiration date is one year after ampule is opened or the manufacturers expiration date which ever is first Order when down to one unopened ampule.

* Vendor must be A2LA and/or ISO9000 certified.

Table 5
Quantitation References

Compound		Quantitation Reference
Pyrene-d10		None
g-BHC-d6		Pyrene-d10
Hexachlorobenzene13C6		Pyrene-d10
Heptachlor-13C10		Pyrene-d10
Chlorpyrifos-d10		Pyrene-d10
Aldrin-13C12		Pyrene-d10
Octachlorostyrene-13C8		Pyrene-d10
Isodrin-13C12		Pyrene-d10
Oxychlordane-13C10		Pyrene-d10
Heptachlorepox13C10		Pyrene-d10
Endrin-13C12		Pyrene-d10
4,4'-DDD-d4		Pyrene-d10
4,4'-DDT-d4		Pyrene-d10
Methoxychlor-d6		Pyrene-d10
Endrin ketone-13C12		Pyrene-d10
Mirex-13C10		Pyrene-d10
alpha-BHC		g-BHC-d6
Gamma-BHC		g-BHC-d6
Beta-BHC		g-BHC-d6
Delta-BHC		g-BHC-d6
Hexachlorobenzene		Hexachlorobenzene13C6
Heptachlor		Heptachlor-13C10
Chlorpyrifos		Chlorpyrifos-d10
Aldrin		Aldrin-13C12
Octachlorostyrene		Octachlorostyrene-13C8
Isodrin		Isodrin-13C12
Oxychlordane		Oxychlordane-13C10
Heptachlor epoxide		Heptachlorepox13C10
2,4'-DDE		4,4'-DDD-d4
Gamma-Chlordane		Oxychlordane-13C10
Alpha-Chlordane		Oxychlordane-13C10
Trans-Nonachlor		Oxychlordane-13C10
EndosulfanI		Endrin-13C12
4,4'-DDE		4,4'-DDD-d4
2,4'-DDD		4,4'-DDD-d4
Dieldrin		Endrin-13C12
Endrin		Endrin-13C12
2,4'-DDT		4,4'-DDT-d4
Cis-Nonachlor		Oxychlordane-13C10
4,4'-DDD		4,4'-DDD-d4
EndosulfanII		Endrin-13C12

Endrin aldehyde		Endrin-13C12
4,4'-DDT		4,4'-DDT-d4
Endosulfan sulfate		Endrin-13C12
Methoxychlor		Methoxychlor-d14
Endrin ketone		Endrin ketone-13C12
Mirex		Mirex-13C10

Table 6
Target Compound Transitions and Collision Energy

Compound		Ionization Mode	Quan Transition Trace	Qual Transition Trace	Collision energy (v)
					Quan|Qual
Pyrene-d10		EI+	212>208	212>184	20|20
g-BHC-d6		EI+	223.9>187	184.9>150	10|20
Hexachlorobenzene13C6		EI+	289.8>254.8	254.8>219.8	20|20
Heptachlor-13C10		EI+	276.8>241.8	279>244	15|15
Chlorpyrifos-d10		EI+	324>259.8	199.9>171.9	15|20
Aldrin-13C12		EI+	269.8>200	304.8>269.9	30|20
Octachlorostyrene-13C8		EI+	385.7>315.8	315.8>245.8	25|30
Isodrin-13C12		EI+	199.9>130.1	269.8>200.2	30|30
Oxychlordane-13C10		EI+	154>125	190>154	5|5
Heptachlorepox13C10		EI+	362.8>270	362.8>291.8	15|15
Endrin-13C12		EI+	269.8>200	289.9>254	35|10
4,4'-DDD-d4		EI+	243>173.1	243>208	25|10
4,4'-DDT-d4		EI+	243>173.1	243>208	25|10
Methoxychlor-d6		EI+	233>141	233>169	30|20
Endrin ketone-13C12		EI+	327.9>291.8	327.9>255	10|20
Mirex-13C10		EI+	276.8>241.8	279>244	15|20
alpha-BHC		EI+	218.9>182.9	183>147	10|20
Gamma-BHC		EI+	218.9>182.9	183>147	10|20
Beta-BHC		EI+	218.9>182.9	183>147	10|20
Delta-BHC		EI+	218.9>182.9	183>147	10|20
Hexachlorobenzene		EI+	283.8>248.8	248.8>213.9	20|20
Heptachlor		EI+	271.8>236.8	269.8>234.8	15|15
Chlorpyrifos		EI+	314>257.8	196.9>168.9	15|20
Aldrin		EI+	262.9>192.9	293>222	30|20
Octachlorostyrene		EI+	379.7>309.4	307.8>237.8	25|30
Isodrin		EI+	192.9>123	262.9>192.8	30|30
Oxychlordane		EI+	149>121	185>149	5|5
Heptachlor epoxide		EI+	352.8>262.8	352.8>281.8	15|15
2,4'-DDE		EI+	246>176	317.9>248	30|18
Gamma-Chlordane		EI+	373>265.8	373>300.8	20|10
Alpha-Chlordane		EI+	373>265.8	373>300.8	20|10
Trans-Nonachlor		EI+	408.8>299.8	410.9>301.9	20|20
EndosulfanI		EI+	195>159	241>170	6|20
4,4'-DDE		EI+	246>176	317.9>248	30|18
2,4'-DDD		EI+	235>165	235>199	25|10
Dieldrin		EI+	262.8>192.9	279>243	35|10
Endrin		EI+	262.8>192.9	279>243	35|10

Target Compound Transitions and Collision Energy (continued)

Compounds		Ionization Mode	Quan Transition Trace	Qual Transition Trace	Collision energy (v)
2,4'-DDT		EI+	235>165	235>199	25|10
Cis-Nonachlor		EI+	408.8>299.8	410.9>301.9	20|20
4,4'-DDD		EI+	235>165	235>199	25|10
EndosulfanII		EI+	195>159	241>206	6|10
Endrin aldehyde		EI+	344.8>280.7	344.8>244.9	10|20
4,4'-DDT		EI+	235>165	235>199	25|10
Endosulfan sulfate		EI+	271.8>236.7	386.8>288.8	15|8
Methoxychlor		EI+	227>141	227>169.1	30|20
Endrin ketone		EI+	316.9>280.8	316.9>244.9	10|20
Mirex		EI+	271.8>236.8	269.8>234.8	15|15

Table 7
DFTPP Transitions, Collision Energy, Acceptance Criteria

Transitions	Range	Ionization Mode	Transition	Collision energy (v)
DFTPP1	1.000 (base)	EI+	442>198	14
DFTPP2	4.557-11.374	EI+	443>198	14
DFTPP3	1.380-3.834	EI+	198>110	14
DFTPP4	1.545-5.505	EI+	255>186	14
DFTPP5	10.211-34.819	EI+	127>77	20

Note: Relative abundance defined as: abundance (DFTPP1)/abundance (DFTPPX). Criteria

Attachment 2
Data Quality Evaluation Report Template

Title

Introduction

The objective of this data quality evaluation (DQE) report is to assess the data quality of analytical results samples collected at the Willbridge Cove Project Area.

This report is intended as a general data quality assessment designed to summarize data issues.

Analytical Data and Field QA/QC Samples

This DQE report covers XX normal samples, XX field duplicates (FD and XX trip blanks (TB). A list of samples and collection dates is included in Attachment A at the end of this DQE report. Samples were collected between September XX and October XX, 2021. These sample results were reported as XX sample delivery groups (SDG) listed in Table 1. The analyses were subcontracted to XX Laboratory.

Table 1 - Sample Delivery Groups

XXne methods were used to analyze the environmental samples. One or more of the samples were analyzed for the following analytes/methods presented in Table 2.

Table 2 - Analytical Parameters	
Parameter	**Method**

The data validation included a review of the following items: (1) the chain-of-custody (CoC) documentation; (2) holding-time compliance; (3) the required field and laboratory quality control (QC) samples; (4) flagging for method blanks; (5) laboratory control sample/laboratory control sample duplicates (LCS/LCSD); (6) matrix spike/matrix spike duplicate samples (MS/MSD); and, (7) surrogate recoveries.

Data flags are assigned according to the QAPP. Multiple flags are routinely applied to specific sample method/matrix/analyte combinations, but there will be only one final flag. A final flag is applied to the data and is the most conservative of the applied validation flags. The final flag also includes blank sample impacts.

The data flags are those listed in the QAPP and are defined below:

- J = Analyte was present but reported value may not be accurate or precise.
- J+ = Analyte was present but reported value may not be accurate or precise, high bias.
- J- = Analyte was present but reported value may not be accurate or precise, low bias.
- R = This result has been rejected.
- U = This analyte was analyzed for but not detected at the specified detection limit.
- UJ = The analyte was not detected above the detection limit objective; however, the reported detection limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

Findings

The overall summaries of the data validation findings are contained in the following sections and Table 3.

Calibration

Holding Times

Method Blanks

Field Duplicates

Laboratory Control Samples

Laboratory Duplicates

Matrix Spike Samples

Surrogates

Total and Dissolved Metals

Chain of Custody

Overall Assessment

Table 3 – Validation Flags

Attachment A – Samples Associated with DQE

Attachment 3
Field Audit Checklist

Sample containers and preservatives; sample collection and identification; sample custody, handling, and shipping procedures					
Item No.	**SOP/QAPP Reference**	**Requirement Description**	**Documentation Required for Verification**	**Requirement Met? (Yes, No, Partial, NA)**	**Verification / Findings / Comments**
1	SOP-7	Samples received from the laboratory pre-labeled and with the preservative (where needed).	Observe sample containers in cooler/box and verify all bottles are received, labels are complete, and preservative is present as needed		
2	SOP-7	Sample identification and date and time of sampling will be entered on the label immediately after collection.	Observe sample containers in cooler/box and verify label completeness		
3	SOP-7	When samples are required to be stored at 4 degrees Celsius or less, generous amounts of bagged ice will be packed with the samples.	Verify by observation of coolers		
4	SOP-7	Samples will be packed for shipment as follows: · Sample bottles will be sealed in individual plastic bags. · All samples will be transported inside appropriate containers. · All 40milliliter (mL) volatile organic analysis bottles will be placed in blocks of foam or in plastic mesh sleeves (if collected) · All other glass bottles will be placed in plastic mesh sleeves to prevent glass to glass contact. · Bagged ice and/or foam blocks will be used to separate glass bottles. · The original COC form will be packed inside the shipping container. · The containers will be taped shut and sealed with signed custody seals. · Samples that are known or suspected to be highly contaminated (based on field screening data or observation) will be packaged and shipped separately from other samples. · Laboratories will be notified of any known or suspected highly contaminated samples. · These samples will be stored separately from less contaminated samples to minimize the potential for contamination.	Verify by procedure is followed		
5	SOP-7	Any samples defined as hazardous materials will be packaged and shipped in accordance with requirements defined in the Code of Federal Regulations (CFR) (40 CFR 49, Chapter 1, Part 171).	Verify via shipping records		
6	QAPP Section 4.2.1	At a minimum, the chain-of-custody form must contain: · Site name · PM, Project Chemist, and Data Manager names, telephone numbers, and email addresses · Unique sample identification · Date and time of sample collection · Source of sample (including name, location, sample type, and matrix) · Number of containers · Designation of matrix spike and matrix spike duplicate (MS/MSD) · Preservative used · Analyses required · Name of sampler · Custody transfer signatures and dates and times of sample transfer from the field to transporters and to the laboratories · Bill of lading or transporter tracking number (if applicable) · Turnaround time · Lab name, address, and contact information · Laboratory purchase order number · Project number	Verify completeness of COC forms		

Appendix D
Health and Safety Plan –
with Emergency Response Plan

Jacobs

Portland Harbor Willbridge Cove Project Area

Health and Safety Plan
Emergency Response Plan

Final

June 10, 2021

Willbridge Cove Group
Willbridge Cove Project Area Remedial Design

Approval

This site-specific Health and Safety Plan (HSP) has been written for use by Jacobs Engineering Group Inc. (Jacobs) only. Jacobs claims no responsibility for its use by others unless that use has been specified and defined in project or contract documents. The plan is written for the specific site conditions and identified scope(s) of work and must be amended if those conditions or scope(s) of work change.

By approving this HSP, the Responsible Health and Safety Manager (RHSM) certifies that the personal protective equipment has been selected based on the project-specific hazard assessment.

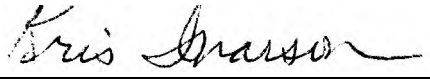
Original Plan

RHSM Approval:

November 20, 2020

John Culley/SPK, CIH — Date

Field Operations Manager Approval:



Kris Ivanson — June 10, 2021

Revisions

Revisions Made By: **Date:**

Description of Revisions to Plan:

Revisions Approved By:

Date

Emergency Contacts

Medical Emergency – 911 **Facility Medical Response #:** **Local Ambulance #:**	**WorkCare – Injury Reporting** 888-449-7787 For non-life-threatening injuries, call your Supervisor, Health and Safety Manager (HSM), and then WorkCare. See Hospital and Occupational Clinic.
Local Medical Clinic	**Global Environmental Solutions – Health, Safety & Environment** Name: Mark Orman/KNV Phone: 414-712-4138
Fire/Spill Emergency – 911 **Facility Fire Response #:** **Local Fire Dept #:**	**Jacobs Responsible Health and Safety Manager (RHSM)** Name: John Culley/SPK Phone: 206-660-3367
Security & Police – 911 **Facility Security #:** **Local Police #:**	**Jacobs Human Resources (HR) Department** Contact your line of business HR department
Utilities Emergency Phone Numbers Water: Gas: Electric:	**Jacobs Worker's Compensation:** Contact HR department to have form completed
Safety Liaison - Hazardous Waste (SL-HW) Name: Shannon Bartow/PDX Phone: (b) (6) Name: Laura Tochko/PDX Phone: (b) (6) Name: Patrick Terhune/PDX Phone: (b) (6) 68	**Media Inquiries Corporate Strategic Communications** Name: Lorrie Paul Crum Phone: (b) (6)
Project Manager Name: Marilyn Gauthier Phone: (b) (6)	**Automobile Accidents** See Attachment 4
Federal Express Dangerous Goods Shipping Phone: 800-238-5355	**CHEMTEL (hazardous material spills)** **Phone: 800-255-3924**

Hospital Name/Address: Legacy Emanuel Medical Center
2801 N Gantenbein Ave., Portland, OR

Hospital Phone #: 503-413-2200

Directions to Hospital

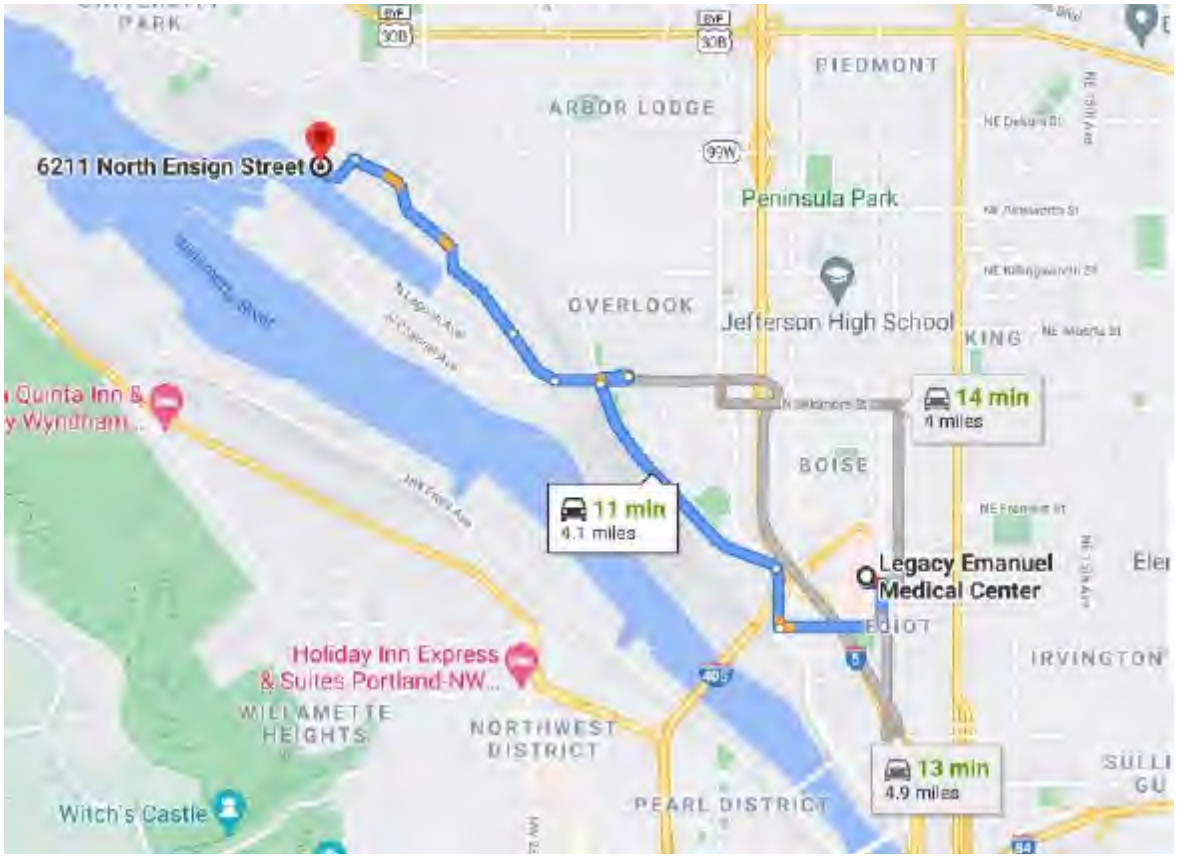
Directions:

1. Take N Basin Ave to N Channel Ave - 1.5 mi

2. Take N Going St, N Skidmore St and N Vancouver Ave to N Stanton St - 2.6 mi

3. Turn right onto N Stanton St, destination on right

See maps next page


6211 North Ensign Street
PIEDMONT
ARBOR LODGE
Peninsula Park
OVERLOOK
Jefferson High School
KING
BOISE
14 min
4 miles
11 min
4.1 miles
Legacy Emanuel
Medical Center
IRVINGTON
Holiday Inn Express
& Suites Portland-NW...
WILLAMETTE
HEIGHTS
NORTHWEST
DISTRICT
13 min
4.9 miles
PEARL DISTRICT
Witch's Castle

Incident Notification and Reporting

- Notify and submit reports to client as required in the contract.
- Serious Incidents must be reported in accordance with Jacobs Standard of Practice, *Serious Incident Reporting Process,* immediately; serious incidents are those that involve any of the following:
  - Work-related death, or life-threatening injury or illness of a Jacobs employee, subcontractor, or public
  - Kidnap/missing person
  - Acts or threats of terrorism
  - Event that involves a fire, explosion, or property damage that requires a site evacuation or is estimated to result in greater than $500,000 in damage
  - Spill or release of hazardous materials or substances that involves a significant threat of imminent harm to site workers, neighboring facilities, the community or the environment

In the event of an emergency, immediately call…. **911**:

- Severe Bleeding
- Loss of consciousness
- Chest Pain
- Broken bones
- All other injuries or illness' (even those that are minor and may only require First Aid) which occur at work, while on business travel, or commute must be reported to your supervisor immediately

After informing their supervisor, the injured employee calls Jacobs' contracted Occupational Nurse:

24-hour Jacobs Emergency Nurse Assistance
1-888-449-7787

- The Occupational Injury Nurse listens to the injured employee to understand the injury/illness.
- Employee is provided guidance on appropriate treatment options (triage).
- Appropriate treatment details are handled by the Occupational Injury Nurse, and HR Groups.
- Nurse communicates and troubleshoots with and for employee through full recovery.
- Complete an Intelex report and notify the HSM.

For "Serious Incidents" call the Global Assistance and Response HOTLINE # +1-443-221-6281

(e.g., work related death, kidnapping terrorism, property damage in excess of $500,000, spill with significant threat, etc. *Refer to Jacobs WIs for additional information.*)

JACOBS

BIAF Verbal Chain for Reporting all Incidents

Notes:

1. **Each Project/Region shall provide their own Verbal Reporting Chain- customised as required.**
2. Worst Potential Severity (WPS) definitions are to be found in HSEP-05-01A1Rev.0
3. Actual 4 and 5 must be communicated to Legal and Communications by the HSE VP or SVP
4. All incidents involving Jacobs employees or a sub-consultant or subcontractor under Jacobs control (including motor vehicle accidents, injuries, environmental incidents and near-misses) shall be reported as soon as possible **in person or by telephone.**
5. If your Project Manager is not your Line Manager it is essential that a call is made to both.
6. Calls must be made to both the relevant Corporate HSE Representative and the Establishment Manager/Office Leader or Project Manager/Construction Managers and/or Line Manager (as appropriate). These are not alternatives
7. Security and Sustainability Directors / VP's will be notified where appropriate by HSSEQ VP / HSE VP
8. Where required by legislation, a Jacobs HSES&S Manger will make the necessary report to the enforcing authorities
9. WPS definitions can be found in the regional incident reporting HSEP or SOP

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3 Chemical-Specific Training Form
4 Vehicle Accident Guidance
5 Project Activity Self-Assessment Checklists
6 Behavior Based Loss Prevention System Forms
7 Safety Data Sheets
8 Dive Safety Plan

Tables

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Acronyms and Abbreviations

Acronym	Definition
°C	degrees Celsius
°F	degrees Fahrenheit
µg/m3	micrograms per cubic meter
AHA	Activity Hazard Analysis
AQI	Air Quality Index
BBLPS	Behavior Based Loss Prevention System
BCD	Buoyancy Control Device
BG	Business Group
BZO	Beyond Zero Observation Report
CDC	Centers for Disease Control and Prevention
CFR	Code of Federal Regulations
CGI	combustible gas indicator
CPR	cardiopulmonary resuscitation
dBA	A-weighted decibels
DEET	N,N-diethyl-meta-toluamide
EM	Environmental Manager
EPA	U.S. Environmental Protection Agency
ERC	Emergency Response Coordinator
ES	Environmental Services
FA	first aid
FAA	Federal Aviation Administration
FSI	field safety instruction
FWSO	Firm Wide Security Operations

GFCI	ground-fault circuit interrupter
H&S	health and safety
HAZCOM	hazard communication
HAZWOPER	hazardous waste operations and emergency response
HR	human resources
HSE	health, safety, and environment
HSM	Health and Safety Manager
HSP	Health and Safety Plan
Jacobs	Jacobs Engineering Group Inc.
LEL	lower explosive limit
N/A	not applicable
OSHA	Occupational Safety and Health Administration
PFD	personal flotation device
PID	photoionization detector
PIM	potentially infectious material
PM	Project Manager
PPE	personal protective equipment
ppm	parts per million
PTSP	Pre-Task Safety Plan
RHSM	Responsible Health and Safety Manager
SDS	Safety Data Sheet
SL	Safety Liaison
SL-HW	Safety Liaison-Hazardous Waste
SPF	sun protection factor
TSD	treatment, storage, and disposal
TSDF	treatment, storage, and disposal facility

USCG	U.S. Coast Guard
UV	Ultraviolet
WCPA	Willbridge Cove Project Area
WI	Work Instruction
WPS	Worst Potential Severity

Note: If using a screen reader, adjustment to your default settings may be required.

1. Applicability

This Health and Safety Plan (HSP) applies to:

- All Jacobs Engineering Group Inc. staff (Jacobs) working on the site, including subcontractors and tiered subcontractors of Jacobs.
- All visitors to the construction site in the custody of Jacobs (including visitors from the client, the Government, the public, and other staff of any Jacobs company).

This HSP does not apply to the third-party contractors, their workers, their subcontractors, their visitors, or any other persons not under the direct control or custody of Jacobs.

This HSP defines the procedures and requirements for the health and safety (H&S) of Jacobs staff and visitors when they are physically on the work site. The work site includes the project area (as defined by the contract documents) and the project offices, trailers, and facilities thereon.

This HSP will be kept onsite during field activities and will be reviewed as necessary. The HSP will be amended or revised as project activities or conditions change or when supplemental information becomes available. The HSP adopts, by reference, the Jacobs Work Instructions (WIs), as appropriate. In addition, the HSP may adopt procedures from the project Work Plan and any governing regulations. If there is a contradiction between this HSP and any governing regulation, the more stringent and protective requirement shall apply.

All Jacobs staff and subcontractors must sign the employee sign-off form included in this document as Attachment 1 to acknowledge review of this document. Copies of the signature page will be maintained onsite by the Safety Liaison (SL).

2. General Project Information

This section presents the project information, background, and a description of the tasks that will be performed.

2.1 Project Information and Background

PROJECT NO: D3372300

CLIENT: Willbridge Cove Group

PROJECT/SITE NAME: Willbridge Cove Project Area

SITE ADDRESS: 5533 NW Doane Ave., Portland, Oregon

JACOBS PROJECT MANAGER: Marilyn Gauthier

DATE HEALTH AND SAFETY PLAN PREPARED: November 2020

DATE(S) OF SITE WORK: January 01, 2021 to January 01, 2022

DESCRIPTION OF SPECIFIC TASKS TO BE PERFORMED BY JACOBS: See Section 2.3.1 and 2.3.2

2.2 Site Background and Setting

The Willbridge Cove Project Area (WCPA) extends along the west side of the river from RM 7.5 and 8.0. The project area includes all river banks from the top of the bank to the river.

2.3 Description of Tasks

Project tasks are described in the following subsections.

2.3.1 Hazardous Waste Operations and Emergency Response (HAZWOPER)-Regulated Tasks

- Vibracoring
- Sediment sampling
- River bank soil sampling
- Underwater surface sediment sampling (for potential diving operations, a separate Dive Plan will be produced at that time and attached to this HSP)

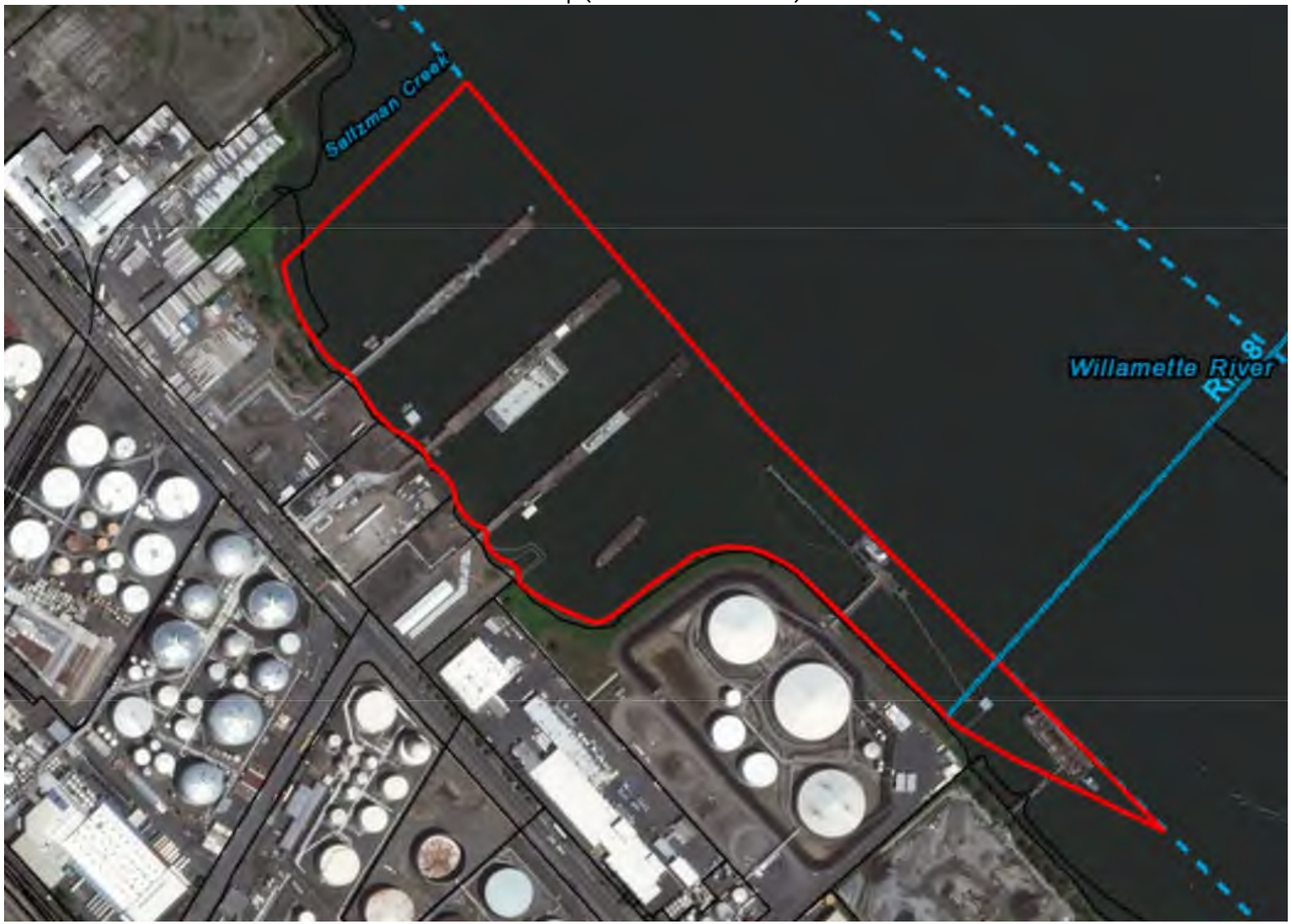
2.3.2 Non-HAZWOPER-Regulated Tasks

Under specific circumstances, the training and medical monitoring requirements of federal or state HAZWOPER regulations are not applicable. It must be demonstrated that the tasks can be performed without the possibility of exposure in order to use non-HAZWOPER-trained personnel.

TASKS**	CONTROLS
• Surveying • Utility Locate • Aerial surveying with a drone	• Brief on hazards, limits of access, and emergency procedures. • Post areas of contamination as appropriate. • Perform air sampling/monitoring as specified in Section 12.0. • Wear appropriate PPE as outlined in Section 13.0.

** activities with no exposure to site contaminants

Site Map (WCPA outlined in red)



3. Project Organization and Responsibilities

The following subsections summarize the roles and responsibilities of key project staff.

3.1 Client Project Coordinator

Contact Name: John Lang, P.E.

Phone: 513-325-2732

3.2 Jacobs

This subsection provides a summary of the key roles held by Jacobs staff.

3.2.1 Project Manager

Name: Marilyn Gauthier

Phone: 425-894 6464

The Project Manager (PM) is responsible for providing adequate resources (budget and staff) for project-specific implementation of the health, safety, and environment (HSE) management process. The PM has overall management responsibility for the tasks listed below. The PM may explicitly delegate specific tasks to other staff, as described in sections that follow, but retains ultimate responsibility for completion of the following in accordance with this document:

- Incorporate standard terms and conditions, and contract-specific HSE roles and responsibilities in contract and subcontract agreements (including flow-down requirements to lower-tier subcontractors).
- Select safe and competent subcontractors by:
  - Choosing potential subcontractors based on technical ability and HSE performance.
  - Implementing the subcontractor prequalification process.
  - Ensuring that acceptable certificates of insurance, including Jacobs as named additional insured, are secured as a condition of subcontract award.
  - Ensuring HSE submittals, subcontract agreements, and appropriate site-specific safety procedures are in place and accepted prior field mobilization.
- Ensure copies of training and medical monitoring records, and site-specific safety procedures are being maintained in the project file accessible to site personnel.
- Provide oversight of subcontractor HSE practices per the site-specific safety plans/procedures.
- Manage the site and interfacing with 3rd parties in a manner consistent with the contract and subcontract agreements and the applicable standard of reasonable care.
- Ensure that the overall, job specific, HSE goals are fully and continuously implemented.
- Support/implement use of stop work orders when subcontractor safety performance is not adequate.

3.2.2 Jacobs Responsible Health and Safety Manager (RHSM)

Name: John Culley/SPK

Phone: 206-660-3367

The RHSM is responsible for all aspects of project safety, including the following tasks:

- Review and evaluate subcontractor HSE performance using the prequalification process.
- Approve HSP and its revisions as well as Activity Hazard Analyses (AHA).
- Review and evaluate subcontractor site-specific safety procedures for adequacy prior to start of subcontractor's field operations.
- Support the oversight (or SL's direct oversight) of subcontractor and tiered subcontractor HSE practices.
- Permit upgrades/downgrades in respiratory protection after reviewing analytical data.
- Conduct audits as determined by project schedule and coordination with PM.
- Participate in incident investigations, lessons learned, loss/near loss reporting.

3.2.3 Jacobs Safety Liaison-Hazardous Waste (SL-HW)

Name: Shannon Bartow/PDX

Phone: 541-337-4415

Name: Laura Tochko/PDX

Phone: 425-891-2855

Name: Patrick Terhune/PDX

Phone: 907-378-1968

The SL is responsible for verifying the project is conducted in a safe manner, including the following specific obligations:

- Verify this HSP is current and amended when project activities or conditions change.
- Verify Jacobs site personnel and subcontractor personnel read the HSP and sign the Employee Sign-Off Form, prior to commencing field activities.
- Verify Jacobs site personnel have completed any required specialty training (e.g., fall protection, confined space entry) and medical surveillance as identified in this HSP.
- Verify that project files available to site personnel include copies of executed subcontracts and subcontractor certificates of insurance (including Jacobs as named additional insured), bond, contractor's license, training and medical monitoring records, and accepted site-specific safety procedures prior to start of subcontractor's field operations.
- Act as "Hazard Communication Coordinator" and perform the responsibilities outlined in the HSP.
- Act as "Emergency Response Coordinator" (ERC) and perform the responsibilities outlined in the HSP.
- Hold/verify that safety meetings are conducted and documented in the project file initially and as needed throughout the course of the project (e.g., as tasks or hazards change).

- Verify that project H&S forms and permits are being used as outlined this HSP.
- Perform oversight and/or assessments of subcontractor HSE practices per the site-specific safety plan and verify that project activity self-assessment checklists are being used as outlined this HSP.
- Coordinate with the RHSM regarding Jacobs and subcontractor operational performance, and 3rd party interfaces.
- Verify appropriate personal protective equipment (PPE) use, availability, and training.
- Calibrate and conduct air monitoring in accordance with the HSP. Maintain all air monitoring records in project file.
- Maintain HSE records and documentation.
- Facilitate Occupational Safety and Health Administration (OSHA) or other government agency inspections, including accompanying the inspector and providing necessary documentation and follow-up.
- Deliver field HSE training as needed based on project-specific hazards and activities.
- Contact the RHSM and PM in the event of an incident.
- When an apparent imminent danger exists, immediately remove all affected Jacobs employees and subcontractors, notify subcontractor safety representative, and stop affected work until adequate corrective measures are implemented. Notify the PM and RHSM as appropriate.
- Document all oral H&S-related communications in a project field logbook, daily reports, or other records.

3.3 Jacobs Subcontractors

Subcontractor: **TBD**
Subcontractor Contact Name: TBD
Telephone: TBD

Subcontractor Tasks: Marine vessel operations/vibracoring

Safety Procedures Required: Subcontractor must submit their AHA for review prior to commencing fieldwork and provide a copy to the SL-HW when arriving onsite.

Subcontractors must comply with the following activities, and are responsible to:

- Comply with all local, state, and federal safety standards.
- Comply with project and owner safety requirements.
- Participate in the project safety program and hold/attend/participate in all required safety meetings.
- Provide a qualified safety representative to interface with Jacobs.
- Maintain safety equipment and PPE for their employees.
- Maintain and replace safety protection systems damaged or removed by the subcontractor's operations.
- Notify the SL of any accident, injury, and/or incident immediately and submit reports to Jacobs within 24 hours.

- Install contractually required general conditions for safety (example: handrail, fencing, fall protection systems, floor opening covers, etc.).
- Conduct and document weekly safety inspections of project-specific tasks and associated work areas.
- Conduct site-specific and job-specific training for all subcontractor employees, including review of the Jacobs HSP, subcontractor HSPs, and subcontractor AHAs and sign appropriate sign-off forms.
- Determine and implement necessary controls and corrective actions to correct unsafe conditions.

The subcontractors listed above are responsible for the H&S procedures specific to their work and are required to submit their plans to Jacobs for review and acceptance before the start of field work. Subcontractors are also required to prepare AHAs before beginning each activity posing hazards to their personnel. The AHA shall identify the principal steps of the activity, potential H&S hazards for each step, and recommended control measures for each identified hazard. In addition, a listing of the equipment to be used to perform the activity, inspection requirements, and training requirements for the safe operation of the equipment listed must be identified.

3.4 Employee Responsibilities

All personnel are assigned responsibility for safe and healthy operations. For any operation, individuals have full authority to stop work and initiate immediate corrective action or control.

Each employee is responsible for the following performance objectives:

- Perform work in a safe manner and produce quality results.
- Perform work in accordance with company policies, and report injuries, illnesses, and unsafe conditions.
- Report all incidents immediately, and file proper forms with a human resources (HR) representative.
- Report all hazardous conditions and/or hazardous activities immediately to supervisor for corrective action.

3.4.1 Employee Authority

Each employee on the project has the obligation and authority to shut down any perceived unsafe work and during employee orientation, each employee will be informed of their authority to do so.

3.5 Client Contractors

Contractor: **TBD**
Contractor Contact Name: TBD
Telephone: TBD

Subcontractor Task: Waste management

This HSP does not cover contractors that are contracted directly to the client or the owner. Jacobs is not responsible for the H&S or means and methods of the contractor's work, and we must never assume such responsibility through our actions (e.g., advising on H&S issues). In addition to these instructions, Jacobs team members should review contractor safety plans so that we remain aware of appropriate precautions that apply to us. Self-assessment checklists in Attachment 5 are to be used by the SL and Jacobs team members to review the contractor's performance ONLY as it pertains to evaluating Jacobs exposure and

safety. The RHSM is the only person who is authorized to comment on or approve contractor safety procedures.

H&S-related communications with contractors should be conducted as follows:

- Request the contractor to brief Jacobs team members on the precautions related to the contractor's work.
- When an apparent contractor non-compliance/unsafe condition or practice poses a risk to Jacobs team members:
  - Notify the contractor safety representative.
  - Request that the contractor determine and implement corrective actions.
  - If necessary, stop affected Jacobs work until contractor corrects the condition or practice.
  - Notify the client, PM, and RHSM as appropriate.

If apparent contractor non-compliance/unsafe conditions or practices are observed, inform the contractor safety representative (Jacobs' obligation is limited strictly to informing the contractor of the observation—the contractor is solely responsible for determining and implementing necessary controls and corrective actions).

If an apparent imminent danger is observed, immediately warn the contractor employee(s) in danger and notify the contractor safety representative (Jacobs' obligation is limited strictly to immediately warning the affected individual(s) and informing the contractor of the observation—the contractor is solely responsible for determining and implementing necessary controls and corrective actions). Document all verbal H&S-related communications in project field logbook, daily reports, or other records.

4. Standards of Conduct

All individuals associated with this project must work injury-free and drug-free and must comply with the following Standards of Conduct, the HSP, and the safety requirements of Jacobs. Commonly accepted standards of conduct help maintain good relationships between people. They promote responsibility and self-development. Misunderstandings, frictions, and disciplinary action can be avoided by refraining from thoughtless or wrongful acts.

4.1 Standards of Conduct Violations

All individuals associated with this project are expected to behave in a professional manner. Violations of the standards of conduct would include, but not be limited to:

- Showing up not ready to work due to use of illegal drugs, alcohol, medication (prescribed or over-the-counter if they impair your senses), or in a fatigued state.
- Failure to perform work.
- Inefficient performance, incompetence, or neglect of work.
- Willful refusal to perform work as directed (insubordination).
- Negligence in observing safety regulations, poor housekeeping, or failure to report on-the-job injuries or unsafe conditions.
- Unexcused or excessive absence or tardiness.
- Unwillingness or inability to work in harmony with others.
- Discourtesy, irritation, friction, or other conduct that creates disharmony.
- Harassment or discrimination against another individual.
- Failure to be prepared for work by wearing the appropriate construction clothing or bringing the necessary tools.
- Violation of any other commonly accepted reasonable rule of responsible personal conduct.

4.2 Disciplinary Actions

The Environmental Services (ES) Business Group (BG) employees, employees working on ES BG projects, and subcontractor employees are subject to disciplinary action for not following HSE rules and requirements. Potential disciplinary action is equally applicable to all employees, including management and supervision. Disciplinary action may include denial of access to the worksite, warnings, reprimands, and other actions up to and including termination depending on the specific circumstances.

4.3 Subcontractor Safety Performance

Jacobs should continuously endeavor to observe subcontractors' safety performance and adherence to their plans and AHAs. This endeavor should be reasonable and include looking for hazards or unsafe practices that are both readily observable and occur in common work areas. Jacobs is not responsible for exhaustive observation for hazards and unsafe practices. Jacobs oversight does not relieve subcontractors of their responsibility for effective implementation and compliance with the established plan(s).

4.3.1 Observed Hazard Form

When apparent non-compliance or unsafe conditions or practices are observed, notify the subcontractor's supervisor/safety representative verbally, document the incident using the Observed Hazard Form included as an attachment to this HSP, and require corrective action.

If necessary, stop the subcontractor's work using the Stop Work Order Form until corrective actions are implemented for observed serious hazards or conditions. Update the Observed Hazard Form to document that corrective actions have been taken. The subcontractor is responsible for determining and implementing necessary controls and corrective actions.

4.3.2 Stop Work Order

Jacobs has the authority, as specified in the contract, and the responsibility to stop work in the event any Jacobs employee observes unsafe conditions or failure of the subcontractor to adhere to its safe work practices. This authority and action does not in any way relieve the subcontractor of its responsibilities for the means and methods of the work or, therefore, of any corrective actions. Failure to comply with safe work practices can be the basis for restriction or removal of the subcontractor staff from the job site, termination of the subcontract, restriction from future work, or all three.

When an apparent imminent danger is observed, immediately stop work and alert all affected individuals. Remove all affected Jacobs employees and subcontractor staff from the danger, notify the subcontractor's supervisory/safety representative, and do not allow work to resume until adequate corrective measures are implemented. Notify the PM, Contract Administrator, and RHSM.

When repeated non-compliance or unsafe conditions are observed, notify the subcontractor's supervisory/safety representative and stop affected work by completing and delivering the Stop Work Order Form (attached to this HSP) until adequate corrective measures are implemented. Consult the Contract Administrator to determine what the contract dictates for actions to pursue in event of subcontractor non-compliance (e.g., work stoppage, back charges, progress payments, removal of subcontractor manager, monetary penalties, or termination of subcontractor for cause).

4.4 Incentive Program

Each project is encouraged to implement a safety incentive program that rewards workers for exhibiting exemplary safety behaviors. Actions that qualify are those that go above and beyond what is expected, like identifying your own hazards with the work and developing your own mitigation techniques, motivating and implementing change in behaviors as it applies to fellow employees or subcontractors, etc. Actions that will be rewarded include spotting and correcting a hazard, bringing a hazard to the attention of your foreman, telling your foreman about an incident, coming up with a safer way to get the work done, stopping a crew member from doing something unsafe, etc. The program will operate throughout the project, covering all workers. The incentive program will be communicated to all employees during the project employee orientation and project safety meetings.

4.5 Reporting Unsafe Conditions/Practices

Responsibility for effective H&S management extends to all levels of the project and requires good communication between employees, supervisors, and management. Accident prevention requires a proactive policy on near misses, close calls, unsafe conditions, and unsafe practices. All personnel must report any situation, practice, or condition which might jeopardize the safety of our projects. All unsafe conditions or unsafe practices will be corrected immediately. Jacobs has zero tolerance of unsafe conditions or unsafe practices.

No employee or supervisor will be disciplined for reporting unsafe conditions or practices. Individuals involved in reporting the unsafe conditions or practices will remain anonymous.

All project employees will adhere to reporting procedures, which consist of the following:

- Upon detection of any unsafe condition or practice, the responsible employee will attempt to safely correct the condition.
- The unsafe condition or practice will be brought to the attention of the worker's direct supervisor, unless the unsafe condition or practice involves the employee's direct supervisor. If so, the SL needs to be notified at once by the responsible employee.
- Either the responsible employee or responsible employee's direct supervisor is responsible for immediately reporting the unsafe condition or practice to the SL.
- The SL will act promptly to correct the unsafe condition or practice.
- Details of the incident or situation will be recorded by the SL in the field logbook and/or use the Observed Hazard Form if a subcontractor was involved.

5. Safety Planning and Change Management

5.1 Daily Safety Meetings and Pre-Task Safety Plans (PTSPs)

Daily safety meetings are to be held with all project personnel in attendance to review the hazards posed and the required HSE procedures and AHAs that apply for each day's project activities. PTSPs serve the same purpose as these general assembly safety meetings, but the PTSPs are held between the crew supervisor and their work crews to focus on those hazards posed to individual work crews.

At the start of each day's activities, the crew supervisor completes the PTSP, provided as an attachment to this HSP, with input from the work crew, during their daily safety meeting. The day's tasks, personnel, tools, and equipment that will be used to perform these tasks are listed, along with the hazards posed and required HSE procedures, as identified in the HSP and AHAs. The use of PTSPs promotes worker participation in the hazard recognition and control process while reinforcing the task-specific hazards and required HSE procedures with the crew each day.

6. Project Hazard Analysis

An H&S risk analysis (see Table D-1-1) has been performed for each task. In the order listed in the bullets below, the RHSM considers the various methods for mitigating the hazards; employees are trained on this hierarchy of controls during their hazardous waste training and reminded of them throughout the execution of projects:

- Elimination of the hazards (e.g., use remote sampling methodology to avoid going into a confined space)
- Substitution (e.g., reduce exposure to vapors by using of a geoprobe instead of test pitting)
- Engineering controls (e.g., ventilate a confined space to improve air quality)
- Warnings (e.g., establish exclusion zones to keep untrained people away from hazardous waste work)
- Administrative controls (e.g., implement a work-rest schedule to reduce chance of heat stress)
- Use of PPE (e.g., use of respirators when action levels are exceeded)

The hazard controls and safe work practices are summarized in the following sections of this HSP:

- Section 7 – General Hazards and Controls
- Section 8 – Project-Specific Hazards and Controls
- Section 9 – Physical Hazards and Controls
- Section 10 – Biological Hazards and Controls
- Section 11 – Contaminants of Concern

6.1 Activity Hazard Analysis (AHAs)

An AHA defines the activity being performed, the hazards posed, and the control measures required to perform the work safely. Workers are briefed on the AHA before doing the work and their input is solicited prior, during, and after the performance of work to continuously identify the hazards posed and control measures required. The AHA shall identify the work tasks required to perform each activity, along with potential HSE hazards and recommended control measures for each hazard. In addition, a listing of the equipment to be used to perform the activity, inspection requirements, and training requirements for the safe operation of the equipment listed must be identified. The hazard controls presented in Section 7 and applicable Jacobs WIs should be used as a basis for preparing AHAs.

AHAs have been prepared for Jacobs chronological activities and are included as Attachment 5 to this HSP. AHAs developed by subcontractors will be reviewed and attached to the HSP upon procurement and prior to mobilization.

Table D-1-1. General Hazard Analysis

Potential Hazards	TASKS			
	Surveying, Utility Locate	River bank soil sampling	Aerial Survey with Drone	Vibracoring; Sediment Sampling
Flying debris/objects		X	X	X
Noise > 85 dBA		X	X	X
Electrical		X	X	X
Suspended loads			X	X
Working over water			X	X
Boat/Marine Vessel				X
Buried utilities, drums, tanks	X			X
Slip, trip, fall	X	X	X	X
Back injury	X	X	X	X
Visible lightning	X	X	X	X
Vehicle traffic	X	X	X	X
Elevated work areas/falls				X
Fires		X		X
Entanglement		X	X	X
Excavation				
Drilling				X
Heavy equipment				X
Railroad Hazards	X		X	X
Marine Vessel				X
Lone Worker (not expected but addressed; see Figure D-8.1for more guidance)	X	X	X	X
Wildfire Smoke	X	X	X	X
Diving Operations				X (separate Dive Plan will be developed at that time)
Exposure to site Contaminant of Concerns (see Section 11 for more details)		X		X
Investigation-derived Waste Management		X		X
Covid-19	X	X		X

Potential Hazards	TASKS			
	Surveying, Utility Locate	**River bank soil sampling**	**Aerial Survey with Drone**	**Vibracoring; Sediment Sampling**
Feral Dogs	X	X		X
Bloodborne Pathogens	X	X		X
Infectious Pathogens	X	X		X

Note:

dBA = A-weighted decibels

7. General Hazards and Controls

This section provides information on general hazards and controls.

7.1 General Practices and Housekeeping

The following are general requirements applicable to all portions of the work.

- Site work should be performed during daylight hours whenever possible.
- Good housekeeping must be maintained at all times in all project work areas.
- Common paths of travel should be established and kept free from the accumulation of materials.
- Keep access to aisles, exits, ladders, stairways, and emergency equipment free from obstructions.
- Provide slip-resistant surfaces, ropes, and/or other devices to be used.
- Specific areas should be designated for the proper storage of materials.
- Tools, equipment, materials, and supplies shall be stored in an orderly manner.
- As work progresses, unessential materials must be neatly stored or removed from the work area.
- Containers should be provided for collecting trash and other debris and removed at regular intervals.
- All spills shall be quickly cleaned up. Oil and grease cleaned from walking and working surfaces.
- Review the safety requirements of each job you are assigned with your supervisor. You are not expected to perform a job that may result in injury or illness to yourself or to others.
- Familiarize yourself with, understand, and follow jobsite emergency procedures.
- Do not fight or horseplay while conducting the firm's business.
- Do not use or possess firearms or other weapons while conducting the firm's business.
- Report unsafe conditions or unsafe acts to your supervisor immediately.
- Report emergencies, occupational illnesses, injuries, vehicle accidents, and near misses immediately.
- Do not remove or make ineffective safeguards or safety devices attached to any piece of equipment.
- Report unsafe equipment, defective or frayed electrical cords, and unguarded machinery.
- Shut down and lock out machinery and equipment before cleaning, adjustment, or repair. Do not lubricate or repair moving parts of machinery while the parts are in motion.
- Do not run in the workplace.
- When ascending or descending stairways, use the handrail and take one step at a time.
- Do not apply compressed air to any person or clothing.
- Do not wear steel taps or shoes with metal exposed to the sole at any Jacobs project location.
- Do not wear finger rings, loose clothing, wristwatches, and other loose accessories when within arm's reach of moving machinery.
- Remove waste and debris from the workplace and dispose of in accordance with federal, state, and local regulations.

- Note the correct way to lift heavy objects (secure footing, firm grip, straight back, lift with legs), and get help if needed. Use mechanical lifting devices whenever possible.
- Check the work area to determine what problems or hazards may exist.

7.2 Driving Safety

Follow the guidelines below when operating a vehicle:

- Refrain from using a cellular phone while driving. Pull off the road, put the vehicle in park, and turn on flashers before talking on a cellular phone.
- Never operate a device's Personal Digital Assistant, e-mail, internet, or text messaging function while driving a vehicle.
- Obey speed limits, be aware of blind spots or other hazards associated with low visibility. Practice defensive driving techniques, such as leaving plenty of room between your vehicle and the one ahead.
- Do no drive while drowsy. Drowsiness can occur at any time but is most likely after 18 hours or more without sleep.
- Maintain focus on driving. Eating, drinking, smoking, or adjusting controls can divert attention away from the road. Take the time to park and perform these tasks when parked rather than while driving.

7.3 Personal Hygiene

Good hygiene is essential for personal health and to reduce the potential of cross-contamination when working on a hazardous waste site. Implement the following:

- Keep hands away from nose, mouth, and eyes during work.
- Keep areas of broken skin (chapped, burned, etc.) covered.
- Wash hands with soap and water prior to eating, smoking, or applying cosmetics.

7.4 Shipping and Transportation of Chemical Products

Chemicals brought to the site might be defined as hazardous materials by the U.S. Department of Transportation. All staff who ship the materials or transport them by road must receive Jacobs training in shipping dangerous goods. All hazardous materials that are shipped (e.g., via Federal Express) or are transported by road must be properly identified, labeled, packed, and documented by trained staff. Contact the RHSM or the Warehouse Coordinator for additional information.

8. Project-Specific Hazard Controls

This section provides safe work practices and control measures used to reduce or eliminate potential hazards. These practices and controls are to be implemented by the party in control of either the site or the particular hazard. Jacobs employees and subcontractors must remain aware of the hazards affecting them regardless of who is responsible for controlling the hazards. Jacobs employees and subcontractors who do not understand any of these provisions should contact the SL-HW for clarification.

8.1 Lone Workers

“Lone workers” are permitted under some circumstances, depending on the work tasks scheduled, isolation, environmental factors, etc. Reference the “Lone Worker Protocol” for additional guidance on the protocol, which is included in this HSP as Figure D-8-1. Field personnel will provide written daily confirmation to the PM (or designee) of the scheduled for the day that are anticipated to require lone workers, documenting the tasks on the PTSP.

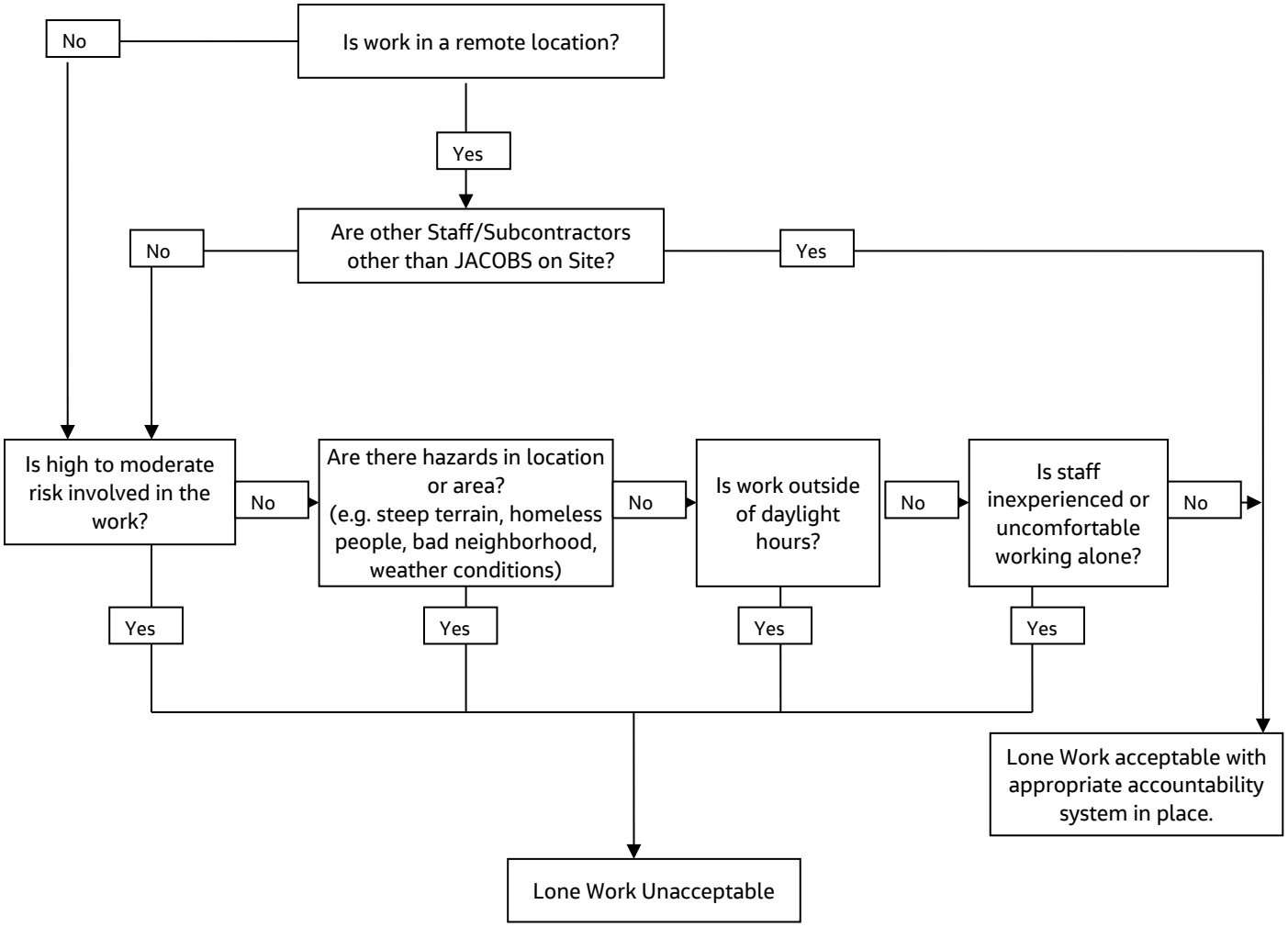


Figure D-8-1. Lone Worker Protocol

8.2 Chemical Spills

- Follow instructions of the incident response commander (typically fire department, U.S. Coast Guard, [USCG] or client representative)
- Avoid contact with commodities at accident sites until the materials have been identified and safe handling procedures determined
- Remain upwind from spill

8.3 Heavy Equipment

- Jacobs authorizes only those employees qualified by training or previous experience to operate material handling equipment.
- Equipment must be checked at the beginning of each shift to ensure the equipment is in safe operating condition and free of apparent damage. The check should include service brakes, parking brakes, emergency brakes, tires, horn, backup alarm, steering mechanism, coupling devices, seat belts and operating controls. All defects shall be corrected before the equipment is placed in service. Documentation of this inspection must be maintained onsite at all times.
- Equipment must be on a stable foundation such as solid ground or cribbing; outriggers are to be fully extended.
- Equipment must not be used to lift personnel; loads must not be lifted over the heads of personnel.
- Equipment, or parts thereof, which are suspended must be substantially blocked or cribbed to prevent shifting before personnel are permitted to work under or between them. All controls shall be in a neutral position, with the motors stopped and brakes set.
- Equipment which is operating in reverse must have a reverse signal alarm distinguishable from the surrounding noise or a signal person when the operators view is obstructed.
- When equipment is used near energized powerlines, the closest part of the equipment must be at least 10' from the powerlines < 50 kV. See Section 8.16 for guidance on distances. A person must be designated to observe clearances and give timely warning for all operations where it is difficult for the operator to maintain the desired clearance by visual means. All overhead powerlines must be considered to be an energized until the electrical utility authorities indicate that it is not an energized line and it has been visibly grounded.
- Underground utility lines must be located before excavation begins.
- Operators loading/unloading from vehicles are responsible for seeing that vehicle drivers are in the vehicle cab or in a safe area.
- The parking brake shall be set whenever equipment is parked, wheels must be chocked when parked on inclines.
- When not in operation, the blade/bucket must be blocked or grounded; the master clutch must be disengaged when the operator leaves the cab. When equipment is unattended, power must be shut off, brakes set, blades/buckets landed and shift lever in neutral.

8.4 Noise Hazards

Previous surveys indicate that heavy equipment, such as drilling or excavation equipment, may produce continuous and impact noise at or above the action level of 85 dBA. All Jacobs personnel within 25 feet of

operating equipment, or near an operation that creates noise levels high enough to impair conversation, shall wear hearing protective devices (either muffs or plugs). Personnel will wash their hands with soap and water prior to inserting ear plugs to avoid initiating ear infections. Additional information regarding Jacobs' Hearing Conservation Program is located in of the Jacobs Corporate Health and Safety Program, Program and Training Manual. Access to this document can be easily obtained on the Jacobs H&S Intranet Site. All Jacobs field staff should complete the training module on Noise Hazards located on the virtual office before field work begins.

8.5 Soil Sampling

- Tie down loose items.
- Utilize a spotter if backing vehicles or equipment towards sampling location.
- Inspect the sampling area for obstructions and Poison Ivy and Poison Oak, or other physical hazards.
- If sample locations are located in dense tall grassy areas, consider utilizing a “Bug-Out” suit or Tyvek to mitigate the potential for tick bites.
- If lifting heavy equipment from vehicle, move items to the rear and get assistance when lifting.
- Be alert for bees, wasps and other insects when sampling. If known allergy, carry medication (e.g. EpiPen), and inform teammates on its location in the event of an emergency and the severity of your allergy.
- Log calibration of direct-reading instrument in either a field log book or on attached form.
- Notify others in area that task is going to be performed, delineate an exclusion zone as applicable.
- Don PPE as specified in Section 13 of this site-specific HSP.
- Position yourself upwind prior to sampling, if possible.
- Do not handle sample jars without nitrile gloves.

8.6 Field Vehicles

- Field vehicles may be personal vehicles, rental vehicles, fleet vehicles, or project vehicles.
- Maintain a first aid (FA) kit, bloodborne pathogen kit, and fire extinguisher in the field vehicle at all times.
- Utilize a rotary beacon on vehicle if working adjacent to active roadway.
- Car rental must meet the following requirements:
  - Dual air bags
  - Antilock brakes
  - Be midsize or larger
- Familiarize yourself with rental vehicle features prior to operating the vehicle:
  - Mirror adjustments
  - Seat adjustments
  - Cruise control features, if offered
  - Pre-program radio stations and Global Positioning System (GPS), if equipped
- Always wear seatbelt while operating vehicle.
- Adjust headrest to proper position.
- Tie down loose items if utilizing a van or pick-up truck.

- Close car doors slowly and carefully. Fingers can get pinched in doors.
- Park vehicle in a location where it can be accessed easily in the event of an emergency. If not possible, carry a phone.
- Have a designated place for storing the field vehicle keys when not in use.
- Ensure backup alarms are functioning, if equipped. Before backing a vehicle, take a walk around the vehicle to identify obstructions/hazards. Use a spotter when necessary to back into or out of an area.

Any accident involving a pool or other leased vehicle, or rental vehicle, refer to Attachment 4.

8.7 Fire Prevention

Fire prevention practices are discussed in the section below.

8.7.1 Fire Extinguishers and General Fire Prevention Practices

- Fire extinguishers shall be provided so that the travel distance from any work area to the nearest extinguisher is less than 100 feet (30.5 meters). When 5 gallons (19 liters) or more of a flammable or combustible liquid is being used, an extinguisher must be within 50 feet (15.2 meters). Extinguishers must:
  - Be maintained in a fully charged and operable condition,
  - Be visually inspected each month, and
  - Undergo a maintenance check each year.
- The area in front of extinguishers must be kept clear.
- Combustible materials stored outside should be at least 10 feet (3 meters) from any building.
- Solvent waste and oily rags must be kept in a fire-resistant container until removed from the site.

8.7.2 Storage of Flammable/Combustible Liquids

- Only approved containers and portable tanks shall be used for storage and handling of flammable and combustible liquids.
- Approved safety cans shall be used for the handling and use of flammable liquids in quantities of 5 gallons (19 liters) or less. Do not use plastic gas cans.
- For quantities of 1 gallon (3.8 liters) or less, the original container may be used for storage and use of flammable liquids.
- Flammable or combustible liquids shall not be stored in areas used for stairways or normally used for the passage of people.

8.7.3 Indoor Storage of Flammable/Combustible Liquids

- No more than 25 gallons (113.7 liters) of flammable or combustible liquids shall be stored in a room outside of an approved storage cabinet.
- Quantities of flammable and combustible liquids in excess of 25 gallons (113.7 liters) shall be stored in an acceptable or approved cabinet.
- Cabinets shall be conspicuously lettered: "FLAMMABLE: KEEP FIRE AWAY."

- Not more than 60 gallons (272.8 liters) of flammable or 120 gallons (545.5 liters) of combustible liquids shall be stored in any one storage cabinet. Not more than three such cabinets may be located in a single storage area.

8.7.4 Outside Storage of Flammable/Combustible Liquids

- Storage of containers (not more than 60 gallons [272.8 liters] each) shall not exceed 1,100 gallons (5000 liters) in any one area. No area shall be within 20 feet (6.1 meters) of any building.
- Storage areas shall be graded to divert spills away from buildings and surrounded by an earthen dike.
- Storage areas shall be free from weeds, debris, and other combustible materials.
- Outdoor portable tanks shall be provided with emergency vent devices and shall not be closer than 20 feet (6.1 meters) to any building.
- Signs indicating no smoking shall be posted around the storage area.

8.7.5 Dispensing of Flammable/Combustible Liquids

- Areas in which flammable or combustible liquids are dispensed in quantities greater than 5 gallons (22.7 liters) shall be separated from other operations by at least 25 feet (7.6 meters).
- Drainage or other means shall be provided to control spills.
- Adequate natural or mechanical ventilation shall be provided to maintain the concentration of flammable vapor at or below 10 percent of the lower flammable limit.
- Dispensing of flammable liquids from one container to another shall be done only when containers are electrically interconnected (bonded).
- Dispensing flammable or combustible liquids by means of air pressure on the container or portable tanks is prohibited.
- Dispensing devices and nozzles for flammable liquids shall be of an approved type.

8.8 Hand and Power Tools

- Tools shall be inspected prior to use and damaged tools will be tagged and removed from service.
- Hand tools will be used for their intended use and operated in accordance with manufacturer's instructions and design limitations.
- Maintain all hand and power tools in a safe condition.
- Use PPE (such as gloves, safety glasses, earplugs, and face shields) when exposed tool hazards.
- Do not carry or lower a power tool by its cord or hose.
- Portable power tools will be plugged into ground-fault circuit interrupter (GFCI) protected outlets.
- Portable power tools will be Underwriters Laboratories listed and have a three-wire grounded plug or be double insulated.
- Disconnect tools from energy sources when they are not in use, before servicing and cleaning them, and when changing accessories (such as blades, bits, and cutters).
- Safety guards on tools must remain installed while the tool is in use and must be promptly replaced after repair or maintenance has been performed.

- Store tools properly in a place where they will not be damaged or come in contact with hazardous materials.
- If a cordless tool is connected to its recharge unit, both pieces of equipment must conform strictly with electrical standards and manufacturer's specifications.
- Tools used in an explosive environment must be rated for work in that environment (that is, intrinsically safe, spark-proof, etc.).
- Working with manual and pistol-grip hand tools may involve highly repetitive movement, extended elevation, constrained postures, and/or awkward positioning of body members (for example, hand, wrist, arm, shoulder, neck, etc.). Consider alternative tool designs, improved posture, the selection of appropriate materials, changing work organization, and sequencing to prevent muscular, skeletal, repetitive motion, and cumulative trauma stressors.

8.8.1 Machine Guarding

- Ensure that all machine guards are in place to prevent contact with drive lines, belts, chains, pinch points or any other sources of mechanical injury.
- Unplugging jammed equipment will only be performed when equipment has been shut down, all sources of energy have been isolated, and equipment has been locked/tagged and tested.
- Maintenance and repair of equipment that results in the removal of guards or would otherwise put anyone at risk requires lockout of that equipment prior to work.

8.9 Hazard Communication/GHS

- Complete an inventory of chemicals brought on site by Jacobs using the chemical inventory form included as an attachment to this HSP.
- Confirm that an inventory of chemicals brought on site by Jacobs subcontractors is available.
- Request or confirm locations of Safety Data Sheets (SDSs) from the client, contractors, and subcontractors for chemicals to which Jacobs employees potentially are exposed.
- Before or as the chemicals arrive on site, obtain SDS for each hazardous chemical and include on the chemical inventory sheet (attached to this HSP) and add the SDS to the attachments of this HSP.
- Label chemical containers with the identity of the chemical and with hazard warnings, and store properly.
- Give employees required chemical-specific hazard communication (HAZCOM) training using the chemical-specific training form included as an attachment to this HSP.
- Store all materials properly, considering compatibility, quantity limits, secondary containment, fire prevention, and environmental conditions.

The following are general guidelines for storing chemicals and other hazardous materials:

- Keep acids away from bases.
- Keep oxidizers (nitric acid, nitrates, peroxides, chlorates) and organics away from inorganic reducing agents (metals).
- Keep flammables and corrosives in appropriate storage cabinets.
- Do not store paper or other combustibles near flammables.

- Use secondary containment and lipped shelving that is secured.
- Have a fire suppression system available.

8.10 Knife Use

Open-bladed knives (e.g., box cutters, utility knives, pocket knives, machetes, and multi-purpose tools with fixed blades such as a Leatherman™) are prohibited at worksites except where the following three conditions are met:

- The open-bladed knife is determined to be the best tool for the job. Example: a knife is needed onboard the boat in the event of emergency where a person is entangled and needs immediate extraction for safety, and the knife is the best tool for that.
- An approved AHA or written procedure is in place that covers the necessary safety precautions (work practices, PPE, and training).
- Knife users have been trained and follow the AHA.

8.11 Asbestos

- Materials suspected of containing asbestos shall be treated as asbestos unless documentation and/or testing results indicate otherwise.
- Where the presence of asbestos is suspected, design all operations to avoid contact.
- Do not disturb waste or other materials labeled “Danger - Asbestos Fibers.”
- Jacobs employees are not to enter regulated work areas unless training, medical monitoring, and PPE requirements established by the Health and Safety Manager (HSM) have been met.
- Do not eat, drink, smoke, chew tobacco or gum, or apply cosmetics in regulated areas.

8.12 Excavation

- Do not enter the excavations unless completely necessary, and only after the competent person has completed the daily inspection and has authorized entry.
- Follow all excavation entry requirements established by the competent person.
- Do not enter excavations where protective systems are damaged or unstable.
- Do not enter excavations where objects or structures above the work location may become unstable and fall into the excavation.
- Do not enter excavations with accumulated water unless precautions have been taken to prevent excavation cave-in.
- Do not enter excavations with the potential for a hazardous atmosphere until the air has been tested and found to be at safe levels.

8.13 Drilling/Direct-push/Vibracoring

- Only authorized personnel are permitted to operate drill rigs.
- Stay clear of areas surrounding drill rigs during every startup.
- Stay clear of the rotating augers and other rotating components of drill rigs.

- Stay as clear as possible of all hoisting operations. Loads shall not be hoisted overhead of personnel.
- Do not wear loose-fitting clothing or other items such as rings or watches that could get caught in moving parts. Long hair must be restrained.
- If equipment becomes energized, personnel shall be instructed not to touch any part of the equipment or attempt to touch any person who may be in contact with the electrical current. The utility company or appropriate party shall be contacted to have line de-energized prior to approaching the equipment.
- Smoking around drilling operations is prohibited.
- Wear the appropriate PPE when sampling (refer to Section 13) especially when splash hazards exist.
- Use the appropriate lifting procedures when unloading equipment and sampling.
- Allow ample space for personnel to lift/store drilling rods.
- Avoid movement near rig. Do not place hands near hammer.
- If dermal contact with the groundwater and acid used in sample preservation, wash exposed skin thoroughly with soap and water.
- Avoid eating and drinking on site and during sampling.
- Use ear plugs during sampling if sampling involves a generator.
- The drill rig must be equipped with a kill wire or switch, and personnel are to be informed of its location.
- Be aware and stand clear of heavy objects that are hoisted overhead.
- The driller to verify the rig is properly maintained in accordance with the company's maintenance program.
- The driller is to verify that all machine guards are in place while the rig is in operation.
- The driller is responsible for housekeeping (maintaining a clean work area).
- The drill rig should be equipped with at least one fire extinguisher.
- If the drill rig comes into contact with electrical wires and becomes electrically energized, do not touch any part of the rig or any person in contact with the rig, and stay as far away as possible. Notify emergency personnel immediately.
- The drill rig should be leveled and stabilized with jacks and adequate cribbing before raising the mast and during drilling operations. Cribbing materials should be made from materials that are capable of supporting the weight of the rig. Care should be taken in muddy, soggy soil, or partially frozen areas. In addition to cribbing, guy wires should be used to improve stability if the rig is located on wet, partially frozen ground, or in areas with loose, caving soil, or in an area subject to frequent gusty winds.

8.14 Utilities (underground)

The following subsections provide guidance for working near underground utilities.

8.14.1 Local Utility Mark-Out Services

Name: Oregon One Call

Phone: 811

An assessment for underground utilities must be conducted where there is a potential to contact underground utilities or similar subsurface obstructions during intrusive activities. Intrusive activities include excavation, trenching, drilling, hand augering, soil sampling, or similar activities.

The assessment must be conducted before any intrusive subsurface activity and must include at least the following elements:

1) A background and records assessment of known utilities or other subsurface obstructions.
2) Contacting and using the designated local utility locating service.
3) Conducting an independent field survey to identify, locate, and mark potential underground utilities or subsurface obstructions. *Note: This is independent of, and in addition to, any utility survey conducted by the designated local municipal utility locating service above.*
4) A visual survey of the area to validate the chosen location.

When any of these steps identifies an underground utility within 6 feet (2 meters) of intrusive work, then non-aggressive means must be used to physically locate the utility before a drill rig, backhoe, excavator or other aggressive method is used.

Aggressive methods are never allowed within 3 feet of an identified high-risk utility (see paragraph below).

Any deviation from these requirements must be approved by the Responsible HS Manager and the PM.

8.14.2 Background and Records Assessment of Known Utilities

Identify any client- or location-specific permit and/or procedural requirements (e.g., dig permit or intrusive work permit) for subsurface activities. For military installations, contact the Base Civil Engineer and obtain the appropriate form to begin the clearance process.

Obtain available utility diagrams and/or as-built drawings for the facility. Review locations of possible subsurface utilities including sanitary and storm sewers, electrical lines, water supply lines, natural gas lines, fuel tanks and lines, communication lines, lighting protection systems, etc. Note: Use caution in relying on as-built drawings as they are rarely 100 percent accurate. Request that a facility contact with knowledge of utility locations review and approve proposed locations of intrusive work.

8.14.3 Designated Local Utility Locating Service

Contact your designated local utility locating service (e.g., Dig-Safe, Blue Stake, One Call) to identify and mark the location of utilities. Call 811 in the U.S. or go to www.call811.com to identify the appropriate local service group. Contacting the local utility locating service is a legal requirement in most jurisdictions.

8.14.4 Independent Field Survey (Utility Locate)

The organization conducting the intrusive work (Jacobs or subcontractor) shall arrange for an independent field survey to identify, locate, and mark any potential subsurface utilities in the work area. This survey is in addition to any utility survey conducted by the designated local utility locating service.

The independent field survey provider shall determine the most appropriate instrumentation/technique or combinations of instrumentation/techniques to identify subsurface utilities based on their experience and expertise, types of utilities anticipated to be present, and specific site conditions.

A Jacobs or subcontractor representative must be present during the independent field survey to observe the utility locate and verify that the work area and utilities have been properly identified and marked. If there is any question that the survey was not performed adequately or the individual was not qualified, then arrangements must be made to obtain a qualified utility locate service to resurvey the area. Obtain documentation of the survey and clearances in writing and signed by the party conducting the clearance. Maintain all documentation in the project file.

If the site owner (military installation or client) can provide the independent field survey, Jacobs or the subcontractor shall ensure that the survey includes:

Physically walking work location for visual cues and identify, locate, and mark underground utility locations:

Having qualified staff available and instrumentation to conduct the locate.

Agreeing to document the survey and clearances in writing.

Should any of the above criteria not be met, Jacobs or subcontractor must arrange for an alternate independent utility locate service to perform the survey.

The markings from utility surveys must be protected and preserved until the markings are no longer required. If the utility location markings are destroyed or removed before intrusive work commences or is completed, the PM, SL, or designee must notify the independent utility locate service or the designated local utility locating service to resurvey and remark the area.

8.14.5 Visual Assessment before and during Intrusive Activities

Perform a "360 degree" assessment. Walk the area and inspect for utility-related items such as valve caps, previous linear cuts, patchwork in pavement, hydrants, manholes, utility vaults, drains, and vent risers in and around the dig area.

The visual survey shall include all surface landmarks, including manholes, previous liner cuts, patchwork in pavement, pad-mounted transformers, utility poles with risers, storm sewer drains, utility vaults, and fire hydrants.

If any unanticipated items are found, conduct further research before initiating intrusive activities and implement any actions needed to avoid striking the utility or obstruction.

8.14.6 Subsurface Activities within 6 feet of an Underground Utility or if there is Uncertainty

When aggressive intrusive activities will be conducted within 6 feet (2 meters) of an underground utility or when there is uncertainty about utility locations, locations must be physically verified by non-aggressive means such as air or water knifing, hand digging, or post-hole digging. Non-conductive tools must be used if electrical hazards may be present. If intrusive activities are within 6 feet (2 meters) and parallel to a marked existing utility, the utility location must be exposed and verified by non-aggressive methods every 100 feet (30.5 meters). Check to see if the utility can be isolated during intrusive work.

8.14.7 Intrusive Activities within 3 feet of an Underground Utility

Use non-aggressive methods (hand digging, vacuum excavation, etc.) to perform intrusive activities within 3 feet of a high-risk utility (i.e., a utility that cannot be de-energized or would cause significant impacts to repair/replace). Hazardous utilities shall be de-energized whenever possible.

8.14.8 Spotter

A spotter shall be used to monitor for signs of utilities during advancement of intrusive work (e.g., sudden change in advancement of auger or split spoon, presence of pea gravel or sand in soil, presence of concrete or other debris in soil, refusal of auger or excavating equipment). If any suspicious conditions are encountered stop work immediately and contact the PM or RHSM to evaluate the situation. The spotter must have a method to alert an operator to stop the intrusive activity (e.g., air horn, hand signals).

8.15 Personal Hygiene

Good hygiene is essential for personal health and to reduce the potential of cross-contamination when working on a hazardous waste site. Implement the following:

- Keep hands away from nose, mouth, and eyes during work
- Keep areas of broken skin (chapped, burned, etc.) covered
- Wash hands with soap and water prior to eating, smoking, or applying cosmetics
- Dispose of PPE properly
- Wash boots before leaving the site, or wear and then dispose of boot covers

8.16 Exposure to Public Vehicular Traffic

The following precautions must be taken when working around traffic, and in or near an area where traffic controls have been established by a contractor.

- Exercise caution when exiting traveled way or parking along street – avoid sudden stops, use flashers, etc.
- Park in a manner that will allow for safe exit from vehicle, and where practicable, park vehicle so that it can serve as a barrier.
- All staff working adjacent to traveled way or within work area must wear reflective/high-visibility safety vests.
- Eye protection should be worn to protect from flying debris.
- Remain aware of factors that influence traffic related hazards and required controls – sun glare, rain, wind, flash flooding, limited sight distance, hills, curves, guardrails, width of shoulder (i.e., breakdown lane), etc.
- Always remain aware of an escape route -- behind an established barrier, parked vehicle, guardrail, etc.
- Always pay attention to moving traffic – never assume drivers are looking out for you.
- Work as far from traveled way as possible to avoid creating confusion for drivers.
- When workers must face away from traffic, a “buddy system” should be used, where one worker is looking towards traffic.
- When working on highway projects, obtain a copy of the contractor’s traffic control plan.

- Work area should be protected by a physical barrier – such as a K-rail or Jersey barrier.
- Review traffic control devices to ensure they are adequate to protect your work area. Traffic control devices should: (1) convey a clear meaning, (2) command respect of road users, and (3) give adequate time for proper traffic response. The adequacy of these devices is dependent on limited sight distance, proximity to ramps or intersections, restrictive width, duration of job, and traffic volume, speed, and proximity.
- Lookouts should be used when physical barriers are not available or practical. The lookout continually watches approaching traffic for signs of erratic driver behavior and warns workers. Vehicles should be parked at least 40 feet away from the work zone and traffic. Minimize the amount of time that you will have your back to oncoming traffic.

8.17 Utilities (Overhead)

The following subsection provides guidance for working near overhead utilities.

8.17.1 Proximity to Power Lines

No work is to be conducted within 50 feet (15.2 meters) of overhead power lines without first contacting the utility company to determine the voltage of the system. No aspect of any piece of equipment is to be operated within 50 feet (15.2 meters) of overhead power lines without first making this determination.

Operations adjacent to overhead power lines are PROHIBITED unless one of the following conditions is satisfied:

- Power has been shut off, positive means (such as lockout) have been taken to prevent the lines from being energized, lines have been tested to confirm the outage, and the utility company has provided a signed certification of the outage.
- The minimum clearance from energized overhead lines is as shown in the table below, or the equipment will be repositioned and blocked to ensure that no part, including cables, can come within the minimum clearances shown in the table.

MINIMUM DISTANCES FROM POWERLINES

Powerlines Nominal System Kv	Minimum Required Distance, Feet (Meters)
0-50	10
51-200	15
201-350	20
351-500	25
501-750	35
751-1,000	45
Over 1,000	Established by utility owner/operator or by a professional engineer in electrical power transmission/distribution

These distances have been determined to eliminate the potential for arcing based on the line voltage.

- The power line(s) has been isolated through the use of insulating blankets which have been properly placed by the utility. If insulating blankets are used, the utility will determine the minimum safe operating distance; get this determination in writing with the utility representative's signature.

All inquiries regarding electric utilities must be made in writing and a written confirmation of the outage/isolation must be received by the PM prior to the start of work.

8.18 Encountering Illegal Activity or Homeless in the Field

- Always have a buddy.
- Keep in mind you may encounter illegal activity or homeless persons within your work areas.
- If you encounter an illegal activity, immediately leave the area. Do not wait around to use your phone, mark on maps, mark locations on GPS units, etc. when in the vicinity of the illegal activities. Contact the authorities and mark maps once you are safely miles away.
- If you are confronted by a suspected criminal or homeless person, do not escalate the situation. Explain to them that you are not anyone of authority. If you are told to surrender something to a criminal, do so and leave the area. If you are asked for money by a homeless person, use your best judgment, but it is best not to give them any so they do not continue to return. It is better for your safety and theirs.

8.19 Working Above or Near Water

- Fall protection should be provided to prevent personnel from falling into water. Where fall protection systems are not provided and the danger of drowning exists, USCG approved personal flotation devices (PFDs), or a life jacket, shall be worn.
- Inspect PFDs prior to use. Do not use defective PFDs.
- A minimum of one ring buoy with 90 feet of 3/8-inch solid-braid polypropylene (or equal) rope must be provided for emergency rescue.
- Use sampling and other equipment according to the manufacturers' instructions.
- No smoking is permitted on board vessels or during refueling operations.
- The boat skipper has the final authority with regard to boat safety and navigational safety.

8.20 Slips, Trips and Falls

The following subsections provide guidance on preventing slips, trips, and falls in differing conditions.

8.20.1 General

- Institute and maintain good housekeeping practices.
- Designate foot traffic paths in and out of sites, when necessary, to ensure paths are kept free from slip, trip, and fall hazards or to deter personnel from taking “shortcuts” where slip, trip, hazards may be.
- Mitigate icy conditions by keeping foot traffic paths clear of ice and snow.
- Watch footing to avoid trip hazards, animal holes, or other obstacles, especially in tall grassy areas.

8.20.2 Muddy Conditions

- Muddy conditions present a slipping hazard. Use mats or similar surface if footing cannot be stabilized.
- Take shortened steps across muddy areas.
- Use a walking staff or other similar means to assist with balance.

8.20.3 Steep Slopes/Uneven Ground/Rock and Vertical Slopes

- Be aware that escarpments can slough. Avoid these areas.
- Exercise caution in relying on rocks and trees/tree stumps to support yourself – many times they are loose.
- Whenever possible, switchback your way up/down steep areas; maintain a slow pace with firm footing.
- Employees walking in ditches, swales and other drainage structures adjacent to roads or across undeveloped land must use caution to prevent slips and falls which can result in twisted or sprained ankles, knees, and backs.
- When possible observe conditions from a flat surface; do not enter steep ditch or side of a steep road bed.
- If steep terrain must be negotiated coordinate with RHSM to evaluate the need for ladders/ropes to provide stability.

8.20.4 Stream Crossing

Traversing streams present significant hazards, including drowning, hypothermia, and abrasions. When crossing streams, be sure to implement the bulleted items below.

- When walking in streams, first plan the route. Look ahead for exits should there be any difficulty during the crossing, and "read" the water for spots to avoid such as drop offs, sunken logs, and tricky currents.
- Do seek out the safest route – narrow, low flow, shallow. Evaluate deeper and faster moving sections with caution. Backtracking is often dangerous or impossible once committed.
- If streams to be crossed are deeper than "knee deep", find an alternate crossing location that is less deep.
- Streams should be crossed while facing upstream, stepping side to side, and using a sturdy walking stick. When possible, wade a stream diagonally, moving downstream. Move slowly, keeping the foot on the upstream side in the lead and pointed forward. Your rear, or anchor, foot should point downstream and be at right angles to the lead foot. Move the lead foot forward about half a step, feeling for a solid hold. Next, move the anchor foot forward the same distance – shuffle across so that your anchor foot never passes the lead. This way both feet are always in position to lend support. If you must turn around, do so toward the upstream direction.
- Do not attempt to cross above rocky rapids or a cascade. Step on submersed rocks with great care.
- If you are working in streams, algae covered rocks should be assumed slippery until tested. Always be alert for unstable and extremely slippery rocks.
- Rocks with green moss or attached plants offer better traction, look for gravel and sand pockets among the stream boulders, which are much more stable, and use a wading staff (if not carrying one, find a suitable one nearby) to steady your balance while crossing. Use a solid wading staff instead of the collapsible type.
- Be cautious of areas where there are submerged or partially submerged trees/tree branches – these can create entanglement hazards during a crossing or a "swim".

- If streams crossed are deeper than "knee deep", personnel must use either ropes and/or wear waders.
- Choose the right waders (with RHSM/SL involvement).
- Footwear with felt-bottom soles are ideal for rocky bottom streams. The rough texture cuts through algae growing on the rocks and grips well. For very slippery conditions, consider studded felt soles or a slipover, studded sandal. However, felt soles do not provide good traction on muddy, slippery banks. Cleated soles work well for mud or sand bottom streams (a hard-molded tread pattern similar to a hiking boot).
- Wear a wading belt with chest waders to keep your waders from billowing out like a parachute; the currents will carry you and move you in ways you do not want to move.
- Never wade alone.
- If the wader fills with water, do not panic. Waders full of water weigh less in water than on land and the water inside does not add any weight as long as you are in the water. Also, a common fear is that air trapped in the waders will raise the feet higher than the head and force the face underwater is unfounded. Waders do streamline your legs and kicking is useless. Follow these steps if the waders fill with water:
  - Do not try to take them off in the water
  - In calm water, wade or swim to shore
  - In fast moving water, ride the current:
  - Pull your feet up in front of you, bend your knees
  - Point your feet downstream (so the feet, not the head will bounce off the rocks)
  - Sculling with your hands will help direct to the nearest shallow area
  - When you reach calm water, go ashore and empty your waders
  - Do not waste energy in the vertical position going for the bottom; this position is virtually impossible to maintain and leads quickly to exhaustion (major cause of drowning)
  - Concentrate on getting out of the water and not saving the equipment
- The higher the elevation you are at, the steeper the stream gradient is. This means the stream can rise quicker and return to lower flow more quickly.
- Always wait out a swollen stream if at all possible.
- If you do slip into the water and are being swept downstream, do not panic. Cold water will be a shock for 2-3 seconds. Pull your knees up, face your feet downstream and lean back, using your hands as best you can to navigate and get to the bank. Keep your head up; you do not want your head underwater banging into rocks. If you stay calm, you can reach water where you can stand up or swim to the bank.
- When walking along stream banks and not entering streams, wear work boots.Walk cautiously when wading in water. Always wear waders. Always check depth of water when wading. Avoid entering deep or fast moving water.
- When boating or using a barge, always properly wear PFDs. Keep weight centered in boat/kayak. Avoid sudden shifts in position or weight. Enter and exit the boat cautiously and one at a time.
- When loading/unloading boat from vehicle, avoid carrying and loading in a way which might cause back strain. Walk slowly and carefully when carrying equipment or the kayak.
- Ensure all personnel entering area are briefed of potential hazards before entering work area by the SL-HW.
- Do not overload the boat with personnel, equipment, and supplies.

- Plan path to avoid high angle entry and rock climbing; with the least amount of obstructions.
- Wear high traction safety footwear.
- Plan steps before making them.
- If stuck in mud, move slowly.
- Ensure good grip on boat and balance. Do not stand in boat.
- Vessel operations will be suspended if supervisors judge weather or current conditions become unsafe.
- Know obstructions and shallows. Proceed slowly.
- Boats should be handled by experienced personnel only.
- Moor or anchor boats securely when not in use.
- Load small craft evenly to avoid listing.
- Counterbalance small craft when pulling equipment or debris out of the water. Keep the vessel as level as possible.
- Keep boat free from tripping hazards.
- Be aware of boat position and movement and communicate with the operator.
- Good hygiene practices (Section 7.3) should be used at all times. Contact with the waste containers should be considered potentially contaminated. No eating, drinking, smoking, or chewing tobacco while performing this task.

8.21 Boating Safety

Personnel who will operate a boat during the course of a project will first demonstrate to the site manager that they are experienced in operating boats similar to those used for the project, and are knowledgeable of the USCG Boating Safety requirements (33 *Code of Federal Regulations* [CFR] Subchapter S). Project boats will be operated by experienced and licensed or certified USCG marine vessel operators only. Boat operators will also possess basic mechanical knowledge necessary to troubleshoot common mechanical problems that can and do occur. The boat operator will be responsible for the safety of all personnel on board the boat he or she is operating and for the integrity of all boat and safety equipment.

Each designated boat operator will give a safety briefing to all occupants of the boat before leaving the shore. Boats are to be occupied during use by a minimum of one qualified operator plus one additional person. The boat skipper has the final authority with regard to boat safety and navigational safety. Use the Marine Vessel Checklist provided as Table D-8-1 of this HSP to evaluate and verify necessary equipment before leaving shore.

Table D-8-1. Marine Vessel Checklist

Item	Yes	N/A
PFDs		
Visual Distress Signals		
Anchor and Anchor Line		
Sound-Producing Devices		
Navigation Lights and Shapes		

Item	Yes	N/A
Fire Extinguishers		
Alternative Propulsion (for example, paddles)		
Overall Vessel Condition Satisfactory		
Marine Sanitation Device		
Ropes and Buoys		
FA Kit and Bloodborne Pathogen Kit		
Nonslip Deck		
Personnel Access Ladder		

Note:

N/A = not applicable

8.21.1 Boat Requirements

All project boats will meet or exceed USCG requirements for safety equipment, as applicable to the operation and type of boat. These requirements are summarized in the following subsections for small craft (less than 40 feet in length).

8.21.1.1 Flame Arresters

All gasoline engines, except outboard motors, installed in a boat must have an approved flame arrestor (backfire preventer) fitted to the carburetor.

8.21.1.2 Sound Signaling Devices

Boats will carry at least one air horn or similar sound signaling device. Radio or cell phone communication must be in place as well.

8.21.1.3 Personal Flotation Devices

All personnel and passengers will wear an approved PFD at all times when operating or being transported in a boat. A positively buoyant wet suit or dry suit may be substituted for a PFD. PFDs will be Type II or higher (capable of turning its wearer in a vertical or slightly backward position in the water). In addition, each boat will be equipped with at least one Type IV PFD, designed to be thrown to a person in the water and grasped and held by the user until rescued. Two common examples of a Type IV PFD are (1) a buoyant boat cushion equipped with straps and (2) a float ring.

8.21.1.4 Fire Extinguishers

Each boat will carry at least one Type B-I or B-II fire extinguisher (for use in gasoline, oil, and grease fires) approved by Underwriters Laboratories. Each fire extinguisher will be inspected to ensure that it is sufficiently charged and that the nozzles are free and clear. Discharged fire extinguishers will be replaced or recharged immediately.

8.21.1.5 Emergency Planning

As part of the project HSP and AHAs, planned response actions must address potential emergencies such at fire, sinking, flooding, severe weather, man over-board, or hazardous material incidents.

The boat captain is required to assess the weather forecast for each day and monitor the weather conditions throughout the day while on the water. If the weather forecast is not favorable or conditions on the water become hazardous due the weather, the boat captain has the final authority to cease all boating operations and return to port until conditions improve and all personnel feel safe.

8.21.1.6 Load Capacity

Boats will not be loaded beyond the weight capacity (passengers and gear) printed on the USCG information plate attached to the stern. In addition, several factors must be considered when loading a boat: distribute the load evenly, keep the load low, do not stand up in a small boat or kayak, and do not overload the boat.

8.21.1.7 Tool Kit

All motorized boats will carry a tool kit sufficient for the boat operator to troubleshoot common mechanical problems such as fouled spark plugs, a flooded carburetor, or electrical shorts. Boats operated in remote areas will also carry appropriate spare parts (such as propellers, shear pins, patch kits, air pumps). The tool kit will be maintained by the boat operator, and supplies used up will be replaced immediately.

8.21.1.8 Communications

All boats operated will carry a two-way radio or cellular telephone that enables communication back to the field camp or other pre-established location.

8.21.1.9 Good Housekeeping

Personnel using a boat will properly stow and secure all gear and equipment against unexpected shifts when underway. Decks and open spaces must be kept clear and free from clutter and trash to minimize slip, trip, and fall hazards.

8.21.1.10 Fuel Management

Personnel will use the “one-third rule” in boating fuel management. Use one-third of the fuel to get to the destination, one-third to return, and keep one-third in reserve.

No smoking is permitted on board vessels or during refueling operations.

8.21.1.11 Pollution Control

The Refuse Act of 1989 prohibits the throwing, discharging, or depositing of any refuse matter of any kind (including trash, garbage, oil, and other liquid pollutants) into U.S. waters. The Federal Water Pollution Control Act prohibits the discharge of oil or hazardous substances in quantities that may be harmful into U.S. navigable waters. No person may intentionally drain oil or oily wastes from any source into the bilge of any vessel. Larger vessels equipped with toilet facilities must be equipped with a USCG approved marine sanitation device.

Employees will report any significant oil spills to water to the PM who must report the spill to the USCG or other applicable regulatory agency. The procedure for incident reporting and investigation will be followed when reporting the spill.

8.22 Wildfire Smoke

Wildfire smoke can irritate your respiratory system and cause an immune response, which may lead to inflammation that affects other parts of your body. Common symptoms include eye irritation, runny nose, sore throat, mild cough, phlegm production, wheezy breathing, or headaches. If wildfire smoke is present in your area, the following thresholds and protective measures will be established:

Determine what your current regional Air Quality Index (AQI) is by researching the Internet for your local air quality district's website. There you should find a listing of updated information on the region's AQI. Note: different districts and websites have different ways of expressing the AQI number, whether it's particulate matter 2.5 or 10, or a combination of both, and whether other elements are factored into the AQI, so follow what the website lists as the "AQI."

AQI < 150 micrograms per cubic meter (µg/m^3) → No special action required; however, if experiencing irritation from smoke, don the N95 mask as stated below and notify the HSM. Training is required per Appendix D of 29 CFR 1910.134.

AQI > 150 < 500 µg/m^3 → Don N95 filtering masks with one-way filter (voluntary use)

AQI ≥ 500 µg/m^3 → Stop work; Notify PM and HSM (non-emergency work might be halted)

8.23 Safe Diving Operations

8.23.1 Applicability

Diving is defined as the activity when an employee works under water using an apparatus which supplies compressed breathing gas at ambient pressure. Field experience demonstrates that Jacobs personnel may be exposed to various hazards while diving to perform their related work. The related standard operating procedure applies to diving and related support operations conducted in connection with all types of work and employment, including general industry, construction, research, and field investigative activities.

8.23.2 Regulatory Drivers

- OSHA Regulations 29 CFR 1910, Subpart T
- Some State-OSHA plans may have more requirements. When state requirements differ from Federal, these differences will be identified. Prior to performing and diving operations, these should be referenced.

8.23.3 Training Requirements:

- Each dive team member shall have experience or training necessary to perform assigned tasks in a safe and healthful manner.
- Training certification will be based on the dive and work plans. State or private organizations may be consulted for this requirement.
- In some instances, Jacobs personnel or a Safety Liaison may require specialized training based on the dive and work plans.

- FA/cardiopulmonary resuscitation (CPR) training will be mandatory for all dive team members.

8.23.4 **Competent Person Requirements:**

A diving competent person must be provided to oversee and inspect all diving activities. Jacobs generally requires that subcontractors provide their own competent person. The competent person must have training in and knowledge about the hazards associated with diving and use of protective systems, as well as the skills to perform the tasks required.

8.23.5 SCUBA Requirements

- Tank supplied with "normal" air
- Regulator set with emergency "octopus"
- Buoyancy Control Device (BCD) and weight belt
- PPE (i.e., outer garments) will be based on the work to be performed and temperature of the water

8.23.6 Planning Activities

- A separate Dive Plan will be developed for safe diving operations if applicable and will be attached to this HSP.
- A list shall be kept at the dive location of the telephone or call numbers of the following: an operational decompression chamber, nearest hospital, available physicians, acceptable means of transportation, and the nearest USCG Rescue Coordination Center if needed.
- FA supplies with standard FA handbook must be onsite, appropriate for diving operations, and approved by a physician.
- The pre-dive assessment will include the diving mode, surface and underwater conditions, breathing gas supply (including reserves), thermal protection, equipment and systems, dive team assignments, physical fitness of team members, repetitive dive designation or residual inert gas status of members, decompression and treatment procedures, and emergency procedures.
- Communication procedures will be determined as part of planning and incorporated into the Dive Plan.
- Equipment inspections will be performed before diving and inspection requirements will be detailed in the Dive Plan.

8.23.7 Safe Work Practices

- SCUBA Divers must have a BCD capable of maintaining them at the surface in a face-up position, a manually activated inflation source independent of the breathing supply, an oral inflation device, and an exhaust valve.
- Compressed gas cylinders shall be stored in a ventilated area, protected from excessive heat, and be secured from falling.
- A cylinder pressure gauge capable of being monitored by the diver during the dive shall be worn by each SCUBA diver. A timekeeping device shall be available at each dive location.
- Equipment modification, repair, test, calibration or maintenance service must be recorded by means of a tagging or logging system, and include the date and nature of work performed, and the name or initials of the person performing the work.
- The breathing gas supply system must be inspected prior to each dive, including reserve breathing gas supplies, masks, thermal protection, and other equipment associated with the dive operation.

- A capable means of supporting the diver must be provided for entering and exiting the water. The means provided for exiting the water must extend below the water surface and a means must be provided to assist an injured diver from the water.
- The dive operation must be terminated when a diver requests termination, a diver fails to respond correctly to communications or signals from a dive team member, communications are lost between any parties onsite, or a diver begins to use diver-carried or dive location reserve breathing gas.
- No diving will be performed at depths deeper than 130 feet sea water.
- When diving at depths deeper than 100 feet sea water or outside the no-decompression limits, a decompression chamber must be near and serviceable.
- No diving will be performed when currents exceed 1 knot unless line-tended.
- There will be no diving in enclosed or physically confining spaces unless line-tended and they are first evaluated by the HSM.
- A standby diver shall be available while a diver is in the water, or there will be a diving buddy.
- A diver shall be line-tended from the surface or accompanied by another diver in continuous visual contact during the diving operation (buddy).
- A diver-carried reserve breathing gas supply shall be provided for each diver or each diver will have a separate regulator connection (octopus) to the underwater breathing apparatus.
- Except when heavy gear is worn, divers shall be equipped with a weight belt or assembly capable of quick release.

9. Physical Hazards and Controls

This section provides information on physical hazards and controls.

9.1 Ultraviolet (UV) Radiation (Sun Exposure)

Health effects regarding UV radiation are confined to the skin and eyes. Overexposure can result in many skin conditions, including erythema (redness or sunburn), photoallergy (skin rash), phototoxicity (extreme sunburn acquired during short exposures to UV radiation while on certain medications), premature skin aging, and numerous types of skin cancer. Implement the controls detailed in the following subsections to avoid sunburn.

9.1.1 Limit Exposure Time

- Rotate staff so the same personnel are not exposed all of the time.
- Limit exposure time when UV radiation is at peak levels (approximately 2 hours before and after the sun is at its highest point in the sky).
- Avoid exposure to the sun or take extra precautions when the UV index rating is high.

9.1.2 Provide Shade

- Take lunch and breaks in shaded areas.
- Create shade or shelter through the use of umbrellas, tents, and canopies.
- Fabrics such as canvas, sailcloth, awning material, and synthetic shade cloth create good UV radiation protection.
- Check the UV protection of the materials before buying them. Seek protection levels of 95 percent or greater and check the protection levels for different colors.
- If UV and/or heat strain become too intense onboard the boat, and personnel cannot seek adequate protection or shade within the cabin of the boat, boating operations will cease until there are favorable conditions or provisions to protect staff can be made.

9.1.3 Clothing

- Reduce UV radiation damage by wearing proper clothing; for example, long-sleeved shirts with collars and long pants. The fabric should be closely woven and should not let light through.
- Head protection should be worn to protect the face, ears, and neck. Wide-brimmed hats with a neck flap or “Foreign Legion” style caps offer added protection.
- Wear UV-protective sunglasses or safety glasses. These should fit closely to the face. Wrap-around style glasses provide the best protection.

9.1.4 Sunscreen

- Apply sunscreen generously to all exposed skin surfaces at least 20 minutes before exposure, allowing time for it to adhere to the skin.
- Re-apply sunscreen at least every 2 hours, and more frequently when sweating or performing activities where sunscreen may be wiped off.

- Choose a sunscreen with a high sun protection factor (SPF). Most dermatologists advocate SPF 30 or higher for significant sun exposure.
- Waterproof sunscreens should be selected for use in or near water, and by those who perspire sufficiently to wash off non-waterproof products.
- Check for expiration dates, because most sunscreens are only good for about 3 years. Store in a cool place out of the sun.
- Remember—no sunscreen provides 100 percent protection against UV radiation. Other precautions must be taken to avoid overexposure.

9.2 Temperature Extremes

Each employee is responsible for the following:

- Recognizing the symptoms of heat or cold stress.
- Taking appropriate precautionary measures to minimize their risk of exposure to temperature extremes (refer to Sections 9.2.1 and 9.2.2).
- Communicating any concerns regarding heat and cold stress to their supervisor or SL.

9.2.1 Heat

Heat-related illnesses are caused by more than just temperature and humidity factors.

Physical fitness influences a person's ability to perform work under heat loads. At a given level of work, the more fit a person is, the less the physiological strain, the lower the heart rate, the lower the body temperature (indicates less retrained body heat—a rise in internal temperature precipitates heat injury), and the more efficient the sweating mechanism.

Acclimatization is the degree to which a worker's body has physiologically adjusted or acclimatized to working under hot conditions. Acclimatization affects their ability to do work. Acclimatized individuals sweat sooner and more profusely than un-acclimatized individuals. Acclimatization occurs gradually over 1 to 2 weeks of continuous exposure, but it can be lost in as little as 3 days in a cooler environment.

Dehydration reduces body water volume. This reduces the body's sweating capacity and directly affects its ability to dissipate excess heat.

The ability of a body to dissipate heat depends on the ratio of its surface area to its mass (surface area/weight). **Heat dissipation** is a function of surface area, while heat production depends on body mass. Therefore, overweight individuals (those with a low ratio) are more susceptible to heat-related illnesses because they produce more heat per unit of surface area than if they were thinner. Monitor these persons carefully if heat stress is likely.

When wearing **impermeable clothing**, the weight of an individual is not as important in determining the ability to dissipate excess heat because the primary heat dissipation mechanism, evaporation of sweat, is ineffective.

Table D-9-1. Symptoms and Treatment of Heat Stress

	Heat Syncope	Heat Rash	Heat Cramps	Heat Exhaustion	Heat Stroke
Signs and Symptoms	Sluggishness or fainting while standing erect or immobile in heat.	Profuse tiny raised red blister-like vesicles on affected areas, along with prickling sensations during heat exposure.	Painful spasms in muscles used during work (arms, legs, or abdomen); onset during or after work hours.	Fatigue, nausea, headache, giddiness; skin clammy and moist; complexion pale, muddy, or flushed; may faint on standing; rapid thready pulse and low blood pressure; oral temperature normal or low.	Red, hot, dry skin; dizziness; confusion; rapid breathing and pulse; high oral temperature.
Treatment	Remove to cooler area. Rest lying down. Increase fluid intake. Recovery usually is prompt and complete.	Use mild drying lotions and powders and keep skin clean for drying skin and preventing infection.	Remove to cooler area. Rest lying down. Increase fluid intake.	Remove to cooler area. Rest lying down, with head in low position. Administer fluids by mouth. Seek medical attention.	Cool rapidly by soaking in cool–but not cold–water. Call ambulance and get medical attention immediately!

9.2.1.1 Precautions

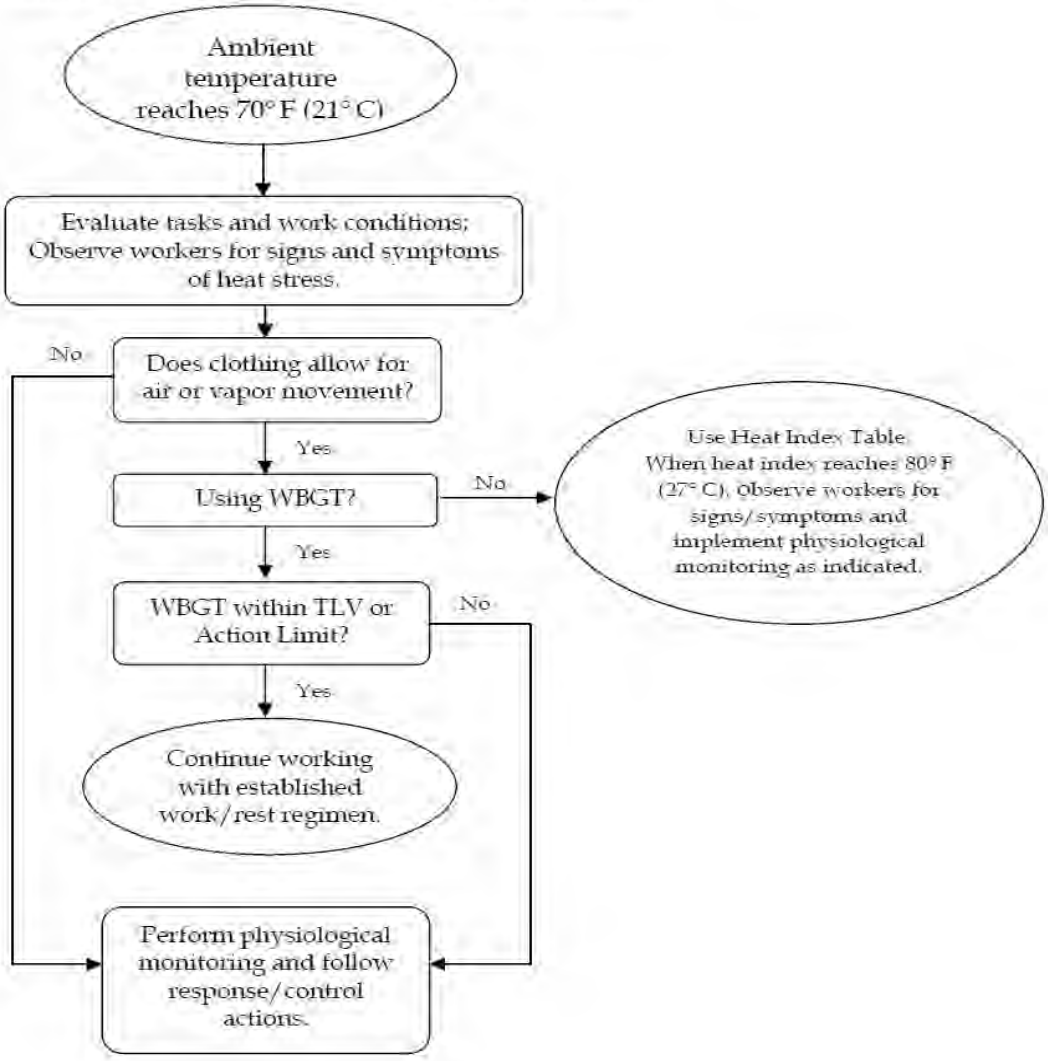
- Drink 16 ounces of water before beginning work. Disposable cups and water maintained at 50 degrees Fahrenheit (°F) (10 degrees Celsius [°C]) to 60°F (15.6°C) should be available. Under severe conditions, drink 1 to 2 cups every 20 minutes, for a total of 1 to 2 gallons (7.5 liters) per day. Do not use alcohol in place of water or other nonalcoholic fluids. Decrease your intake of coffee and caffeinated soft drinks during working hours.
- Acclimate yourself by slowly increasing workloads (e.g., do not begin with extremely demanding activities).
- Use cooling devices, such as cooling vests, to aid natural body ventilation. These devices add weight, so their use should be balanced against efficiency.
- Use mobile showers or hose-down facilities to reduce body temperature and cool protective clothing.
- Conduct field activities in the early morning or evening and rotate shifts of workers, if possible.
- Avoid direct sun whenever possible, which can decrease physical efficiency and increase the probability of heat stress. Take regular breaks in a cool, shaded area. Use a wide-brim hat or an umbrella when working under direct sun for extended periods.
- Provide adequate shelter/shade to protect personnel against radiant heat (sun, flames, hot metal).
- Maintain good hygiene standards by frequently changing clothing and showering.
- Observe one another for signs of heat stress. Persons who experience signs of heat syncope, heat rash, or heat cramps should consult the SL to avoid progression of heat-related illness.

9.2.1.2 Thermal Stress Monitoring

The following procedures should be implemented when the ambient air temperature exceeds 70°F (21°C), the relative humidity is high (greater than 50 percent), or when the workers exhibit symptoms of heat stress:

- The radial pulse should be used to measure heart rate for 30 seconds, as early as possible in the resting period.
- The heart rate at the beginning of the rest period should not exceed 110 beats per minute, or 20 beats per minute above resting pulse.
- If the heart rate is higher, the next work period should be shortened by 33 percent, while the length of the rest period stays the same.
- If the pulse rate still exceeds 110 beats per minute at the beginning of the next rest period, the next work cycle should be further shortened by 33 percent.
- Continue this procedure until the rate is maintained below 110 beats per minute, or 20 beats per minute above resting pulse.
- Alternately, the oral temperature can be measured before the workers have something to drink.
- If the oral temperature exceeds 99.6°F (37.6°C) at the beginning of the rest period, the next work cycle should be shortened by 33 percent.
- Continue this procedure until the oral temperature is maintained below 99.6°F (37.6°C). While an accurate indication of heat stress, oral temperature is difficult to measure in the field.

Thermal Stress Monitoring Flow Chart



9.2.2 Cold

Precautions related to cold exposure are presented in this section.

9.2.2.1 General

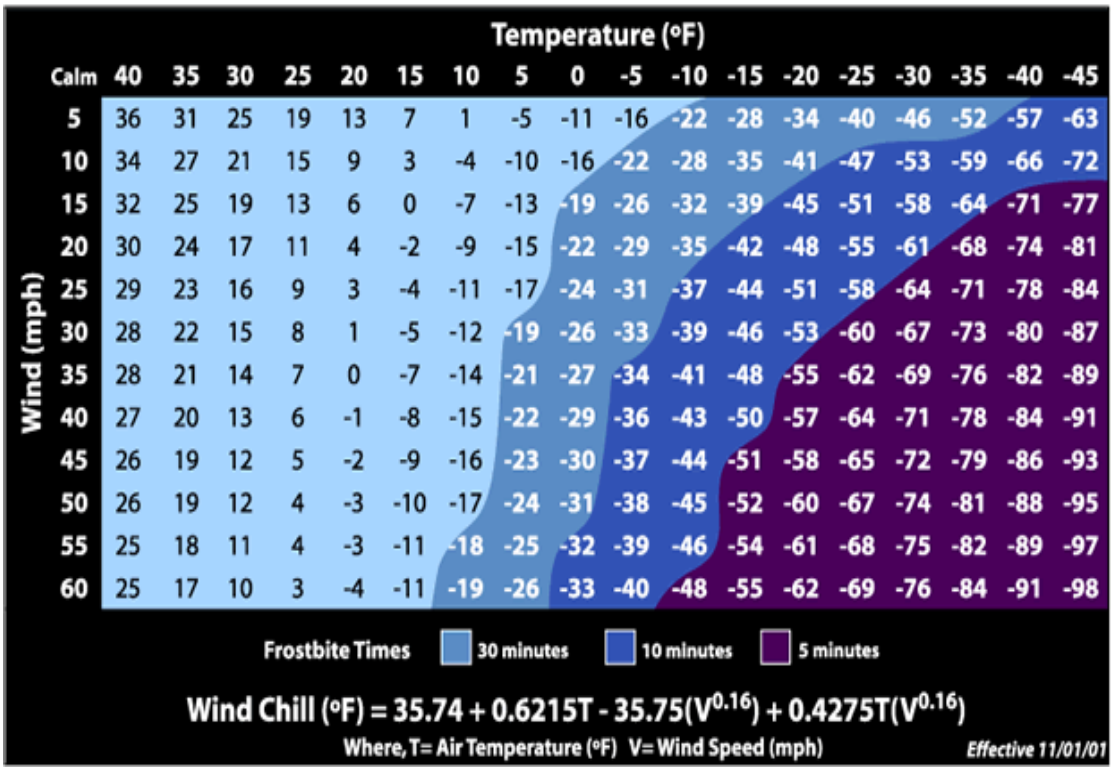
- Low ambient temperatures increase the heat lost from the body to the environment by radiation and convection. In cases where the worker is standing on frozen ground, the heat loss is also due to conduction.
- Wet skin and clothing, whether because of water or perspiration, may conduct heat away from the body through evaporative heat loss and conduction. Thus, the body cools suddenly when chemical protective clothing is removed if the clothing underneath is perspiration soaked.
- Movement of air across the skin reduces the insulating layer of still air just at the skin's surface. Reducing this insulating layer of air increases heat loss by convection.
- Non-insulating materials in contact or near contact with the skin, such as boots constructed with a metal toe or shank, conduct heat rapidly away from the body.
- Certain common drugs, such as alcohol, caffeine, or nicotine, may exacerbate the effects of cold, especially on the extremities. These chemicals reduce the blood flow to peripheral parts of the body, which are already high risk areas because of their large surface area to volume ratios. These substances may also aggravate an already hypothermic condition.

9.2.2.2 Precautions

- Be aware of the symptoms of cold-related disorders and wear proper layered clothing for the anticipated fieldwork. Appropriate rain gear is a must in wet weather.
- Wind-Chill Index (chart provided below) is used to estimate the combined effect of wind and low air temperatures on exposed skin. The wind-chill index does not consider the body part that is exposed, the level of activity, or the amount or type of clothing worn. For those reasons, it should only be used as a guideline to warn workers when they are in a situation that can cause cold-related illnesses.
- Persons who experience initial signs of immersion foot, frostbite, and/or hypothermia should report it immediately to their supervisor/PM to avoid progression of cold-related illness. Observe one another for initial signs of cold-related disorders.
- Obtain and review weather forecast – be aware of predicted weather systems along with sudden drops in temperature, increase in winds, and precipitation.

Table D-9-2. Symptoms and Treatment of Cold Stress

	Immersion (Trench) Foot	Frostbite	Hypothermia
Signs and Symptoms	Feet discolored and painful; infection and swelling present.	Blanched, white, waxy skin, but tissue resilient; tissue cold and pale.	Shivering, apathy, sleepiness; rapid drop in body temperature; glassy stare; slow pulse; slow respiration.
Treatment	Seek medical treatment immediately.	Remove victim to a warm place. Re-warm affected area quickly in warm–but **not** hot–water. Have victim drink warm fluids, but **not** coffee or alcohol. Do not break blisters. Elevate the injured area and get medical attention.	Remove victim to a warm place. Have victim drink warm fluids, but **not** coffee or alcohol. Get medical attention.



9.3 Radiological Hazards

Refer to Jacobs' Core Standard, Radiological Control, and Radiological Controls Manual for additional requirements. There are no radiological hazards and therefore controls are not applicable.

10. Biological Hazards and Controls

Biological hazards are everywhere and change with the region and season. If you encounter a biological hazard that has not been identified in this plan, contact the RHSM so that a revision to this plan can be made. Whether it is contact with a poisonous plant, a poisonous snake, or a bug bite, do not take biological hazards lightly. If there is a chance of an allergic reaction or infection, or to seek medical advice on how to properly care for the injury, contact the occupational nurse at 1-888-449-7787.

10.1 COVID-19 and Other Communicable Disease Prevention

1) Review and follow Jacobs Global Security Companywide Travel Restriction, Preparedness Pamphlet and FAQ.
2) Review signs and symptoms of Coronavirus with all field staff. Utilize Centers for Disease Control and Prevention (CDC) guidance document to review COVID-19 awareness information.
3) If project team member has specific COVID-19 concerns (e.g., in high risk category, high risk locations), speak with PM and supervisor. Accommodations shall be made by the PM and supervisor.
4) If showing signs of COVID-19 (e.g., fever, dry cough, shortness of breath), do not report to the project site for work. Contact project SL, WorkCare (if you suspect it is work-related), supervisor, PM, and HSM.
5) Contact personal care physician for COVID-19 symptom reporting.
6) Monitor local public health agencies communications. Follow all local agency guidance and restrictions.
7) Ensure SL has cell phone number of all field staff for communication to project teams.
8) Identify project-specific telework opportunities where feasible (e.g., work from hotel room).
9) Based on new CDC recommendations, staff should wear a face covering (e.g., bandana, sewn mask, t-shirt, or non-N95 dust mask) if they cannot maintain a 6-foot physical distance from others when working in the field (not in-lieu of).
10) Frequent hand washing is required with soap and water. Hand wash facilities are typically required at temporary job locations (e.g., at porta-potty and field office). If a handwashing sink is not feasible (e.g., mobile staff), utilize disinfectant hand wipes or hand sanitizer.
11) Be familiar with cough and sneeze etiquette (i.e., cough or sneeze into elbow) and avoid touching eyes, nose, mouth.
12) Avoid sharing personal items with co-workers (e.g., cups).
13) Use disinfectant to frequently clean shared surfaces, including rental cars, hotel room touch points, and temporary office trailer touch points (e.g., door handles, workstations).
14) If entering residential project site locations, ask occupants if they have signs of illness prior to entry. Do not enter residential locations with occupants that have signs/symptoms.
15) At temporary office entry points, post CDC Stop the Spread, Social Distancing, and Hand Washing Notice Posters (provided at the end of this list).
16) Limit air travel.
17) Avoid mass transit.

18) Use local staff and contractors when possible. Minimize use of contractors residing in hot spot areas deploying to remote areas where incidences of community transmission are low or undetected. In this case consider additional testing/evaluation.

19) Do not carpool with others.

20) Avoid large crowds.

21) Increase social distancing – Use the 6-foot rule.

22) Do not shake hands. Use forms of non-contact greeting.

23) Try not to touch your face.

24) Augment site equipment for handwashing – Make sure you have soap and water handwashing facilities at your site or have access to facilities with running water, do not rely on hand sanitizer alone.

25) Keep site meetings to a minimum, update State and Locals via conference calls rather than site visits.

26) Conduct site meetings (e.g., morning safety tailgate) in an outdoor area for maximum ventilation

27) Do not hold Public Meetings.

28) Do not share PPE.

29) Masks or bandanas are encouraged to minimize face touching and distribution of any sneezed particles. N95 masks are less effective if any facial hair is present,

30) Place hand sanitizer near doorways to encourage use before touching door handles.

31) Use U.S. Environmental Protection Agency (EPA) registered cleaners (https://www.epa.gov/pesticide-registration/list-n-disinfectants-use-against-sars-cov-2) or alternative disinfectants as follows:

    a) Isopropyl alcohol (aka isopropanol or IPA), minimum 70 percent, recommended sit time 5 minutes

    b) Household hydrogen peroxide, minimum 3 percent (Note: opened/expired hydrogen peroxide will likely be less than 3 percent, start with unopened/unexpired bottle), recommended sit time 2 minutes (test as it may bleach fabrics)

    c) Quaternary ammonium, recommended sit time 5 to10 minutes dependent on the mixture (via spray bottle) (Note: not compatible with bleach)

    d) A 10 percent bleach (1 part household bleach [5 percent] to 9 parts water); recommended sit time up to 10 minutes depending on the label (spray bottle) (Note: not compatible with quaternary ammonium)

32) The virus lives on hard surfaces up to 3 days, but for a shorter duration on porous surfaces. Pay close attention to the wet cleaner contact time listed on the label and use of products that are incompatible (e.g., quaternary ammonium and bleach) and can create toxic byproducts (e.g., chlorine gas).

33) Disinfect site trailers, porta johns, handwash stations, etc. with EPA registered cleaners or the suggested alternatives listed above, paying close attention to all touch points several times a day for restrooms, at least once a day for workstations, and before switching users.

34) Disinfect vehicles (wipe down steering wheel, door handles, touch points, etc.) before check out and upon return with an EPA registered cleaner or one of the suggested alternatives listed above. Implement this for all vehicles (EPA, ERRs, START).

35) While at hotels, disinfect your own room with an EPA registered cleaner or one of the suggested alternatives listed above and use the NO HOUSEKEEPING Sign. Keep the number of people coming in and out of your room to a minimum.

36) Avoid hotel breakfast buffets. This invites too many people handling food and utensils.

37) Bring a cooler of food for meals or use drive-thru to minimize crowd exposure at restaurants. Restaurants are closing their dining areas and offering drive-in and takeout options only. Refer to local Public Health sites for information.

Note: All references are frequently updated. Check Jacobs and CDC COVID-19 webpages frequently for updates.

10.2 Bees and Other Stinging Insects

Bees and other stinging insects may be encountered almost anywhere and may present a serious hazard, particularly to people who are allergic. Watch for and avoid nests. Keep exposed skin to a minimum. For known allergies, carry the appropriate medication (e.g., EpiPen) and inform teammates of its location in the event of an emergency as well as the severity of your allergy. If you are stung, contact the occupational nurse at 1-888-449-7787. If a stinger is present, remove it carefully with tweezers. Wash and disinfect the wound, cover it, and apply ice. Watch for an allergic reaction if you have never been stung before. Call 911 if the reaction is severe.

10.3 Feral Dogs

Avoid all dogs – both leashed and stray. Do not disturb a dog while it is sleeping, eating, or caring for puppies. If a dog approaches to sniff you, stay still. An aggressive dog has a tight mouth, flattened ears and a direct stare. If a dog threatens you, remain calm, do not scream, and avoid eye contact. If you say anything, speak calmly and firmly. Do not turn and run, try to stay still until the dog leaves, or back away slowly until the dog is out of sight or you have reached safety (e.g., a vehicle). If attacked, retreat to a vehicle or attempt to place something between you and the dog. If you fall or are knocked to the ground,

curl into a ball with your hands over your head and neck and protect your face. If bitten, contact the occupational nurse at 1-888-449-7787. Report the incident to the local authorities.

10.4 Bloodborne Pathogens

Exposure to bloodborne pathogens may occur when rendering FA or CPR, or when coming into contact with landfill waste or waste streams containing potentially infectious material (PIM).

Employees trained in FA/CPR or exposed to PIM must complete Jacobs' 1-hour bloodborne pathogen computer-based training module annually. When performing FA/CPR the following shall apply:

- Observe universal precautions to prevent contact with blood or other PIMs. Where differentiation between body fluid types is difficult or impossible, consider all body fluids to be PIMs.
- Always wash your hands and face with soap and running water after contacting PIMs. If washing facilities are unavailable, use an antiseptic cleanser with clean paper towels or moist towelettes.
- If necessary, decontaminate all potentially contaminated equipment and surfaces with chlorine bleach as soon as possible. Use 1 part chlorine bleach (5.25 percent sodium hypochlorite solution) diluted with 10 parts water for decontaminating equipment or surfaces after initially removing blood or other PIMs. Remove contaminated PPE as soon as possible before leaving a work area.

Jacobs will provide exposed employees with a confidential medical examination should an exposure to PIM occur. This examination includes the following procedures:

- Documenting the exposure
- Testing the exposed employee's and the source individual's blood (with consent)
- Administering post-exposure prophylaxis

10.5 Infectious Pathogens

Exposure to pathogens may occur when rendering FA or CPR, when coming into contact with landfill waste, or when working near sanitary waste streams containing PIM. Exposure control includes using basic hazardous waste characterization PPE (e.g., nitrile gloves, safety glasses, covering exposed skin, etc.); refer to Section 13 for PPE guidelines during site activities. If you encounter what appears to be medical waste and/or drug paraphernalia, DO NOT touch! If you encounter human waste on the ground or within a water body, DO NOT touch and avoid contact. Contact the HSM for further guidance. Prudent vaccinations will be offered before an employee participates in a task in which an exposure is possible. At a minimum, all employees should use good personal hygiene (e.g., wash hands regularly with warm, soapy water, especially before and after eating, smoking, and using the restroom) and report any inadvertent contact or needle sticks immediately.

10.6 Mosquito Bites

Mosquitoes are believed to be the primary source for exposure to the West Nile Virus as well as several other types of encephalitis. The following guidelines should be followed to reduce the risk of these concerns for working in areas where mosquitoes are prevalent:

- Stay indoors at dawn, dusk, and in the early evening.
- Wear long-sleeved shirts and long pants whenever you are outdoors.
- Spray clothing with repellents containing permethrin or N,N-diethyl-meta-toluamide (DEET) since mosquitoes may bite through thin clothing.

- Apply insect repellent sparingly to exposed skin. An effective repellent will contain 35 percent DEET. Repellents may irritate the eyes and mouth, so avoid applying repellent to the hands.
- Whenever you use an insecticide or insect repellent, be sure to read and follow the manufacturer's DIRECTIONS FOR USE, as printed on the product.
- Note: Vitamin B and "ultrasonic" devices are NOT effective in preventing mosquito bites.

11. Contaminants of Concern

Table D-11-1 summarizes the contaminants of concern, their occupational exposure limit, and the signs and symptoms of exposure. The table also includes the maximum concentration of each contaminant of concern and the associated location and media that was sampled (e.g., groundwater, soil boring, surface soil). These concentrations were used to determine engineering and administrative controls described in the "Project-Specific Hazard Controls" section of this HSP, as well as PPE and site monitoring requirements.

Table D-11-1. Contaminants of Concern

Contaminant	Location and Maximum[a] Concentration	Exposure Limit[b]	IDLH[c]	Symptoms and Effects of Exposure	PIP[d] (eV)
Gasoline Range Organics	Potential	300 ppm	ND Ca	Eye, skin and mucous membrane irritation; dermatitis, headache, fatigue, blurred vision, dizziness, slurred speech, confusion, convulsions, chemical pneumonia on aspiration, possible liver and kidney damage.	UK
4,4-dichlorodiphenyldichloroethane (DDD)	Potential	NL	NL	No information found in reference material.	UK
4,4-dichlorodiphenyldichloroethene (DDE)	Potential	NL	NL	No information found in reference material.	UK
Dichlorodiphenyltrichloroethane (DDT)	Potential	1 mg/m^3	500 Ca	Paresthesia of tongue, lips, hand, and face; tremors; dizziness; confusion; headache; fatigue; convulsions; eye and skin irritation; vomiting.	UK
Diesel Range Organics	Potential	100 mg/m^3 (REL)	NL	Primary system effect is central nervous system depression. Inhalation of vapors may cause nausea, confusion, drowsiness, convulsions, and coma. Liquid may cause skin and eye irritation.	UK
Benzo(a)pyrene (as Coal Tar Pitch Volatiles)	Potential	0.2 mg/m^3	NL Ca	Skin irritation and carcinogen.	UK
2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) (Dioxin/Furans)	Potential in saturated sediment	Ca	Ca	Eye irritation, nausea, vomiting, abdominal pain, allergic dermatitis, teratogen effects, mutagen effects, carcinogenic effects.	UK
Polychlorinated biphenyls (PCBs)	SD: 0.5 mg/kg	0.5 mg/m^3	5 Ca	Eye and skin irritation, acne-form dermatitis, liver damage, reproductive effects.	UK

Notes:

[a] Specify sample-designation and media: SB (Soil Boring), A (Air), D (Drums), GW (Groundwater), L (Lagoon), TK (Tank), SS (Surface Soil), SL (Sludge), SW (Surface Water), SD (Sediment).

[b] Appropriate value of permissible exposure limit, recommended exposure limit, or threshold limit value listed.

[c] IDLH = immediately dangerous to life and health (units are the same as specified "Exposure Limit" units for that contaminant); NL = No limit found in reference materials; ND = Not determined for the substance; Ca = Potential occupational carcinogen.

[d] eV = electron volt; mg/kg = milligrams per kilogram; mg/m^3 = milligrams per cubic meter;; PIP = photoionization potential; ppm = parts per million; UK = Unknown.

Potential Routes of Exposure

Dermal: Contact with contaminated media. This route of exposure is minimized through use of engineering controls, administrative controls and proper use of PPE.	**Inhalation:** Vapors and contaminated particulates. This route of exposure is minimized through use of engineering controls, administrative controls and proper use of respiratory protection when other forms of control do not reduce the potential for exposure.	**Other:** Inadvertent ingestion of contaminated media. This route should not present a concern if good hygiene practices are followed (e.g., wash hands and face before drinking or smoking).

12. Site Monitoring

When performing site monitoring, record all the information, such as in a field logbook. Note date and time, describe monitoring location (e.g., in breathing zone, at source, site location, etc.), and what action/result is being recorded. If any action levels are reached, note it in the field logbook and note the action taken.

Copies of all project exposure records (e.g., copies of field logbook pages where air monitoring readings are recorded and associated calibration) shall be maintained in the project files.

12.1 Air Monitoring Specifications (Field Characterization Tasks)

Air monitoring specification are provided in Table D-12-1, below.

Table D-12-1. Air Monitoring Specifications

Instrument	Tasks	Action Levels[a]		Frequency[b]	Calibration
PID: MiniRAE or MultiRAE with 10.6 eV lamp	• Vibracoring • Sediment sampling • Riverbank soil sampling	<5 ppm → Level D ≥5 ppm → Stop work; Notify HSM		Initially and periodically during task	Daily
O_2: MultiRAE or equivalent[c]	• Vibracoring • Sediment sampling • Riverbank soil sampling	20.9% → >23.5% →	Normal O_2 Fire Hazard; notify HSM	Initially and periodically during task	Daily
LEL/CGI: MultiRAE or equivalent[c]	• Vibracoring • Sediment sampling • Riverbank soil sampling	<10% LEL: → >10% LEL: →	No explosion hazard Explosion hazard; evacuate or vent	Initially and periodically during task	Daily
Dust Monitoring: Visual	During all invasive tasks	No Visual Dust → Continue operations Visual Dust → Implement control measures		Continuous	N/A

Notes:

[a] Action levels apply to sustained breathing-zone measurements above background for more than 5 minutes.

[b] The exact frequency of monitoring depends on field conditions and is to be determined by the SL-HW; generally, every 5 to 15 minutes if acceptable; it may be appropriate to do so more frequently. Monitoring results should be recorded. Documentation should include instrument and calibration information, time, measurement results, personnel monitored, and place/location where measurement is taken (for example, "Breathing Zone/MW-3," "at surface/SB-2," etc.).

[c] If the measured percent of O_2 is less than 10, an accurate LEL reading will not be obtained. Percent LEL and percent O_2 action levels apply only to ambient working atmospheres, and not to confined space entry. More stringent percent LEL and O_2 action levels are required for confined space entry.

CGI = combustible gas indicator; LEL = lower explosive limit; O_2 = oxygen PID = photoionization detector

12.2 Calibration Specifications

For calibration specifications, refer to the respective manufacturer's instructions for proper instrument-maintenance procedures.

Instrument	Gas	Span	Reading	Method
PID: MiniRAE, 10.6 eV bulb	100 ppm isobutylene	CF=53	53 ppm ±5 ppm	1.5 ppm REG T-Tubing
PID: MultiRAE 10.6 eV bulb	Per Manufacturer's Specification			

Calibrate air monitoring equipment daily (or prior to use) in accordance with the instrument's instructions. Document the calibration in the field logbook (or equivalent) and include the following information:

- Instrument name
- Serial Number
- Owner of instrument (e.g., Jacobs, HAZCOM)
- Calibration gas (including type and lot number)
- Type of regulator (e.g., 1.5 ppm)
- Type of tubing (e.g., direct or T-tubing)
- Ambient weather condition (e.g., temperature and wind direction)
- Calibration/instrument readings
- Operator's name and signature
- Date and time

12.3 Air Sampling

Method Description: Based on current site conditions, recent analytical data, and the tasks planned in this work order, air sampling will not be required at this time. If site conditions or tasks change, notify the HSM to reevaluate the need for air sampling.

13. Personal Protective Equipment

PPE requirements are as follows:

- PPE must be worn by employees when actual or potential hazards exist and engineering controls or administrative practices cannot adequately control those hazards.
- Employees must be trained to properly wear and maintain the PPE.
- PPE must be inspected prior to use and after any occurrence to identify any deterioration or damage.
- PPE must be maintained in a clean and reliable condition.
- Damaged PPE shall not be used and must either be repaired or discarded.
- PPE shall not be modified, tampered with, or repaired beyond routine maintenance.
- Refer to Table D-13-1 PPE Specifications for further requirement details.

Table 13-1. PPE Specifications[a]

Task	Level	Body	Head	Respirator[b]
• Aerial surveying with drone • Surveying • Utility locating Note: with no exposure to site contaminants	Non-HW	Work clothes; steel-toe, leather work boots; work gloves. **Gloves:** Chemical-resistant nitrile gloves when sampling **Personal Flotation Device**: A USCG approved PFD required when there is a risk of falling into the water (e.g., onboard a boat, on a pier, or on the riverbank when there is a steep slope in the water); or if entering water above the knees. **Waders**: If entering the water along the riverbanks	Hardhat Safety glasses Ear protection[d]	None required
• Vibracoring • Sediment sampling • River bank soil sampling	Modified D	**Coveralls:** Cotton coveralls; or uncoated Tyvek if cotton cannot be kept clean. Rain gear can be used as a substitute **Boots:** Steel-toe, chemical-resistant boots OR steel-toe, leather work boots **Gloves:** Inner surgical-style nitrile and outer chemical-resistant nitrile gloves. Work gloves if handling heavy or sharp metal objects. **Personal Flotation Device**: A USCG approved PFD required when there is a risk of falling into the water (e.g., onboard a boat, on a pier, or on the riverbank when there is a steep slope in the water); or if entering water above the knees. **Waders**: If entering the water along the riverbanks	Hardhat Safety glasses Face shield[c] Ear protection[d]	None required (unless Wildfire Smoke is present; refer to Section 8.23 for further guidance)
• Underwater sediment sampling (potential diving operations)	Modified D	If this operation is carried-out, a separate Dive Plan will be developed prior to mobilization, and attached to this HSP. All PPE requirements will be addressed in that document.		

[a] Modifications are as indicated. Jacobs will provide PPE only to Jacobs employees (Refer to the *JACOBS Personal Protective Equipment Reimbursement Guidelines*).

[b] No facial hair that would interfere with respirator fit is permitted.

[c] Areas requiring the use of face shields are to be determined by the SL-HW.

[d] Ear protection should be worn when conversations cannot be held at distances of 1 meter (3 feet) or less without shouting.

[e] Performing a task that requires an upgrade to a higher level of protection (e.g., Level D to Level C) is permitted only when the PPE requirements have been approved by the HSM, and an SL-HW qualified at that level is present.

Reasons for Upgrading or Downgrading Level of Protection

Upgrade[e]	Downgrade
• Request from individual performing tasks. • Change in work tasks that will increase contact or potential contact with hazardous materials. • Occurrence or likely occurrence of gas or vapor emission. • Known or suspected presence of dermal hazards. • Instrument action levels (Section 12) exceeded.	• New information indicates that situation is less hazardous than originally thought. • Change in site conditions that decreases the hazard. • Change in work task that will reduce contact with hazardous materials.

14. Worker Training and Qualification

14.1 Jacobs Worker Training

14.1.1 Hazardous Waste Operations Training

All employees engaging in hazardous waste operations or emergency response shall receive appropriate training as required by 29 CFR 1910.120 and 29 CFR 1926.65. At a minimum, the training shall have consisted of instruction in the topics outlined in 29 CFR 1910.120 and 29 CFR 1926.65. Personnel who have not met these training requirements shall not be allowed to engage in hazardous waste operations or emergency response activities.

14.1.1.1 Initial Training

General site workers engaged in hazardous waste operations shall, at the time of job assignment, have received a minimum of 40 hours of initial H&S training for hazardous waste site operations, unless otherwise noted in the above-referenced standards.

Employees who may be exposed to health hazards or hazardous substances at treatment, storage, and disposal (TSD) operations shall receive a minimum of 24 hours of initial training to enable the employee to perform their assigned duties and functions in a safe and healthful manner.

Employees engaged in emergency response operations shall be trained to the level of required competence in accordance with 29 CFR 1910.120.

14.1.1.2 Three-Day Actual Field Experience

General site workers for hazardous waste operations shall have received three days of actual experience (on-the-job training) under the direct supervision of a trained, qualified supervisor and shall be documented. If the field experience has not already been received and documented at a similar site, this supervised experience shall be accomplished and documented at the beginning of the assignment of the project.

14.1.1.3 Refresher Training

General site workers and TSD workers shall receive 8 hours of refresher training annually (within the previous 12-month period) to maintain qualifications for fieldwork. Employees engaged in emergency response operations shall receive annual refresher training of sufficient content and duration to maintain their competencies or shall demonstrate competency in those areas at least annually.

14.1.1.4 Eight-Hour Supervisory Training

Onsite management or supervisors, who will be responsible for or supervise employees engaged in hazardous waste site operations, will have received at least 8 hours of additional specialized training on managing such operations. Employees designated as SL-HW are considered 8-hour HAZWOPER Site Safety Supervisor trained.

14.1.2 First Aid/CPR

FA and CPR training consistent with the requirements of a nationally recognized organization such as the American Red Cross Association or National Safety Council shall be administered by a certified trainer. A minimum of two personnel per active field operation will have FA and CPR training. Bloodborne pathogen training located on Jacobs' Virtual Office is also required for those designated as FA/CPR trained.

14.1.3 SL Training

SLs are trained to implement the HSE program on Jacobs field projects. A qualified SL is required to be identified in the site-specific HSP/field safety instructions (FSIs) for Jacobs field projects. SLs must also meet the requirements of the worker category appropriate to the type of field project (e.g., construction or hazardous waste). In addition, the SLs shall have completed additional safety training required by the specific work activity on the project that qualifies them to implement the HSE program (e.g., fall protection, excavation, etc.).

14.1.4 Site-Specific Training

Prior to commencement of field activities, all field personnel assigned to the project will have completed site-specific training that will address the contents of applicable HSPs, including the activities, procedures, monitoring, and equipment used in the site operations. Site-specific training will also include site and facility layout, potential hazards, risks associated with identified emergency response actions, and available emergency services. This training allows field workers to clarify anything they do not understand and to reinforce their responsibilities regarding safety and work operations for their particular activity.

14.1.5 Project-Specific Training Requirements

Project-specific training for this project includes:

- SL Training – The Jacobs SL-HW must have current SL-HW.
- FA/CPR - The assigned SL-HW onsite must have current FA/CPR training.
- Fire Extinguisher - The assigned SL-HW onsite must take the online fire extinguisher training course.
- Waste Management - The assigned SL-HW onsite must take the online waste management training course.
- Bloodborne Pathogen - The assigned SL-HW must take the Jacobs online bloodborne pathogen training course.
- Dangerous Goods Shipping Training - The SL-HW onsite must take the online danger goods training course.
- Federal Aviation Administration (FAA) Training and License – Operators of aerial drones must maintain all necessary FAA training and licenses and file any necessary flight plans.
- Boating Safety Training – The boat captain will provide initial comprehensive safety training to all staff members traveling onboard the vessel; then daily briefings about safety that day and any lessons learned.

15. Medical Surveillance and Qualification

All site workers participating in hazardous waste operations or emergency response will maintain an adequate medical surveillance program in accordance with 29 CFR 1910.120 or 29 CFR 1926.65 and other applicable OSHA standards. Documentation of employee medical qualification (e.g., physician's written opinion) will be maintained in the project files and made available for inspection.

15.1 Hazardous Waste Operations and Emergency Response

Jacobs personnel expected to participate in onsite hazardous waste operations or emergency response are required to have a current medical qualification for performing this work. Medical qualification shall consist of a qualified physician's written opinion regarding fitness for duty at a hazardous waste site, including any recommended limitations on the employee's assigned work. The physician's written opinion shall state whether the employee has any detected medical conditions that would place the employee at increased risk of material impairment of the employee's health from work in hazardous waste operations or emergency response, or from respirator use.

15.2 Job or Site-Specific Medical Surveillance

Due to the nature of hazards for a particular job or work site, specialized medical surveillance may be necessary. This surveillance could include biological monitoring for specific compounds, or specialized medical examinations.

15.3 Respirator User Qualification

Personnel required to wear respirators must have a current medical qualification to wear respirators. Medical qualification shall consist of a qualified physician's written opinion regarding the employee's ability to safely wear a respirator in accordance with 29 CFR 1910.134.

15.4 Hearing Conservation

Personnel working in hazardous waste operations or operations that fall under 29 CFR 1910.95 and exposed to noise levels in excess of the 85 dBA time-weighted average shall be included in a hearing conservation program that includes annual audiometric testing.

16. Site-Control Plan

16.1 Site-Control Procedures

- The SL will implement site-control procedures.
- The SL will conduct a site safety briefing before starting field activities or as tasks and site conditions change:
  - Topics for briefing on site safety include general discussion of the HSP, site-specific hazards, locations of work zones, PPE requirements, equipment, special procedures, and emergencies.
  - The SL records attendance at safety briefings in a logbook and documents the topics discussed.
- Establish support, contamination reduction, and exclusion zones. Delineate with flags or cones as appropriate. Support zone should be upwind of the site. Use access control at entry and exit from each work zone.
- Establish onsite communication consisting of the following:
  - Line-of-sight and hand signals
  - Air horn
  - Two-way radio or cellular telephone if available
- Establish offsite communication.
- Establish and maintain the "buddy system."
- The SL conducts initial air monitoring and determines appropriate level of protection.
- The SL is to conduct periodic inspections of work practices to determine the effectiveness of this plan. Deficiencies are to be noted, reported to the RHSM, and corrected.
- For protection of the general public, the site will implement the Site Security Plan dated December 20, 2010. Fence repairs and gates will be installed by the client around the perimeter of the site, and a security firm will be contracted by the client to provide periodic inspections and 24-hour security.
- The security team will have appropriate training on interfacing with the general public.
- The SL will instruct Jacobs staff members to immediately report breaches in security made by the general public during remediation activities, if not already recognized by the security team. The general public will be challenged before they can gain access to any exclusion zone or contamination reduction zone.
- Signage will be installed by the client at the main entrance reminding the general public for their own protection to remain outside the fenced area. A point of contact will also be provided in the event they have questions or concerns.
- Strict adherence to the air monitoring protocols in Section 12 will be followed.
- The boat captain is required to assess the weather forecast for each day and monitor the weather conditions throughout the day while on the water. If the weather forecast is not favorable or conditions on the water become hazardous due the weather, the boat captain has the final authority to cease all boating operations and return to port until conditions improve and all personnel feel safe.

16.2 HAZWOPER Compliance Plan

State or federal HAZWOPER standards cover certain parts of the site work; therefore, training and medical monitoring are required. Anticipated HAZWOPER tasks listed in the "General Project Information" section of this HSP might occur consecutively or concurrently with respect to non-HAZWOPER tasks (also specified in the "General Project Information" section).

This section outlines procedures to be followed when the approved non-HAZWOPER activities do not require 24- or 40-hour training; non-HAZWOPER-trained personnel also must be trained in accordance with all other state and federal OSHA requirements:

- In many cases, air sampling, in addition to real-time monitoring, must confirm that there is no exposure to gases or vapors before non-HAZWOPER-trained personnel are allowed on the site, or while non-HAZWOPER-trained staff is working in proximity to HAZWOPER activities. Other data (e.g., soil) also must document that there is no potential for exposure. The RHSM must approve the interpretation of these data.
- When non-HAZWOPER-trained personnel are at risk of exposure, the SL must post the exclusion zone and inform non-HAZWOPER-trained personnel of the:
  - Nature of the existing contamination and its locations
  - Limitations of their access
  - Emergency action plan for the site
- Periodic air monitoring with direct-reading instruments conducted during regulated tasks also should be used to ensure that non-HAZWOPER-trained personnel (e.g., in an adjacent area) are not exposed to airborne contaminants.
- When exposure is possible, non-HAZWOPER-trained personnel must be removed from the site until it can be demonstrated that there is no longer a potential for exposure to H&S hazards.
- Remediation treatment system start-ups: Once a treatment system begins to pump and treat contaminated media, the site is, for the purposes of applying the HAZWOPER standard, considered a treatment, storage, and disposal facility (TSDF). Therefore, once the system begins operation, only HAZWOPER-trained personnel (minimum of 24 hour of training) will be permitted to enter the site. All non-HAZWOPER-trained personnel must not enter the TSDF area of the site.

17. Decontamination

The SL must establish and monitor the decontamination procedures and their effectiveness. Decontamination procedures found to be ineffective will be modified by the SL. The SL must ensure that procedures are established for disposing of materials generated on the site.

17.1 Decontamination Specifications

Personnel

- Boot wash/rinse
- Glove wash/rinse
- Outer-glove removal
- Body-suit removal
- Inner-glove removal
- Respirator removal
- Hand wash/rinse
- Face wash/rinse
- Shower ASAP
- Dispose of PPE in municipal trash, or contain for disposal
- Dispose of personnel rinse water to facility or sanitary sewer, or contain for offsite disposal

Sample Equipment

- Wash/rinse equipment
- Solvent-rinse equipment
- Contain solvent waste for offsite disposal

Heavy Equipment

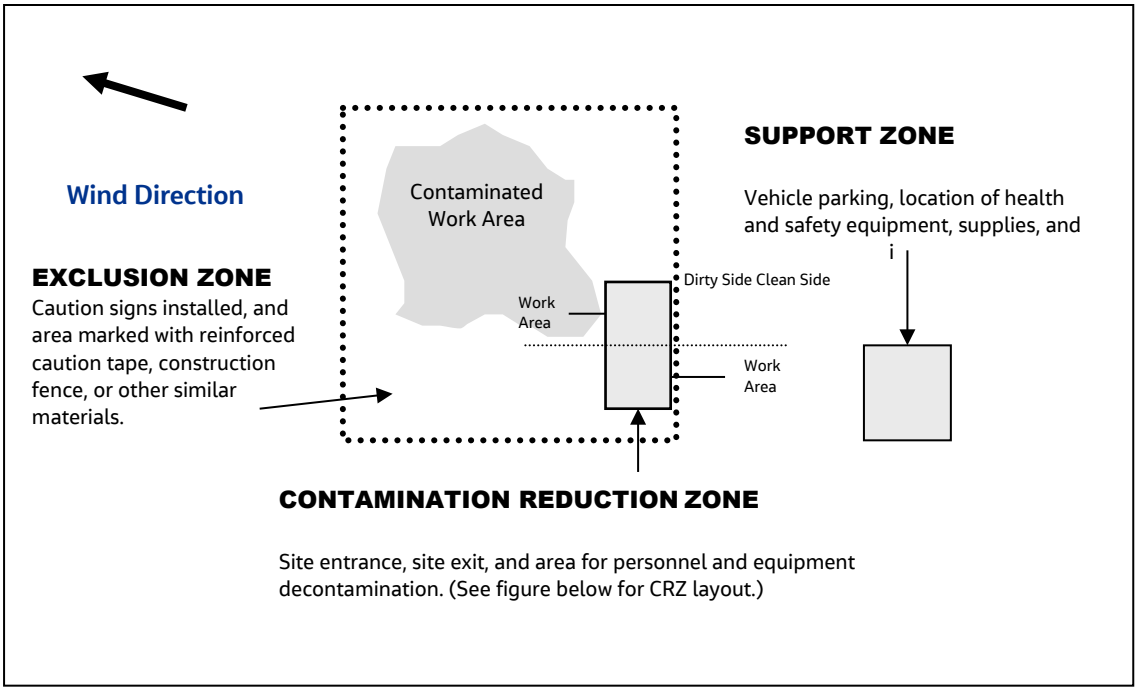
- Power wash
- Steam clean
- Dispose of equipment rinse water to facility or sanitary sewer, or contain for offsite disposal

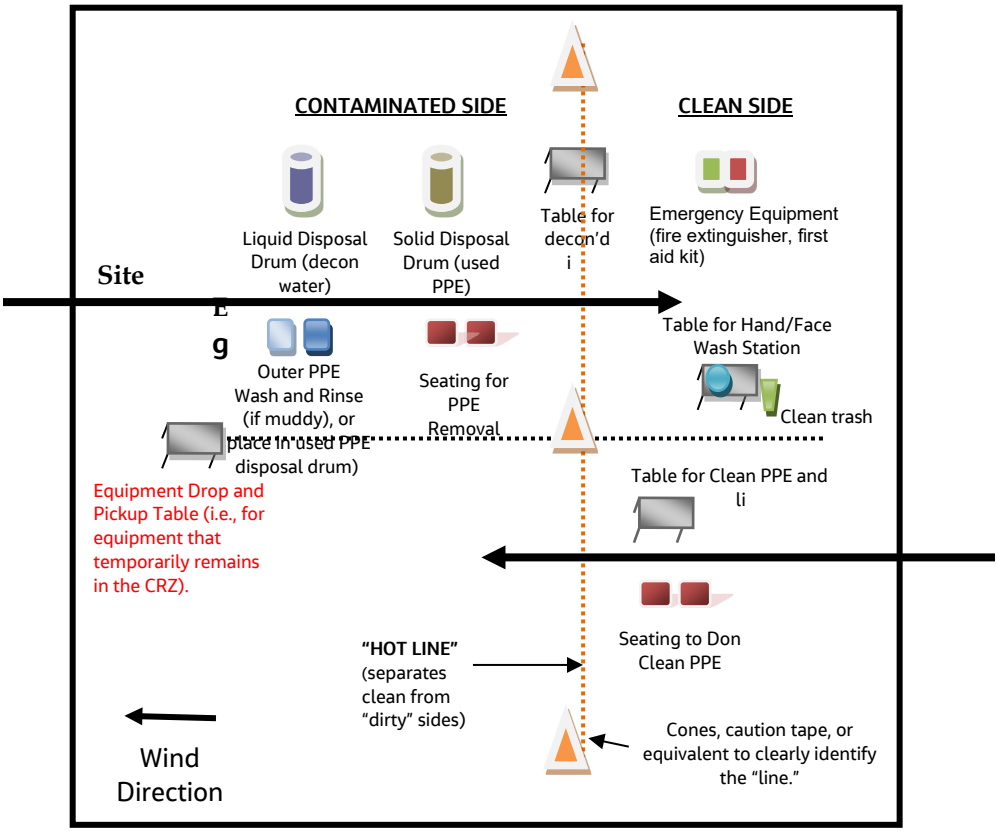
17.2 Diagram of Personnel-Decontamination Line

No eating, drinking, or smoking is permitted in contaminated areas and in exclusion or decontamination zones. The SL should establish areas for eating, drinking, and smoking. Contact lenses are not permitted in exclusion or decontamination zones.

The following figure illustrates a conceptual establishment of work zones, including the decontamination line. Work zones are to be modified by the SL to accommodate task-specific requirements.

Work Area - Set up appropriately based on wind direction





18. Emergency Response Plan

18.1 Pre-Emergency Planning

The ERC, typically the SL or designee, performs the applicable pre-emergency planning tasks before starting field activities and coordinates emergency response with Jacobs onsite parties, the facility, and local emergency-service providers as appropriate. Pre-Emergency Planning activities performed by the ERC include:

- Review the facility emergency and contingency plans where applicable.
- Determine what onsite communication equipment is available (e.g., two-way radio, air horn).
- Determine what offsite communication equipment is needed (e.g., nearest telephone, cell phone).
- Confirm and post the "Emergency Contacts" page and route to the hospital located in this section in project trailer(s) and keep a copy in field vehicles along with evacuation routes and assembly areas. Communicate the information to onsite personnel and keep it updated.
- Review changed site conditions, onsite operations, and personnel availability in relation to emergency response procedures.
- Where appropriate and acceptable to the client, inform emergency room and ambulance and emergency response teams of anticipated types of site emergencies.
- Designate one vehicle as the emergency vehicle; place hospital directions and map inside; keep keys in ignition during field activities.
- Inventory and check site emergency equipment, supplies, and potable water.
- Communicate emergency procedures for personnel injury, exposures, fires, explosions, and releases.
- Rehearse the emergency response plan before site activities begin, including driving route to hospital. Drills should take place periodically but no less than once a year.
- Brief new workers on the emergency response plan.
- The ERC will evaluate emergency response actions and initiate appropriate follow-up actions.

18.2 Emergency Equipment and Supplies

The ERC should mark the locations of emergency equipment on the site map and post the map.

Emergency Equipment and Supplies	Location
20 (or two 10) class A,B,C fire extinguisher	Required w/ heavy equipment and boat
FA kit	Field Vehicle
Bloodborne pathogen kit	Field Vehicle
Additional equipment (specify): Cell phone	Field Vehicle

18.3 Incident Response

In fires, explosions, or chemical releases, actions to be taken include the following:

- Notify appropriate response personnel.
- Shut down Jacobs operations and evacuate the immediate work area.

- Account for personnel at the designated assembly area(s).
- Assess the need for site evacuation, and evacuate the site as warranted.
- Notify and submit reports to clients as required in contract.

Small fires or spills posing minimal safety or health hazards may be controlled with onsite spill kits or fire extinguishers without evacuating the site. When in doubt, evacuate. Follow the incident reporting procedures in the "Incident Notification, Reporting, and Investigation" section of this HSP.

18.4 Emergency Medical Treatment

Emergency medical treatment is needed when there is a life-threatening injury (such as severe bleeding, loss of consciousness, breathing/heart has stopped). When in doubt if an injury is life-threatening or not, treat it as needing emergency medical treatment:

- Notify 911 or other appropriate emergency response authorities as listed in Emergency Contacts at the front of this HSP.
- The ERC will assume charge during a medical emergency until the ambulance arrives or until the injured person is admitted to the emergency room.
- Prevent further injury, perform decontamination (if applicable) where feasible; lifesaving, FA, and medical treatment takes priority.
- Initiate FA and CPR where feasible.
- Notify supervisor, and if the injured person is a Jacobs employee, the supervisor will call the occupational nurse at 1-888-449-7787 and make other notifications as required by Jacobs WIs.
- Make certain that the injured person is accompanied to the emergency room.
- Follow the Serious Incident Reporting process in the HSP, and complete incident report using the Intelex system on the Virtual Office.
- Notify and submit reports to client as required in contract.

18.5 Evacuation

- Evacuation routes, assembly areas, and severe weather shelters (and alternative routes and assembly areas) are to be specified on the site map.
- Evacuation route(s) and assembly area(s) will be designated by the ERC or designee before work begins.
- Personnel will assemble at the assembly area(s) upon hearing the emergency signal for evacuation.
- The ERC and a "buddy" will remain on the site after the site has been evacuated (if safe) to assist local responders and advise them of the nature and location of the incident.
- The ERC will account for all personnel in the onsite assembly area.
- A designated person will account for personnel at alternate assembly area(s).
- The ERC will follow the incident reporting procedures in the "Incident Notification, Reporting and Investigation" section of this HSP.

18.6 Evacuation Signals

Signal	Meaning
Grasping throat with hand	Emergency-help me.
Thumbs up	OK; understood.
Grasping buddy's wrist	Leave area now.
Continuous sounding of horn	Emergency; leave site now.

18.7 Inclement Weather

The boat captain is required to assess the weather forecast for each day and monitor the weather conditions throughout the day while on the water. If the weather forecast is not favorable or conditions on the water become hazardous due to weather, the boat captain has the final authority to cease all boating operations and return to port until conditions improve and all personnel feel safe.

Sudden inclement weather can rapidly encroach upon field personnel. Preparedness and caution are the best defenses. Field crew members performing work outdoors should carry clothing appropriate for inclement weather. Personnel are to take heed of the weather forecast for the day and pay attention for signs of changing weather that indicate an impending storm. Signs include towering thunderheads, darkening skies, or a sudden increase in wind. If stormy weather ensues, field personnel should discontinue work and seek shelter until the storm has passed.

Protective measures during a lightning storm include seeking shelter; avoiding projecting above the surrounding landscape (do not stand on a hilltop, seek low areas); staying away from open water, metal equipment, railroad tracks, wire fences, and metal pipes; and positioning people several yards apart. Some other general precautions include:

- Know where to go and how long it will take to get there. If possible, take refuge in a large building or vehicle. Do not go into a shed in an open area.
- The inclination to see trees as enormous umbrellas is the most frequent and most deadly mistake. Do not go under a large tree that is standing alone. Likewise, avoid poles, antennae and towers.
- If the area is wide open, go to a valley or ravine, but be aware of flash flooding.
- If you are caught in a level open area during an electrical storm and you feel your hair stand on end, drop to your knees, bend forward, and put your hands on your knees or crouch. The idea is to make yourself less vulnerable by being as low to the ground as possible and taking up as little ground space as possible. Lying down is dangerous, since the wet earth can conduct electricity. Do not touch the ground with your hands.
- Do not use telephones during electrical storms, except in the case of emergency.

Remember that lightning may strike several miles from the parent cloud, so work should be stopped/restarted accordingly. The lightning safety recommendation is 30-30: Seek refuge when thunder sounds within 30 seconds after a lightning flash; and do not resume activity until 30 minutes after the last thunder clap.

High winds can cause unsafe conditions, and activities should be halted until wind dies down. High winds can also knock over trees, so walking through forested areas during high-wind situations should be avoided. If winds increase, seek shelter or evacuate the area. Proper body protection should be worn in case the winds hit suddenly, because body temperature can decrease rapidly.

19. Spill Containment Procedures

Jacobs and subcontractor personnel working at the project site shall be knowledgeable of the potential HSE concerns associated with petroleum and other hazardous substances that could potentially be released at the project site.

The following is a list of criteria that must be addressed in Jacobs' or the subcontractor's plans in the event of an oil/petroleum spill or release of any other hazardous substance. In the event of a large quantity spill notify emergency services. Personnel discovering a spill shall (only if safe to do so):

- Stop the spill immediately (if possible) or note source. If unsafe conditions exist, then leave the area, call emergency services, inform nearby personnel, notify the site supervisors, and initiate incident reporting process. The SL shall be notified immediately.
- Extinguish sources of ignition (e.g., flames, sparks, hot surfaces, cigarettes, etc.)
- Clear personnel from the spill location and barricade the area.
- Utilize available spill control equipment in an effort to ensure that fires, explosions, and releases do not occur, recur, or spread.
- Use sorbent materials to control the spill at the source.
- Construct a temporary containment dike of sorbent materials, cinder blocks, bricks, or other suitable materials to help contain the spill.
- Attempt to identify the character, exact source, amount, and extent of the released materials. Identification of the spilled material should be made as soon as possible so that the appropriate clean-up procedure can be identified.
- Assess possible hazards to human health or the environment as a result of the release, fire, or explosion.
- A Spill Report shall be completed, including a description of the event, root causes, and corrective actions.

20. Behavior Based Loss Prevention System (BBLPS)

A BBLPS is a system to prevent or reduce losses using behavior based tools and proven management techniques to focus on behaviors or acts that could lead to losses.

The four basic Loss Prevention tools that will be used by Jacobs projects to implement the BBLPS include:

- AHA
- PTSP
- Self-Assessment Checklists
- Beyond Zero Observation Reports (BZOs)

The SL or designated Jacobs representative onsite is responsible for implementing the BBLPS on the project site. The PM remains accountable for its implementation. The SL or designee shall only oversee the subcontractor's implementation of their AHAs and PTSPs processes on the project.

20.1 Activity Hazard Analysis

An AHA defines the activity being performed, the hazards posed, and control measures required to perform the work safely. Workers are briefed on the AHA before doing the work and their input is solicited prior, during and after the performance of work to further identify the hazards posed and control measures required.

An AHA will be prepared before beginning each project activity posing H&S hazards to project personnel using the AHA form provided in Attachment 6. The AHA shall identify the work tasks required to perform each activity, along with potential H&S hazards and recommended control measures for each work task. In addition, a listing of the equipment to be used to perform the activity, inspection requirements and training requirements for the safe operation of the equipment listed must be identified.

An AHA shall be prepared for all field activities performed by Jacobs and subcontractor activities during the course of the project. Hazard Controls (found in Sections 8 and its subsections of the HSP), the Hazard Analysis Table (Table D-1-1), and applicable Jacobs WIs should be used as a basis for preparing AHAs.

Jacobs subcontractors are required to provide AHAs specific to their scope of work on the project for acceptance by Jacobs. Each subcontractor shall submit AHAs for their field activities, as defined in their work plan/scope of work, along with their project-specific safety plan/accident prevention plan. Additions or changes in Jacobs or subcontractor field activities, equipment, tools, or material to perform work, or an additional/different hazard encountered that requires additional/different hazard control measures, will require either a new AHA to be prepared or an existing AHA to be revised.

20.2 Pre-Task Safety Plans

Daily safety meetings are held with all project personnel in attendance to review the hazards posed and required H&S procedures/AHAs, which apply for each day's project activities. The PTSPs serve the same purpose as these general assembly safety meetings, but the PTSPs are held between the crew supervisor and their work crews to focus on those hazards posed to individual work crews. At the start of each day's activities, the crew supervisor completes the PTSP, provided in Attachment 6, with input from the work crew, during their daily safety meeting. The day's tasks, personnel, tools and equipment that will be used to perform these tasks are listed, along with the hazards posed and required H&S procedures, as identified in the AHA. The use of PTSPs, better promotes worker participation in the hazard recognition and control

process, while reinforcing the task-specific hazard and required H&S procedures with the crew each day. The use of PTSPs is a common safety practice in the construction industry.

20.3 Project Activity Self-Assessment Checklists

In addition to the hazard controls specified in this document, Project Activity Self-Assessment Checklists are contained in Attachment 5. The Project Activity Self-Assessment Checklists are based upon minimum regulatory compliance and some site-specific requirements may be more stringent. The objective of the self-assessment process is to identify gaps in project safety performance, and prompt for corrective actions in addressing these gaps. The self-assessment checklists, including documented corrective actions, shall be made part of the permanent project records and maintained by the SL.

The self-assessment checklists will also be used by the SL in evaluating the subcontractors and any client contractors' compliance on site.

The self-assessment checklists for the following tasks and exposures are required when the task or exposure is initiated and weekly thereafter while the task or exposure is taking place. The checklists shall be completed by the SL or other Jacobs representative and maintained in project files:

- Drilling
- Hand and Power Tools

20.4 StepBack Process

(Reference BIAF Global Guide, BIAF-350-G-01, HSE StepBack Process)

The StepBack process applies to all Jacobs employees and subcontractors that are performing tasks in an office or at a site location. It is a critical thinking process to supplement HSE planning tools such as the PTSP, AHAs, and HSPs and should be applied at the start of shift, after a break, when the task or location change, when adjacent work may present additional hazards, or any other hazard or change to task is identified. Training for initial roll-out will be provided via 8-hour HAZWOPER refreshers or a briefing from the RHSM.

The process is comprised of three key steps:

Identify: Prior to and while executing the task, "StepBack" and identify any new hazards or changes to the environment, including reviewing personal physical and mental preparedness. Ask the questions on the card (see wallet card or use the form attached to this plan); if "yes" is the answer to all questions, the task may proceed. If you answer "no" to any of the questions, STOP work and contact your HSM/Environmental Manager (EM). Together you will work through the following steps to identify corrective actions.

Evaluate: Assess the risk associated with the new hazard or change to the environment to understand the level of risk.

Act: Take appropriate action. Engage with project management or supervisors as necessary to identify the risk mitigation measures. Mitigation measures (changes to means/methods, use of different PPE than specified in the AHA, or similar) would require RHSM involvement and potentially revision to the AHA and or HSP.

Completion: After the job has finished ask:

- Did you feel safe doing the job?
- Were others nearby working safely?
- Can any improvements be made next time?

If any of these questions yield a "no" response, follow up with feedback to the PM, RHSM, or your supervisor.

20.5 Beyond Zero Observation Reports

(Reference Jacobs Business Management System WI, JJ-HS-WI-0306-JJ, 20.5 Beyond Zero Observation)

BZOs are a tool:

- That can be used for both a planned or an unplanned observation of behaviors or condition in the work area.
- For improving leadership, workforce behaviors, and peer to peer communication.
- To provide positive reinforcement, and to correct unsafe behaviors or unsafe conditions.
- To document something you witness outside of work hours that others can learn from.

BZOs are a required element of our BeyondZero Culture of Caring. As a general rule, the BZO program will be used in place of the legacy Jacobs Safe Behavior or Work Observation programs, unless stated otherwise based on client program needs.

Performing a BZO is something that everyone should be considering both in the workplace and outside of the workplace as often as possible. The minimum frequency for submitting a BZO is once per week.

Everyone is asked to participate: all office staff, management, PMs, supervisors, SLs, and field staff whether in an office, traveling, at a project site, or anywhere in between.

After you have discussed the observation with the affected parties (see ***notes** below), use the BZO app on your phone or tablet, or use your computer to log onto JacobsConnect and enter the BZO into the system. Once submitted, parts of the BZO cannot be changed, so reach out to your HSM/EM if you need help entering an observation. Once submitted, the BZO will be routed to your supervisor (and PM if you entered the project number). A feature to this system is you can attach photos.

***Note**: Entering the correct Worst Potential Severity (WPS) code is important! The WPS code is a way of rating an event based on the likelihood of what could have happened versus what actually happened. When a WPS of 3, 4, or 5 is indicated, the BZO form is elevated to higher levels of management so please be sure you have notified your HSM, Supervisor, and/or PM of the event prior to submitting a BZO with a WPS of 3 or greater. Likewise, if any follow-up action is needed, regardless of WPS, notify the HSM and/or PM and supervisor.

***Note: For any incident with a WPS greater than 3, or when further action is necessary, notify your HSM/EM and PM/Supervisor as soon as possible.**

Worst Potential Severity Table

WPS	Injury -Illness	Environment	Property Damage
5	Fatality or total permanent disability	Serious offsite impact, significant remediation required	USD$> 3 million
4	Partial disability; life changing; intensive care	Significant offsite impact, some remediation required	USD$ 300K-3 million
3	Urgent treatment, surgery	Release significantly above reportable limit of some local impact	USD$ 30K-300K
2	Medical treatment to prevent deterioration	Release above reportable limit or minor impact	USD$ 3K-30k
1	Simple, immediate treatment	Small release contained onsite and no impact	USD$ less than 3K

How do I complete a BZO?

Go to JacobsConnect and use the electronic BZO tool;

Download the BZO app from the Jacobs Appstore: https://appstore.jacobs.com; or

Use the hard copy form attached to this plan.

BZOs can be chosen by the Center of Excellence as a winner of the "BZO of the Month."

StepBack Risk Questions	YES	NO
Is there enough time to complete the task safely and are people focused (not fatigued, distracted)?		
Is the right person(s) completing the task? (experience/training)		
Are adequate support and resources available?		
Are the correct tools, equipment, and PPE in place and are they in good operating condition?		
Are control measures in place to protect people/the environment (barriers, LOTO, adjacent activities)?		
Have environmental issues (waste, hazardous materials, stormwater) been identified and mitigated?		
Are there safe access and egress to and from the work area?		
Are emergency planning/response measures adequate, including spills/releases?		
Are conditions the same since the task was last assessed?		
Is there a pre-task plan developed and approved for the task?		
If you answer '**NO**' to any of the above, **STOP** work, and contact your HSM or EM		

SAFETY OBSERVATION REPORT					
Project Name:			**Project Manager:**		
Project #:			**Health & Safety Mgr.:**		
Office Observation Information					
Office:					
Observation Information					
Observer Name:		**Company:**		**Date & Time:**	
Position/Title of worker observed:		**Company:**			
Observation Type:	☐ Safe Behavior ☐ Safe Condition ☐ Unsafe Behavior ☐ Unsafe Condition ☐ Opportunity for Improvement ☐ Other (specify):				
Work or Task Observed:					
Describe Observation:					
Type of incident prevented?					
WPS (*see table below):	☐ 1 ☐ 2 ☐ 3 ☐ 4 ☐ 5				
Remedial Action Taken?	☐ Not Applicable ☐ No ☐ Yes (describe):				
Further Action Needed?	☐ No Action ☐ Outstanding Action ☐ Urgent Action (describe action needed):				

21. Incident Notification, Reporting, and Investigation

21.1 General Information

This section applies to the following:

- All injuries involving employees, third parties, or members of the public
- Damage to property or equipment
- Interruptions to work or public service (e.g., hitting a utility)
- Incidents which attract negative media coverage
- Near misses
- Spills, leaks, or regulatory violations
- Motor vehicle accidents

Documentation, including incident reports, investigation, analysis and corrective measure taken, shall be kept by the SL and maintained onsite for the duration of the project.

21.2 Section Definitions

Incident: an undesired event which results or could have resulted in loss through injury, damage to assets, or environmental harm. This includes all of the definitions below.

Accident: an incident involving actual loss through injury, damage to assets, or environmental harm.

Near Miss: an unsafe act or incident which, in other circumstances, could have resulted in loss through injury, damage to assets, or environmental harm.

Serious Incident:

- All fatalities including contractors, subcontractors, third parties, or members of the public
- Kidnap/Missing Person
- Event that involves a fire, explosion, or property damage that requires a site evacuation or is estimated to result in greater than $500,000 in damage
- Acts or threats of terrorism
- Spill or release of hazardous materials or substances that involves a significant threat of imminent harm to site workers, neighboring facilities, the community, or the environment

21.3 Reporting Requirements

All employees and subcontractors' employees shall immediately report any incident (including "near misses," as defined in Section 21.2) in which they are involved or witness to their supervisor.

The Jacobs or subcontractor supervisor, upon receiving an incident report, shall inform his immediate superior and the Jacobs SL.

The SL shall immediately report the following information to the RHSM and PM by phone and e-mail:

- Project Name/Site Manager
- Date and time of incident

- Description of incident
- Extent of know injuries/damage
- Level of medical attention
- Preliminary root cause/corrective actions

The SL shall complete an entry into the Hours and Incident Tracking System (Intelex) database system located on Jacobs' Virtual Office (or if not available, use the hard copy Incident Report Form and Root Cause Analysis Form and forward it to the RHSM) within 24 hours and finalize those forms within 3 calendar days.

The Jacobs team shall comply with all applicable statutory incident reporting requirements, such as those by OSHA and the police.

21.4 Intelex System

It is the policy of Jacobs to maintain an Intelex entry for all work-related injuries and illnesses sustained by its employees in accordance with recordkeeping and insurance requirements. An Intelex entry will be maintained for other incidents (property damage, fire or explosion, spill, release, potential violation, and near misses) as part of our loss prevention and risk reduction initiative.

21.5 Injury Management/Return-to-Work (for Jacobs staff Only)

21.5.1 Background

The Injury Management Program has been established to provide orderly, effective and timely medical treatment and return-to-work transition for an employee who sustains a work-related injury or illness. It also provides guidance and assistance with obtaining appropriate treatment to aid recovery, keep supervisors informed of employee status, and to quickly report and investigate work-related injury/illnesses to prevent recurrence.

21.5.2 The Injury Management/Return-to-Work Notification Process:

- Employee informs their Supervisor.
- Employee calls the Injury Management Program toll free number 1-888-449-7787 immediately and speaks with the Occupational Injury Nurse. This number is operable 24 hours per day, 7 days a week.
- Supervisor ensures employee immediately calls the Injury Management Program number. Supervisor makes the call with the injured worker or for the injured worker if needed.
- Nurse assists employee with obtaining appropriate medical treatment, and as necessary schedules clinic visit for employee (calls ahead and assists with any necessary follow-up treatment) with the supervisor or SL accompanying the employee if a clinic visit is necessary to ensure that employees receive appropriate and timely care.
- Supervisor/SL completes the Intelex entry or Incident Report Form immediately (within 24 hours) and forwards it to the PM and RHSM.
- Nurse notifies appropriate Jacobs staff by e-mail (e.g., supervisor, H&S, HR, Workers' Compensation).
- Nurse communicates and coordinates with and for employee on treatment through recovery.
- Supervisor ensures suitable duties are identified and available for injured/ill workers who are determined to be medically fit to return-to-work on transitional duty (temporary and progressive).

- Supervisor ensures medical limitations prescribed (if any) by physician are followed until the worker is released to full duty.

21.6 Serious Incident Reporting Requirements

The Serious Incident Reporting Requirements ensures timely notification and allows for positive control over flow of information so that the incident is handled effectively, efficiently, and in conjunction with appropriate corporate entities. This standard notification process integrates Health, Safety, Security, Environment and Firm Wide Security Operations (FWSO) requirements for the consistent reporting of and managing of serious events throughout our operations.

21.6.1 Serious Incident Determination

The following are general criteria for determining whether an incident on Jacobs owned or managed facilities or program sites is considered serious and must be immediately reported up to Group President level through the reporting/notification process:

- Work-related death, or life-threatening injury or illness of a Jacobs employee, subcontractor, or member of the public
- Kidnap/missing person
- Acts or threats of terrorism
- Event that involves a fire, explosion, or property damage that requires a site evacuation or is estimated to result in greater than $500,000 in damage
- Spill or release of hazardous materials or substances that involves a significant threat of imminent harm to site workers, neighboring facilities, the community or the environment

21.6.2 Serious Incident Reporting

If an incident meets the "Serious Incident" criteria, the PM is to immediately contact the 24-hour Serious Incident Reporting – Global Assistance and Response HOTLINE # +1-443-221-6281 and then follow the standard incident reporting procedure.

For all serious incidents, this standard reporting process is implemented immediately so as to ultimately achieve notification to the BG President within 2 hours of incident onset or discovery, and notification to appropriate corporate Crisis Management Support Team.

21.7 Incident Root Cause Analysis

The accident analysis is essential if all causes of the incident are to be identified for the correct remedial actions to be taken to prevent the same and similar type of incident from recurring. The investigation team will consist of the SL (with support from RHSM), appropriate subcontractor personnel as necessary, the PM, and the responsible supervisor. More participants may be involved as needed to complete the investigation.

The Root Cause Analysis Form must be completed for all Loss Incidents and Near Loss Incidents. This form must be submitted to the investigation team for review.

For minor losses or near losses, the information may be gathered by the supervisor or other personnel immediately following the loss. Based on the complexity of the situation, this information may be all that

is necessary to enable the investigation team to analyze the loss, determine the root cause, and develop recommendations. More complex situations may require the investigation team to revisit the loss site or re-interview key witnesses to obtain answers to questions that may arise during the investigation process.

Photographs or videotapes of the scene and damaged equipment should be taken from all sides and from various distances. This point is especially important when the investigation team will not be able to review the loss scene.

The investigation team must use the Root Cause Analysis Flow Chart to assist in identifying the root cause(s) of a loss. Any loss may have one or more root causes and contributing factors. The root cause is the primary or immediate cause of the incident, while a contributing factor is a condition or event that contributes to the incident happening but is not the primary cause of the incident. Root causes and contributing factors that relate to the person involved in the loss, his or her peers, or the supervisor should be referred to as "personal factors." Causes that pertain to the system within which the loss or injury occurred should be referred to as "job factors."

21.7.1 Personal Factors

- Lack of skill or knowledge
- Correct way takes more time and/or requires more effort
- Short-cutting standard procedures is positively reinforced or tolerated
- Person thinks there is no personal benefit to always doing the job according to standards

21.7.2 Job Factors

- Lack of or inadequate operational procedures or work standards
- Inadequate communication of expectations regarding procedures or standards
- Inadequate tools or equipment

The root cause(s) could be any one or a combination of these seven possibilities or some other uncontrollable factor. In the vast majority of losses, the root cause is very much related to one or more of these seven factors. Uncontrollable factors should be used rarely and only after a thorough review eliminates all seven other factors.

21.7.3 Corrective Actions

Include all corrective actions taken or those that should be taken to prevent recurrence of the incident. Include the specific actions to be taken, the employer and personnel responsible for implementing the actions, and a timeframe for completion. Be sure the corrective actions address the causes.

Once the investigation report has been completed, the PM shall hold a review meeting to discuss the incident and provide recommendations. The responsible supervisors shall be assigned to carry out the recommendations and shall inform the SL upon successful implementation of all recommended actions.

- The RHSM will inform the Responsible EM of any environmental incidents.
- Evaluation and follow-up of the Incident Report Form will be completed by the RHSM, Responsible EM, or FWSO. The BG HSE Lead will review all BG incidents and modify as required.
- Incident Investigations must be initiated and completed as soon as possible but no later than 72 hours after the incident.

22. Records and Reports

An organized project filing system is essential for good documentation and recordkeeping. There are many benefits to an organized filing system:

- Other Jacobs employees can easily and quickly find documents
- Records are readily available for review
- Records may be needed during OSHA investigations, audits, or other legal matters
- Records may be needed on short notice in case of an accident, illness, or other emergency
- Systematic recordkeeping aids in overall project organization

The project filing system shall be established at the beginning of the project and maintained throughout all phases of construction. The information contained in the filing system shall be updated regularly and/or as specified in this document. The PM and SL are responsible for collecting documentation and maintaining a complete and organized filing system.

Below are examples of records that must be maintained as the project progresses:

- Exposure records includes air monitoring data (including calibration records), SDSs, and exposure modeling results
- Physical hazard exposure records include noise, ionizing radiation, non-ionizing radiation, vibration, and lasers exposure assessments and measurements
- Respiratory Fit Test Records
- Training Records
- Injury/illness reports and investigations
- Federal or State Agency Inspection Records
- Other Records
  - Ergonomic evaluations
  - HSE audits and assessments
  - Project-Specific HSE Plans
  - Confined Space Entry Permits
  - Equipment inspections
  - Equipment maintenance

Attachment 1
Health and Safety Plan Employee Sign-off Form

EMPLOYEE SIGN-OFF FORM

Health and Safety Plan

The JACOBS project employees and subcontractors listed below have been provided with a copy of this HSP, have read and understood it, and agree to abide by its provisions.

Project Name: **Project Number:**

EMPLOYEE NAME (Please print)	**EMPLOYEE SIGNATURE**	**COMPANY**	**DATE**

Attachment 2
Chemical Inventory/Register Form

CHEMICAL INVENTORY/REGISTER FORM

Refer to HSM for instructions on completing this form.

Location:

HCC:

☐ Office ☐ Warehouse ☐ Laboratory ☐ Project:

Project No.:

Regulated Product	Location	Container labeled (✓if yes)	SDS available (✓if yes)

SDS for the listed products will be maintained at:

Attachment 3
Chemical-Specific Training Form

CHEMICAL-SPECIFIC TRAINING FORM

Refer to HSM for instructions on completing this form.

Location:	Project # :
HCC:	Trainer:

TRAINING PARTICIPANTS:

NAME	SIGNATURE	NAME	SIGNATURE

REGULATED PRODUCTS/TASKS COVERED BY THIS TRAINING:

The HCC shall use the product SDS to provide the following information concerning each of the products listed above.

☐ Physical and health hazards

☐ Control measures that can be used to provide protection (including appropriate work practices, emergency procedures, and PPE to be used)

☐ Methods and observations used to detect the presence or release of the regulated product in the workplace (including periodic monitoring, continuous monitoring devices, visual appearance or odor of regulated product when being released, etc.)

Training participants shall have the opportunity to ask questions concerning these products and, upon completion of this training, will understand the product hazards and appropriate control measures available for their protection.

Copies of SDSs, chemical inventories, and Jacobs' written HAZCOM program shall be made available for employee review in the facility/project HAZCOM file.

Attachment 4
Vehicle Accident Guidance

VEHICLE ACCIDENT INSTRUCTIONS (U.S. & Canada)

Jacobs Owned or Leased Vehicle Damage only:

You are not required to submit the Vehicle Incident Report to Risk Management for incidents involving ONLY damage to Jacobs personnel or property. (Example: owned/leased/rental vehicles where single vehicle runs off road). In these instances:

- Notify your supervisor, HSE representative, and Fleet Management (JacobsUSAFleet@Jacobs.com)
- If our employee is injured, follow procedures for reporting a workers' compensation claim
- If you have questions, contact your Risk Management representative

Vehicle Damage or Injury to Members of the Public:

- Complete and submit the attached Vehicle Intake Form.
- If necessary, move vehicles to a safe location and wait for police.
- Aid the Injured - Do not move injured individuals unless absolutely necessary. Warn other drivers.
- Call the Police – Give exact location and advise if medical help is needed.
- Do not Comment – Do not make/sign any statement concerning who was at fault. Give out only information required by authorities.
- Notification – Report the accident to your Department Manager, your HSE Representative, and Global Risk Management.
- Serious accidents and accidents with injuries: Report as soon as possible, immediately following the accident. Do not wait for a copy of the police report to notify Global Risk Management of the incident.
- Accidents without injuries: Report within 24 hours of the accident.
- Vehicle Accident Report – The Jacobs Vehicle Accident Report must be completed and sent to your HSE Representative and AutoClaims@Jacobs.com

Rental Car Incidents:

- For incidents involving a rental car that was rented through BCD Travel or Concur Travel for approved work business, you must file an auto claim directly with the rental car company.
- For incidents involving Rental Vehicles with injuries to Members of the Public, complete and submit the attached Intake Form to Global Risk Management.

Questions

Contact: Zane Wilson (Zane.Wilson@Jacobs.com), Global Risk Management Department Phone: 214 583-8417

E-mail: AutoClaims@Jacobs.com

E-mail Completed form to: AutoClaims@Jacobs.com For Questions Contact: 214 583-8417 Location Code: _____

Company/Subsidiary Name: _____ Line of Business:

People & Places Solutions ☐ Critical Mission Solutions ☐ Corporate Functions☐

Incident Location

Date of Accident: _____ Time of Accident: ☐ a.m. ☐ p.m. Location of Accident: Client Facility/Project Site ☐ Highway ☐ Other (specify)☐_____

Address: _____

Nearest Intersection: City_____ State _____

Company Vehicle Information

Company Vehicle Driver: _____ Driver Date of Birth: _____

Office/Project Assigned to: Name of Passenger(s):

Work Address: _____

Home Address: _____ Home/Cell Phone: _____

Supervisor Name: _____ Supervisor Phone: _____

Vehicle Owner: _____Company Owned ☐ Leased ☐ Rental ☐Personal ☐ If rental vehicle, name of agency:_____

If rental, reservation through: BCD Travel ☐ Concur ☐ Rental Desk ☐

If leased vehicle, name of agency:_____

Vehicle Number: _____Make & Model: _____

Vehicle License Number: _____Vehicle ID Number _____

Has Driver Completed Jacobs Recognized Driver Training: Yes ☐ No ☐ Unknown ☐

Other Driver(s) Information

Other Driver(s) Name: _____

Home Address: _____

Phone Number: Home/Cell:_____Work: _____

Vehicle Owner: _____ Relation to Driver: _____

Vehicle Make & Model _____

Insurance Company _____

Insurance Agent: _____Agent Phone Number:_____

Policy Number: _____

Incident Description

Contact with: Other Vehicle(s) ☐ Fixed Object ☐ Pedestrian ☐ Other ☐

Details: _____

_____

_____

_____

_____

Witnesses: _____

Citations Issued: Jacobs Driver: Yes ☐ No ☐ Other Driver: Yes ☐ No ☐

Police Contacted: Yes ☐ No ☐ Agency: _____

Officer name/badge: _____

Injuries

Jacobs Employee Yes ☐ No ☐ Other Driver/Passengers: Yes ☐ No ☐

If yes, describe _____

Other Driver/Passengers: Yes ☐ No ☐

If yes, describe _____

Vehicle Damages

Company Vehicle: _____ Location of Company Vehicle _____

Other Vehicle Damage: _____ Location of Other Vehicle: _____

Property Damage (Do not include vehicle damages listed above)

Property Owner: _____

Describe Damages: _____

Contact info for Owner: _____

Comments: _____

_____

_____

_____

_____

Diagram of Incident (attach additional documents if needed):

Report Submitted by: _____ Reporter's E-mail Address: _____

Attachment 5
Project Activity
Self-Assessment Checklists

HSE Self-Assessment Checklist - DRILLING

This checklist shall be used by Jacobs personnel **only** and shall be completed at the frequency specified in the project's HSP/FSI.

This checklist is to be used at locations where: 1) Jacobs employees are potentially exposed to hazards associated with drilling operations (complete Sections 1 and 3), and/or 2) Jacobs oversight of a drilling subcontractor is required (complete entire checklist).

SL may consult with drilling subcontractors when completing this checklist but shall not direct the means and methods of drilling operations nor direct the details of corrective actions. Drilling subcontractors shall determine how to correct deficiencies and we must carefully rely on their expertise. Items considered to be imminently dangerous (possibility of serious injury or death) shall be corrected immediately or all exposed personnel shall be removed from the hazard until corrected.

Completed checklists shall be sent to the HSM for review.

Project Name: _____Project No.: _____

Location: _____ PM: _____

Auditor: _____ Title: _____ Date: _____

This specific checklist has been completed to:

☐ Evaluate Jacobs employee exposures to drilling hazards
☐ Evaluate a Jacobs subcontractor's compliance with drilling H&S requirements
Subcontractors Name: _____

- Check "Yes" if an assessment item is complete/correct.
- Check "No" if an item is incomplete/deficient. Deficiencies shall be brought to the immediate attention of the drilling subcontractor. Section 3 must be completed for all items checked "No."
- Check "N/A" if an item is not applicable.
- Check "N/O" if an item is applicable but was not observed during the assessment.

SECTION 1	Yes	No	N/A	N/O
PERSONNEL SAFE WORK PRACTICES				
1. Only authorized personnel operating drill rig	☐	☐	☐	☐
2. Personnel cleared during rig startup	☐	☐	☐	☐
3. Personnel clear of rotating parts	☐	☐	☐	☐
4. Personnel not positioned under hoisted loads	☐	☐	☐	☐
5. Loose clothing and jewelry removed	☐	☐	☐	☐
6. Personnel instructed not to approach equipment that has become electrically energized	☐	☐	☐	☐
7. Smoking is prohibited around drilling operation	☐	☐	☐	☐
8. Personnel wearing appropriate PPE, per HSP/FSI	☐	☐	☐	☐

HSE Self-Assessment Checklist - DRILLING

SECTION 2	Yes	No	N/A	N/O
GENERAL				
9. Daily safety briefing/meeting conducted with crew	☐	☐	☐	☐
10. Daily inspection of drill rig and equipment conducted before use	☐	☐	☐	☐
DRILL RIG PLACEMENT				
11. Location of underground utilities identified	☐	☐	☐	☐
12. Safe clearance distance maintained from overhead powerlines	☐	☐	☐	☐
13. Drilling pad established, when necessary	☐	☐	☐	☐
14. Drill rig leveled and stabilized	☐	☐	☐	☐
DRILL RIG TRAVEL				
15. Rig shut down and mast lowered and secured prior to rig movement	☐	☐	☐	☐
16. Tools and equipment secured prior to rig movement	☐	☐	☐	☐
17. Only personnel seated in cab are riding on rig during movement	☐	☐	☐	☐
18. Safe clearance distance maintained while traveling under overhead powerlines	☐	☐	☐	☐
19. Backup alarm or spotter used when backing rig	☐	☐	☐	☐
DRILL RIG OPERATION				
20. Kill switch clearly identified and operational	☐	☐	☐	☐
21. All machine guards are in place	☐	☐	☐	☐
22. Rig ropes not wrapped around body parts	☐	☐	☐	☐
23. Pressurized lines and hoses secured from whipping hazards	☐	☐	☐	☐
24. Drill operation stopped during inclement weather	☐	☐	☐	☐
25. Air monitoring conducted per HSP/FSI for hazardous atmospheres	☐	☐	☐	☐
26. Rig placed in neutral when operator not at controls	☐	☐	☐	☐
DRILL RIG MAINTENANCE				
27. Defective components repaired immediately	☐	☐	☐	☐
28. Lockout/tagout procedures used prior to maintenance	☐	☐	☐	☐
29. Cathead in clean, sound condition	☐	☐	☐	☐
30. Drill rig ropes in clean, sound condition	☐	☐	☐	☐
31. Fall protection used for fall exposures of 6 feet or greater	☐	☐	☐	☐
32. Rig in neutral and augers stopped rotating before cleaning	☐	☐	☐	☐
33. Good housekeeping maintained on and around rig	☐	☐	☐	☐
DRILLING AT HAZARDOUS WASTE SITES				
34. Waste disposed of according to HSP	☐	☐	☐	☐
35. Appropriate decontamination procedures being followed, per HSP	☐	☐	☐	☐
DRILLING ONBOARD A BOAT				
36. Has marine vessel checklist D-8-1 in Section 8.22 been completed	☐	☐	☐	☐

HSE Self-Assessment Checklist - DRILLING

SECTION 3

Complete this section for all items checked "No" in Sections 1 or 2. Deficient items must be corrected in a timely manner.

Item #	Corrective Action Planned/Taken	Date Corrected

HSE Self-Assessment Checklist—HAND AND POWER TOOLS

This checklist shall be used by Jacobs personnel **only** and shall be completed at the frequency specified in the project's HSP/FSI. This checklist is to be used at locations where: (1) Jacobs employees are exposed to hand and power tool hazards and/or (2) Jacobs provides oversight of subcontractor personnel who are exposed to hand and power tool hazards.

SL may consult with subcontractors when completing this checklist but shall not direct the means and methods of hand and power tool use nor direct the details of corrective actions. Subcontractors shall determine how to correct deficiencies and we must carefully rely on their expertise. Items considered to be imminently dangerous (possibility of serious injury or death) shall be corrected immediately or all exposed personnel shall be removed from the hazard until corrected.

Completed checklists shall be sent to the HSE Staff for review.

Project Name: _____ Project No.: _____

Location: _____ PM: _____

Auditor: _____ Title: _____ Date: _____

This specific checklist has been completed to:

☐ Evaluate Jacobs employee exposure to hand and power tool hazards.

☐ Evaluate a Jacobs subcontractor's compliance with hand and power tool requirements.

Subcontractors Name: _____

- Check "Yes" if an assessment item is complete/correct.
- Check "No" if an item is incomplete/deficient. Deficiencies shall be brought to the immediate attention of the subcontractor. Section 3 must be completed for all items checked "No."
- Check "N/A" if an item is not applicable.
- Check "N/O" if an item is applicable but was not observed during the assessment.

SECTION 1

	Yes	No	N/A	N/O
SAFE WORK PRACTICES				
1. All tools operated according to manufacturer's instructions and design limitations.	☐	☐	☐	☐
2. All hand and power tools maintained in a safe condition and inspected/tested before use.	☐	☐	☐	☐
3. Defective tools are tagged and removed from service until repaired.	☐	☐	☐	☐
4. PPE is selected and used according to tool-specific hazards anticipated.	☐	☐	☐	☐
5. Power tools are not carried or lowered by their cord or hose.	☐	☐	☐	☐
6. Tools are disconnected from energy sources when not in use, servicing, cleaning, etc.	☐	☐	☐	☐
7. Safety guards remain installed or are promptly replaced after repair.	☐	☐	☐	☐
8. Tools are stored properly.	☐	☐	☐	☐
9. Cordless tools and recharging units conform to electrical standards and specifications.	☐	☐	☐	☐
10. Tools used in explosive environments are rated for such use.	☐	☐	☐	☐
11. Knife or blade hand tools are used with the proper precautions.	☐	☐	☐	☐
12. Consider controls to avoid muscular skeletal, repetitive motion, and CTS.	☐	☐	☐	☐

HSE Self-Assessment Checklist—HAND AND POWER TOOLS

SECTION 2	Yes	No	N/A	N/O
GENERAL				
13. PPE is selected and used according to tool-specific hazards anticipated.	☐	☐	☐	☐
14. Tools are tested daily to assure safety devices are operating properly.	☐	☐	☐	☐
15. Damaged tools are removed from service until repaired.	☐	☐	☐	☐
16. Power operated tools designed to accommodate guards have guards installed.	☐	☐	☐	☐
17. Rotating or moving parts on tools are properly guarded.	☐	☐	☐	☐
18. Machines designed for fixed locations are secured or anchored.	☐	☐	☐	☐
19. Floor and bench-mounted grinders are provided with properly positioned work rests.	☐	☐	☐	☐
20. Guards are provided at point of operation, nip points, rotating parts, etc.	☐	☐	☐	☐
21. Fluid used in hydraulic-powered tools is approved fire-resistant fluid.	☐	☐	☐	☐
ELECTRIC-POWERED TOOLS				
22. Electric tools are approved double insulated or grounded and used accordingly.	☐	☐	☐	☐
23. Electric cords are not used for hoisting or lowering tools.	☐	☐	☐	☐
24. Electric tools used in damp/ wet locations approved for such locations or GFCI installed.	☐	☐	☐	☐
25. Hand-held tools are equipped with appropriate on/off controls appropriate for the tool.	☐	☐	☐	☐
26. Portable, power-driven circular saws are equipped with proper guards.	☐	☐	☐	☐
ABRASIVE WHEEL TOOLS				
27. All employees using abrasive wheel tools are wearing eye protection.	☐	☐	☐	☐
28. All grinding machines are supplied with sufficient power to maintain spindle speed.	☐	☐	☐	☐
29. Abrasive wheels are closely inspected and ring-tested before use.	☐	☐	☐	☐
30. Grinding wheels are properly installed.	☐	☐	☐	☐
31. Cup-type wheels for external grinding are protected by the proper guard or flanges.	☐	☐	☐	☐
32. Portable abrasive wheels used for internal grinding are protected by safety flanges.	☐	☐	☐	☐
33. Safety flanges are used only with wheels designed to fit the flanges.	☐	☐	☐	☐
34. Safety guards on abrasive wheel tools are mounted properly and of sufficient strength.	☐	☐	☐	☐
PNEUMATIC-POWERED TOOLS				
35. Tools are secured to hoses or whip by positive means to prevent disconnection.	☐	☐	☐	☐
36. Safety clips or retainers are installed to prevent attachments being expelled.	☐	☐	☐	☐
37. Safety devices are installed on automatic fastener feed tools as required.	☐	☐	☐	☐
38. Compressed air not used for cleaning unless reduced to < 30 psi, with PPE, and guarded.	☐	☐	☐	☐
39. Manufacturer's safe operating pressure for hoses, pipes, valves, etc. are not exceeded.	☐	☐	☐	☐
40. Hoses are not used for hoisting or lowering tools.	☐	☐	☐	☐
41. All hoses >1/2" dia. have safety device at source to reduce pressure upon hose failure.	☐	☐	☐	☐
42. Airless spray guns have required safety devices installed.	☐	☐	☐	☐
43. Blast cleaning nozzles are equipped with operating valves, which are held open manually.	☐	☐	☐	☐
44. Supports are provided for mounting nozzles when not in use.	☐	☐	☐	☐
45. Air receiver drains, handholes, and manholes are easily accessible.	☐	☐	☐	☐
46. Air receivers equipped with drainpipes/valves for removal of accumulated oil and water.	☐	☐	☐	☐
47. Air receivers are completely drained at required intervals.	☐	☐	☐	☐
48. Air receivers are equipped with indicating pressure gauges.	☐	☐	☐	☐
49. Safety, indicating, and controlling devices are installed as required.	☐	☐	☐	☐
50. Safety valves tested frequently and at regular intervals to assure good operating condition.	☐	☐	☐	☐

HSE Self-Assessment Checklist—HAND AND POWER TOOLS

SECTION 2 (continued)

	Yes	No	N/A	N/O
LIQUID FUEL-POWERED TOOLS				
51. Liquid fuel-powered tools are stopped when refueling, servicing, or maintaining.	☐	☐	☐	☐
52. Liquid fuels are stored, handled, and transported in accordance with regulations	☐	☐	☐	☐
53. Liquid fuel-powered tools are used in confined spaces in accordance with regulations	☐	☐	☐	☐
54. Safe operating pressures of hoses, valves, pipes, filters, and other fittings not exceeded.	☐	☐	☐	☐
POWDER-ACTUATED TOOLS				
55. Only trained employee operates powder-actuated tools.	☐	☐	☐	☐
56. Powder-actuated tools are not loaded until just prior to intended firing time.	☐	☐	☐	☐
57. Tools are not pointed at any employee at any time.	☐	☐	☐	☐
58. Hands are kept clear of open barrel end.	☐	☐	☐	☐
59. Loaded tools are not left unattended.	☐	☐	☐	☐
60. Fasteners are not driven into very hard or brittle materials.	☐	☐	☐	☐
61. Fasteners not driven into easily penetrated materials unless suitable backing is provided.	☐	☐	☐	☐
62. Fasteners are not driven into spalled areas.	☐	☐	☐	☐
63. Powder-actuated tools are not used in an explosive or flammable atmosphere.	☐	☐	☐	☐
64. All tools used with correct shields, guards, attachments recommended by manufacturer.	☐	☐	☐	☐
JACKING TOOLS				
65. Rated capacities are legibly marked on jacks and not exceeded.	☐	☐	☐	☐
66. Jacks have a positive stop to prevent over-travel.	☐	☐	☐	☐
67. The base of jacks are blocked or cribbed to provide a firm foundation, when required.	☐	☐	☐	☐
68. Wood blocks are place between the cap and load to prevent slippage, when required.	☐	☐	☐	☐
69. After load is raised, it is cribbed, blocked, or otherwise secured immediately.	☐	☐	☐	☐
70. Antifreeze is used when hydraulic jacks are exposed to freezing temperatures.	☐	☐	☐	☐
71. All jacks are properly lubricated.	☐	☐	☐	☐
72. Jacks are inspected as required.	☐	☐	☐	☐
73. Repair or replacement parts are examined for possible defects.	☐	☐	☐	☐
74. Jacks not working properly are removed from service and repaired or replaced.	☐	☐	☐	☐
HAND TOOLS				
75. Wrenches are not used when jaws are sprung to the point of slippage.	☐	☐	☐	☐
76. Impact tools are kept free of mushroomed heads.	☐	☐	☐	☐
77. Wooden handles of tools are kept free of splinters or cracks and are tightly fitted in tool.	☐	☐	☐	☐

HSE Self-Assessment Checklist—HAND AND POWER TOOLS

SECTION 3

Complete this section for all items checked "No" in Sections 1 or 2. Deficient items must be corrected in a timely manner.

Item #	Corrective Action Planned/Taken	Date Corrected

Attachment 6
Behavior Based Loss Prevention System Forms

Activity Hazard Analysis

Pre-Task Safety Plan

ACTIVITY HAZARD ANALYSIS, ENVIRONMENTAL SAMPLING (SOIL, SW, SEDIMENT)

Activity/Work Task: Environmental Sampling (Soil, SW, Sediment)						**Overall Risk Assessment Code (RAC) (Use highest code)**	**M**
Project Location: Portland Harbor	**Risk Assessment Code (RAC) Matrix**						
Contract Number:	**Severity**	**Probability**					
Date Prepared: November		**Frequent**	**Likely**	**Occasional**	**Seldom**	**Unlikely**	
Prepared by: John Culley	Catastrophic	E	E	H	H	M	
	Critical	E	H	H	M	L	
Reviewed by:	Marginal	H	M	M	L	L	
	Negligible	M	L	L	L	L	
Notes: (Field Notes, Review Comments, etc.)	Step 1: Review each Hazard with identified safety Controls and determine RAC (See above)						
	Probability is the likelihood to cause an incident, near miss, or accident and identified as: Frequent, Likely, Occasional, Seldom or Unlikely	**RAC Chart**					
	Severity is the outcome/degree if an incident, near miss, or accident did occur and identified as: Catastrophic, Critical, Marginal, or Negligible	E =	Extremely high risk				
		H =	High risk				
	Step 2: Identify the RAC (Probability/Severity) as E, H, M, or L for each Hazard on AHA. Annotate the overall highest RAC at the top of AHA.	M =	Moderate risk				
		L =	Low risk				

ACTIVITY HAZARD ANALYSIS, ENVIRONMENTAL SAMPLING (SOIL, SW, SEDIMENT)

Job Steps	Hazards	Controls	RAC
Sampling	Bodily injury from sampling tools	• If working in an MRS, contact the UXOSO for a UXO escort. • Leather gloves should be worn when handling sharp, rough, or slippery surfaces. • Keep hands and body parts clear of hand drill augers when rotating. • Keep back straight and use foot pedal for leverage when pushing soil coring probe or other tools into surface soil. • Take turns collecting increments to avoid ergonomic stress. • Technicians should perform a pre-operational inspection of their equipment. Do not use any equipment that is unsafe. • Use tools in the correct manner to avoid tool damage and personal injury. • Keep all bystanders, children, and pets out of the work area. • Allow ample space for each employee to work safely. • Allow the tool to do the work by using a grip light enough to maintain control. • Be alert to protruding nails or spikes, concrete barriers, or other buried materials. • Clear walkways work areas of equipment, tools, vegetation, excavated material, and debris. • Mark, identify, or barricade other obstructions. • The employee is responsible for complying with all applicable HSE training requirements relating to hand and power tool safety and for providing any additional training necessary to complete their tasks safely. • All hand and power tools should be maintained in a safe condition. • Tools are to be inspected and tested before use. If a tool is found to be defective, it is to be tagged “Do Not Use” and removed from service until repaired. • Power tools are not to be carried or lowered by the cord or hose. • Disconnect tools from energy sources when not in use, before servicing and cleaning, and when changing accessories such as blades, bits, and cutters. • Do not perform any electrical wiring maintenance unless qualified to do so. • Safety guards on tools are to remain installed while the tool is in use and promptly replaced after repair or maintenance has been performed. • Tools are to be stored properly, where they will not be damaged or come in contact with hazardous materials. • Only use a knife or blade if it is the best tool for the job, a separate AHA has been developed, and hand protection (e.g., Kevlar gloves) is provided. Cut away from the body. • Organize work, sequencing to prevent muscular skeletal, repetitive motion, and cumulative trauma stressors. Only employees who have been trained in the operation of the particular tool in use should be allowed to operate a powder-actuated tool. Training and certification must be provided to the safety coordinator before using the tool.	M

ACTIVITY HAZARD ANALYSIS, ENVIRONMENTAL SAMPLING (SOIL, SW, SEDIMENT)

Job Steps	Hazards	Controls	RAC
	Generator safety	• If a generator is used to power sampling equipment, inspect the generator for potential leaks. If there are any leaks, do not use generator. • If a generator is used to power sampling equipment, use a ground-fault circuit interrupter. • Always shut down and allow for cooling before refueling the generator. • Use lifting aids when moving the generator. • Use approved safety cans for fuel; keep out of direct sunlight and extreme temperatures.	L
	Contact with potentially contaminated soil	• Ensure level D PPE (see HSP) is worn: body PPE, eye protection, and nitrile gloves. • Avoid all contact with product and unprotected skin.	L
	Chemicals spilled or splashed into eyes or spilled on hands	• Handle samples slowly and deliberately. • Follow Jacobs WIs for transportation of samples. • Seal containers properly so they do not come open when transporting.	L
	Back injury	• Proper lifting techniques must be used when lifting any object. • Plan storage and staging to minimize lifting or carrying distances. • Use mechanical lifting aids whenever possible. • Have someone assist with the lifting, especially for heavy or awkward equipment and supplies. • Make sure the path of travel is clear before the lift.	M
	Slips, trips, and fall hazards	• Be cautious of drilling and sampling equipment laying on the ground. • Maintain good housekeeping. • Beware of uneven ground surface. • Work at a steady pace. • Keep walking paths open to help prevent tripping hazards. • Be aware that escarpments can slough. Avoid these areas. • Exercise caution in relying on rocks and trees or tree stumps to support yourself; many times they are loose. • Whenever possible, switchback your way up and down steep areas, and maintain a slow pace with firm footing. • Employees walking in ditches, swales, and other drainage structures adjacent to roads or across undeveloped land must use caution to prevent slips and falls, which can result in twisted or sprained ankles, knees, and backs. • Whenever possible, observe the conditions from a flat surface and do not enter a steep ditch or side of a steep road bed. • If steep terrain must be negotiated, coordinate with HSM to evaluate the need for ladders or ropes to provide stability.	L

ACTIVITY HAZARD ANALYSIS, ENVIRONMENTAL SAMPLING (SOIL, SW, SEDIMENT)

Job Steps	Hazards	Controls	RAC
	Hand injuries, cuts, and bruises	• Wear protective gloves. • Use hand tools in a safe manner and keep hand tools in good working condition. • Leather gloves should be worn when handling sharp, rough, or slippery surfaces.	L
	Mechanical hazards of sampling around vibracoring operation, including hammer system and flying debris and objects	• Only authorized personnel are permitted to operate the vibracore rig. • Stay clear of areas surrounding coring device during every startup. • Stay clear of the winches and rigging components of coring device. • Stay as clear as possible of all hoisting operations. Loads should not be hoisted overhead of personnel. • Do not wear loose-fitting clothing or other items such as rings or watches that could get caught in moving parts. Long hair must be restrained. • If equipment becomes electrically energized, personnel should be instructed not to touch any part of the equipment or attempt to touch any person who may be in contact with the electrical current. The utility company or appropriate party should be contacted to have line de-energized before approaching the equipment. • Smoking around drilling operations is prohibited. • Wear the appropriate PPE when sampling (refer to the HSP). • Use the appropriate lifting procedures when unloading equipment and sampling. Lift with legs and not the back. Use lifting aids if >40 pounds or awkward. • Allow ample space for personnel to lift/store coring rods. • Avoid movement near rig. Do not place hands near vibrating equipment. • If dermal contact with the groundwater and acid used in sample preservation occurs, wash exposed skin thoroughly with soap and water. • Avoid eating and drinking onsite and during sampling. • Use ear plugs during sampling if sampling involves a generator. • The rig must be equipped with a kill wire or switch, and personnel are to be informed of its location. • Be aware and stand clear of heavy objects that are hoisted overhead. • The driller is to verify that the rig is properly maintained in accordance with the drilling company's maintenance program. • The boat skipper is to verify that all machine guards are in place while the rig is in operation. • The boat skipper is responsible for housekeeping (maintaining a clean work area). • The boat should be equipped with at least one fire extinguisher. • The boat should be stabilized with spuds or anchor lines during coring operations. • Cease operations on the water if wind is too strong or wakes from other boats are making the operation unsafe.	M

ACTIVITY HAZARD ANALYSIS, ENVIRONMENTAL SAMPLING (SOIL, SW, SEDIMENT)

Job Steps	Hazards	Controls	RAC
	Exposure to potentially contaminated media	• Wear the appropriate PPE when sampling, including safety glasses, nitrile gloves, body PPE, and steel-toed boots (refer to the HSP). • If dermal contact with the contaminated media or sample preservatives occurs, wash exposed skin thoroughly with soap and water. Maintain the material safety data sheets and safety data sheets. • Avoid eating and drinking onsite and during sampling. • Containerize all investigation-derived waste material and transport to the appropriate storage or treatment area.	M
	Struck by or against heavy equipment	• Wear reflective warning vests when exposed to vehicular traffic. • Isolate equipment swing areas. • Make eye contact with operators before approaching equipment. • Understand and review hand signals.	M
	Falling into surface water	• Employees to wear USCG-approved PFDs if there is a risk of drowning • Inspect PFDs before each shift; do not use damaged items.	L
	Working from a boat	• Personnel who will operate a boat during the course of a project will first demonstrate to the site manager that they are experienced in operating boats similar to those used for the project, and is knowledgeable of the USCG Boating Safety requirements (33 CFR Subchapter S). Project boats will be operated by experienced and licensed or certified USCG marine vessel operators only. Boat operators will also possess basic mechanical knowledge necessary to troubleshoot common mechanical problems that can and do occur. The boat operator will be responsible for the safety of all personnel on board the boat he or she is operating and for the integrity of all boat and safety equipment. • Each designated boat operator will give a safety briefing to all occupants of the boat before leaving the shore. Boats are to be occupied during use by not less than one qualified operator plus one additional person. • The boat skipper has the final authority with regard to boat safety and navigational safety. • Complete the boat safety checklist in the site-specific HSP to evaluate and verify necessary equipment before leaving shore. • The boat captain is required to assess the weather forecast for each day and monitor the weather conditions throughout the day while on the water. If the weather forecast is not favorable or conditions on the water become hazardous due the weather, the boat captain has the final authority to cease all boating operations and return to port until conditions improve, and all personnel feel safe. • Keep hands away from hoists, wire, and rope. • When opening sample casing joints, position hands so that fingers will not be injured should the casing slip. • Keep tools clean and dry. • Approved fire extinguishers will be onboard and readily accessible. Personnel will be trained in the proper use of said fire extinguishers. • Keep boat free from tripping hazards.	M

ACTIVITY HAZARD ANALYSIS, ENVIRONMENTAL SAMPLING (SOIL, SW, SEDIMENT)

Job Steps	Hazards	Controls	RAC
		• Be aware of boat position and movement and communicate with the operator. • Good hygiene practices to be used at all times. Contact with the sediment should be considered potentially contaminated. No eating, drinking, smoking or chewing tobacco while performing this task. • Horseplay onboard the boat will NOT be tolerated.	

Acronyms/Abbreviations:

CFR – *Code of Federal Regulations*
HSE – health, safety, and the environment
HSM – Health and Safety Manager
HSP – Health and Safety Plan
MRS – munitions response site
PFD – personal flotation device
PPE – personal protective equipment
SW – surface water
USCG – U.S. Coast Guard
UXO/UXOSO – unexploded ordnance/unexploded ordnance safety officer

Equipment to be used	Training Requirements/Competent or Qualified Personnel Names(s)	Inspection Requirements
Hand Tools Marine vessel	40-hour HAZWOPER/8-hour refresher First aid/CPR Bloodborne Pathogens Dangerous Goods Shipping Waste Management Fire Extinguisher Initial and daily boat safety operations	Inspect tools before each use; look for defects; remove damaged and broken tools from service. Initial boat inspection performed by the SL-HW using checklist in the HSP, and daily inspections performed by the boat captain

	PRINT NAME	SIGNATURE	
Supervisor Name:			**Date/Time:**
Safety Coordinator Name:			**Date/Time:**
Employee Name(s):			**Date/Time:**
			Date/Time:
			Date/Time:
			Date/Time:
			Date/Time:
			Date/Time:
			Date/Time:
			Date/Time:
			Date/Time:

Pre-Task Safety Plan (PTSP)

Project: _____ Location: _____Date:_____

Supervisor: _____ Job Activity:_____
_____

Task Personnel:
_____
_____
_____

List Tasks:

_____
_____
_____
_____

Tools/Equipment Required for Tasks (ladders, scaffolds, fall protection, cranes/rigging, heavy equipment, power tools):

_____
_____
_____

Potential H&S Hazards, including chemical, physical, safety, biological and environmental (check all that apply):

__ Chemical burns/contact	__ Trench, excavations, cave-ins	__ Ergonomics
__ Pressurized lines/equipment	__ Overexertion	__ Chemical splash
__ Thermal burns	__ Pinch points	__ Poisonous plants/insects
__ Electrical	__ Cuts/abrasions	__ Eye hazards/flying projectile
__ Weather conditions	__ Spills	__ Inhalation hazard
__ Heights/fall > 6 feet	__ Overhead Electrical hazards	__ Heat/cold stress
__ Noise	__ Elevated loads	__ Water/drowning hazard
__ Explosion/fire	__ Slips, trip and falls	__ Heavy equipment
__ Radiation	__ Manual lifting	__ Aerial lifts/platforms
__ Confined space entry	__ Welding/cutting	__ Demolition

Other Potential Hazards
(Describe):_____
_____
_____
_____

Hazard Control Measures (Check All That Apply):			
PPE __ Thermal/lined __ Eye __ Dermal/hand __ Hearing __ Respiratory __ Reflective vests __ Flotation device	**Protective Systems** __ Sloping __ Shoring __ Trench box __ Barricades __ Competent person __ Locate buried utilities __ Daily inspections	**Fire Protection** __ Fire extinguishers __ Fire watch __ Non-spark tools __ Grounding/bonding __ Intrinsically safe equipment	**Electrical** __ Lockout/tagout __ Grounded __ Panels covered __ GFCI/extension cords __ Power tools/cord inspected
Fall Protection __ Harness/lanyards __ Adequate anchorage __ Guardrail system __ Covered opening __ Fixed barricades __ Warning system	**Air Monitoring** __ PID/FID __ Detector tubes __ Radiation __ Personnel sampling __ LEL/O2 __ Other	**Proper Equipment** __ Aerial lift/ladders/scaffolds __ Forklift/heavy equipment __ Backup alarms __ Hand/power tools __ Crane with current inspection __ Proper rigging __ Operator qualified	**Welding & Cutting** __ Cylinders secured/capped __ Cylinders separated/upright __ Flash-back arrestors __ No cylinders in CSE __ Flame retardant clothing __ Appropriate goggles
Confined Space Entry __ Isolation __ Air monitoring __ Trained personnel __ Permit completed __ Rescue	**Medical/ER** __ First aid kit __ Eye wash __ FA-CPR trained personnel __ Route to hospital	**Heat/Cold Stress** __ Work/rest regime __ Rest area __ Liquids available __ Monitoring __ Training	**Vehicle/Traffic** __ Traffic control __ Barricades __ Flags __ Signs
Permits __ Hot work __ Confined space __ Lockout/tagout __ Excavation __ Demolition __ Energized work	**Demolition** __ Pre-demolition survey __ Structure condition __ Isolate area/utilities __ Competent person __ Hazmat present	**Inspections:** __ Ladders/aerial lifts __ Lanyards/harness __ Scaffolds __ Heavy equipment __ Cranes and rigging	**Training:** __ Hazwaste __ Construction __ Competent person __ Task-specific (THA) __ HAZCOM
Field Notes:_____ _____ _____			

Name (Print): _____
Signature: _____ Date:_____

Attachment 7
Safety Data Sheet

Attachment 8
Dive Safety Plan

ASSOCIATED UNDERWATER SERVICES

DIVE OPERATIONS PLAN

Name of Project: **Portland Harbor Sediment Sampling**

AUS No.: **2440**

Upon approval of this dive plan please initial on the *Approved* line below, sign and return to sender.

X ~~Approved~~ ACCEPTED AFTER MULTIPLE REVIEWS BY HSE AND JACOBS DIVE SAFETY OFFICER(S)

___ Approved with corrections as noted on submittal data and/or attached sheet(s).

SIGNATURE: _____

TITLE: HEALTH AND SAFETY MANAGER DATE: 6/25/21

Submitted To:

Carrie Andrews
Senior Project Manager | Client Account Manager
Jacobs Engineering
2020 SW 4th Ave, Suite 300, Portland, Oregon 97201
O: (503) 736-4270 M: (503) 348-9500
carrie.andrews@jacobs.com

Submitted By:

Dustin Audley
Associated Underwater Services Inc.
6706 175th St Kenmore, WA 98028
O: (509) 533-6500 M: (206) 889-2540
Dustin@ausdiving.com

In accordance with ADCI consensus standards, OSHA regulations and Associated Underwater Services safe work manual presents the dive operations plan for the following project.

Title: **Portland Harbor Sediment Sampling**

Location: **Northwest Industrial.**
5924 NW Front Ave Portland, OR 97210

Date: **June 29th. 2021**

Client: **Jacods Engineering**

Scope of Work: The scope of work entails divers to collect sediment samples for chemical analysis from six designated locations under three docks. Three sub-samples of surface sediment will be collected from the top one foot (12 inches) of sediment in the immediate vicinity of each designated sampling location. Sample locations will be confirmed by an onsite Jacobs Engineering representative before collected. The volume of the sediment required shall be a minimum of 16 ounces at each location and double when duplicates are required. Samples will be collected with clear acrylic tubes equipped with core catchers or other similar single use sampling devices. The tubes will be capped and labeled with the sample location, subsample number, sampling date, and time prior to transfer to the shore-based Jacobs sample management crew. Immediately after samples are collected by the diver, the sample will be handed to the onsite Jacobs field team manager or designee.

In order to provide information in a clear and concise manner, the Dive Operations Plan has been divided into three sections identified by the following headings:

1.0 General Diving Plan
2.0 Emergency Management Plan
3.0 Activity Hazard Analysis

Section 1.0 addresses such issues as names and duties of each member of the dive team, general safety policies, a description of the work, ambient conditions, planned depth and time durations, and limits. Section 2.0 provides written guidelines for reacting to any emergency situation that may occur at the job site. Section 3.0 provides details on the hazards and safety issues particular to the job at hand using the Activity Hazard Analysis format.

"If for any reason the dive plan is altered in mission, depth, personnel, or equipment, **Marilyn Gauthier - gauthier@jacobs.com - phone number 425 894 6464,** shall be contacted and shall review any revision prior to actual operations."

Respectfully submitted,

Dustin Audley.

ASSOCIATED UNDERWATER SERVICES, INC.

1.0 DIVE PLAN

1.1 Dive Team Assignments and Responsibilities

PROJECT MANAGER: Dustin Audley…... (b) (6)

The dive team shall consist of a __**4**__-person dive team made up of the following:

DIVING SUPERVISOR (DIVING MASTER)

Responsible for dive safety and safe and efficient conduct of all diving activities above and below the water for this project.

Supervisor:	Drew Smith….	(b) (6)
Alternate:	Kris Hunter…..	
Alternate:	Dustin Audley…..	

DIVER:

Perform tasks as required and directed by diving supervisor including the following specific tasks:

Diver: Drew Smith	Diver: Adam Roehl
Diver: Dan Budzil	Diver: Dustin Audley
Diver: Dave Schneider	Diver: Greg Olson
Diver: Kris Hunter	Diver: Vincent O'Neal

STANDBY DIVER:

The standby diver is to be always alert and aware of the project and divers' duties. The standby diver will also assist as needed with rack operator, timekeeping, diver safety, as well as assist the diver in and out of the water, operate deck equipment, assist in topside work as required. The Standby diver will be alert and ready to assist in moment's notice:

Standby Diver: Anyone listed above.

TENDER:

Tend diver's umbilical, assist diver in and out of water, operate deck equipment and assist in topside work as required or directed.

Tender: Drew Smith	Tender: Adam Roehl
Tender: Dan Budzil	Tender: Dustin Audley
Tender: Dave Schneider	Tender: Greg Olson
Tender: Kris Hunter	Tender: Vincent O'Neal

TIMEKEEPER:
Track diver's bottom time.

Timekeeper: Anyone listed above

Diving personnel shall be assigned their duties prior to the start of any dive. These duties are to be assigned by the diving supervisor and may be changed from time to time as required. Divers are trained commercially qualified divers and are experienced in the tasks required in this project. Daily safety briefings scientific sampling procedures will be reviewed to ensure samples will be collected to meet the pre-design investigation work plan's data quality objectives.

All members of the crew will have the following certifications:

- ADC Certified Diver
- First Aid
- O2 Provider
- CPR
- OSHA 40 Hazwoper w/8HR Refresher

*Personnel certification records are provided in attachment #1

1.2 Date, Time, and Location of Dive Operations

Date: **June 29,2021**

Time: **10:00**

Location: **Northwest Industrial.**
5924 NW Front Ave Portland, OR 97210

Boat Launch: Cathedral City Park 8706 N Bradford St, Portland, OR

Sample Locations:

Sample.ID	Type	DD_X	DD_Y	X_IntlFeet	Y_IntlFeet
PD43	Under Dock Grab	-122.73882191200	45.56636380470	7628806.70112000000	700527.58038100000
PD12	Under Dock Grab	-122.73805216100	45.56799771710	7629020.26115000000	701117.63517100000
PD16	Under Dock Grab	-122.73722528900	45.56734279710	7629225.32579000000	700873.07053800000
PD20	Under Dock Grab	-122.73642141500	45.56671752810	7629424.80676000000	700639.47801800000
PD26	Under Dock Grab	-122.73788503100	45.56694359980	7629052.40092000000	700732.25295300000
PD34	Under Dock Grab	-122.73750492100	45.56605775370	7629140.76542000000	700406.69061700000

1.3 Safety Policy

All diving operations shall be conducted in accordance with the Associated Underwater Services, Inc. Safe Practices Manual, this job specific Dive Operations Plan, the Association of Diving Contractors Consensus Standards for Commercial Diving Operations, and OSHA Regulations as well as EPA's dive safety manual: incorporated by reference into the AUS Safe Practices Manual.

AUS Safety Practices Manual:

https://www.dropbox.com/sh/3tzrh74a1yhl983/AABIxe5z22maAd-VXDC6c3vOa?dl=0

EPA Dive Safety Manual:

https://www.epa.gov/diving/epas-diving-safety-program

PD12
PD16
PD20
PD26
PD34
PD43
Kinder Morgan Dock
Chevron Dock
Phillips 66 Dock
Tidewater Barge
-44
-42
-38
-36
-34
-32
-30
-28
-26
-24
-22
-20
-18
-16
-14
-12
-8
-6
-4
-2
2
4
6
8
12
14

Bathymetric survey conducted by David Evans Associates Inc (DEA)
Precision multibeam and single beam survey conducted between March 6, 2018 and June 15, 2018
North American Vertical Datum of 1988 (NAVD88)

Document Path: \\dc1vs01\GIS\Proj\Portland\Harbor\Group\B2_WillbridgeCove\MapFiles\Report_PDI\58_WC_DataGaps_Evaluation_Dock_Samples.mxd

Legend

Proposed Samples

- Under Dock Grab
- Bathymetry (2ft) Contours
- Willbridge Cove Project Area

0 75 150 300 Feet

FIGURE 1-1

PROPOSED UNDER DOCK SAMPLE LOCATIONS
OVERVIEW OF PRELIMINARY SMA EVALUATION
Portland Harbor Superfund Site
Willbridge Cove Project Area
Portland, Oregon

Integral Consulting Inc.

The Diving Supervisor (Diving Master) is ultimately responsible for the safety of all personnel and equipment working on the project. He will report directly to the Company President or Vice President on all matters concerning the safety of the operation.

The Diving Supervisor will conduct daily safety meetings each morning before work begins. Items of discussion will include coordination of the day's activities and a review of pertinent Activity Hazard Analysis forms. All dive personnel, and if applicable, other crew members i.e. crane operators, and barge or boat operators are required to attend the daily safety meetings. These meetings are an opportunity to address daily safety concerns. No work will be allowed to progress without the approval of the safety officer.

The site supervisors will conduct frequent safety inspections of the worksite, equipment and materials. Any deficiencies will be corrected as soon as possible and discussed at the next safety meeting.

After the completion of each dive, the dive supervisor shall:
Question each diver as to his physical condition.
Instruct divers to report any physical problems or adverse physiological effects, including symptoms of decompression sickness or gas embolism.
Advise the diver of the nearest location of an operational decompression chamber.
Alert each diver to the potential hazards of flying after diving.

In addition, the dive supervisor shall:
Take reasonable steps to have diver remain awake and in the vicinity of the decompression chamber for at least one hour.
Instruct diver to remain within two hours travel time of the decompression chamber for the next five hours.
Instruct divers of the hazards of flying at altitudes more than 800 feet within 12 hours after decompression.

A copy of this plan shall be on the dive location at all times.

1.4 Diving Mode

Diving mode shall be surface supplied air utilizing LP Breathing Air compressor for primary air supply and HP flasks for auxiliary air supply. All breathing airlines will be manifolded to allow for isolation to supply diver as required. A hardwire communication system will provide communication between the divers and the dive station. All operations will include CCTV with topside monitoring of video and audio signal.

Diving operations will be conducted from the AUS diving vessel Landing Craft. Divers will enter and exit the water via ladder from the platform. The vessel, dive gear, and other reusable equipment will be decontaminated before and after use at the Willbridge Cove site in accordance with the procedures identified in Attachment 2.

1.5 Description of Work

It is anticipated that diving operations shall include the below listed tasks. Divers will be equipped with a helmet mounted underwater color camera which will be always displayed on a monitor at the dive control station. A digital recorder will be connected inline, and all activities will be recorded. The work will be accomplished in the sequence in which it is listed.

- Launch vessel at Cathedral City Park.
- Conduct safety meeting.
- Sail to first sample location.
- Secure vessel to pier, piling. Set anchor point before deploying diver.
  * Live boating will not pe permitted on this project.
- Confirm there will be no marine traffic in the area when diver is in the water.
- Divers will enter the water at various locations.
- Divers will collect sediment samples at designated location.
- Photograph or video record underwater sampling activities at each location
- Diving equipment, personnel, and the support vessel will be decontaminated by rinsing and scrubbing with river water. Personnel will be decontaminated with buckets or low-pressure hoses while standing on the swim deck of the dive boat. The water will be allowed to return to the river and will not be containerized per the Willbridge Cove waste management plan.
- Demobilize from site.

1.6 Ambient Conditions

Divers will encounter underwater visibility of **0-10** feet. Water temperatures will be approximately **50-80** degrees F. Divers will wear vulcanized rubber dry suits for protection against contaminates. Divers will wear the appropriate undergarments for the current water temperature verified at the time of diving. River stage fluctuation (tidally influenced) Underwater currents in the vicinity of the work site will be verified at the time of dive.

1.7 Maximum Anticipated Depth and Bottom Times

Maximum water depth anticipated is **20** fsw. All diving will be performed within the no decompression limits of the USN Tables.

Dive Tables and Projected Depths

Dives will be conducted from waterline to less than **20 ft**. below waterline.
AIR will be utilized as a breathing media.

U.S. Navy Surface Supplied AIR Dive Tables will be used. There is a no required adjustment for altitude. The formula below will be used to determine the actual altitude adjustment depending on the water depth on the day of the dive.

$$\text{Equivalent Depth} = \text{Altitude Depth} \times \frac{\text{Pressure at Sea Level}}{\text{Pressure at Altitude}}$$

(Formula from "U.S. Navy Diving Manual Revision 7" Chapter 9-13)

Decompression diving is not anticipated. All diving personnel are competent with decompression diving and profiles. Personnel are all trained and experienced with the use of decompression schedules.

Anticipated dive profiles will be reviewed prior to the dive and documented on the Rough Dive Sheet. Dive depth will be calculated prior to the dive by using the current tailrace elevation and confirmed at the time of the dive using the pneumo.

Maximum anticipated dive profile would be **20** feet for **460** minutes.

BOYLE'S LAW

Boyle's law states that at constant temperature, the absolute pressure and the volume of gas are inversely proportional. As pressure increases, the gas volume is reduced; as the pressure is reduced, the gas volume increases

The formula for expressing Boyle's law is: $C = P \times V$

Where: C *is* constant
P *is* absolute pressure
V *is* volume

Boyle's law can also be expressed as: $P1V1 = P2V2$

Where: P1 = initial pressure
V1 = initial volume
P2 = final pressure
V2 = final volume

When working with Boyle's law, absolute pressure may be measured in atmospheres absolute. To calculate absolute pressure using atmospheres absolute:

$$Pata = \frac{\text{Depth fsw x 33 fsw}}{\text{33 fsw}} \quad \text{or} \quad Pata = \frac{\text{psig + 14.7 psi}}{\text{14.7 psi}}$$

$$P2 = \frac{20 + 33}{33} = 1.60 \text{ ATA}$$

$$V1 = \frac{1.60 \times 1.4 \text{ ACFM}}{1 \text{ ATA}} = 2.24 \text{ ACFM}$$

2.24 X 20 = 44.8 Cubic Feet Per Dive

100% in water oxygen will NOT be utilized. Should the dive duration or depth be shallower, the appropriate schedule will be utilized.

Divers will follow travel after diving at altitude discipline. Divers will adhere to Table 9-6 required surface interval before ascent to altitude after diving.

Table 9-7. *No-Decompression Limits and Repetitive Group Designators for No-Decompression Air Dives.*

Depth (fsw)	No-Stop Limit	Repetitive Group Designation															
		A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	Z
10	Unlimited	57	101	158	245	426	*										
15	Unlimited	36	60	88	121	163	217	297	449	*							
20	Unlimited	26	43	61	82	106	133	165	205	256	330	461	*				
25	1102	20	33	47	62	78	97	117	140	166	198	236	285	354	469	992	1102
30	371	17	27	38	50	62	76	91	107	125	145	167	193	223	260	307	371
35	232	14	23	32	42	52	63	74	87	100	115	131	148	168	190	215	232
40	163	12	20	27	36	44	53	63	73	84	95	108	121	135	151	163	
45	125	11	17	24	31	39	46	55	63	72	82	92	102	114	125		
50	92	9	15	21	28	34	41	48	56	63	71	80	89	92			
55	74	8	14	19	25	31	37	43	50	56	63	71	74				
60	63	7	12	17	22	28	33	39	45	51	57	63					
70	48	6	10	14	19	23	28	32	37	42	47	48					
80	39	5	9	12	16	20	24	28	32	36	39						
90	33	4	7	11	14	17	21	24	28	31	33						
100	25	4	6	9	12	15	18	21	25								
110	20	3	6	8	11	14	16	19	20								
120	15	3	5	7	10	12	15										
130	12	2	4	6	9	11	12										
140	10	2	4	6	8	10											
150	8		3	5	7	8											
160	7		3	5	6	7											
170	6			4	6												
180	6			4	5	6											
190	5			3	5												

* Highest repetitive group that can be achieved at this depth regardless of bottom time.

1.8 Equipment List

The following is a list of equipment that is expected to be used during operations to be conducted under this Dive Plan. Two oxygen kits with NRB's will be available to meet the EPA two diver standard. Before diving operations begin an alpha and recreational dive flags will be flown while divers are in the water. Equipment maintenance records are included in attachment #

1. Surface Supplied Dive Station
   - Surface supplied diving equipment
   - Minimum of two 250' dive hoses
   - Pneumofathometer
   - Communications equipment (two-way voice communications)

- Compressors with filters for breathing air.
- High pressure reserve back up Air
- First aid kit x2
- Emergency oxygen (Jumbo 'D' Cylinder 647 Liter Capacity (90min))x2
- Back Board with spider straps
- AED
- US Navy supervisor's manual
- ABC fire extinguisher
- Marine Radio VHF
- Sufficient fuel for the job
- Ladder (water egress)

2. HAZMAT Diving suits (fully contained) vulcanized rubber.
3. 316 stainless – 2-inch diameter x 20-inch-long sediment sampler with acrylic liner and plastic cone catcher
4. Underwater Video Equipment
5. Diving Vessel
6. Miscellaneous tools, probes, tamp measures.

Diver's personally supplied equipment will consist of the following:

1. Commercial diving helmet (Kirby Morgan - 17, 27, 37, 47, 57, or 77)
2. Dry suit (AUS to provide vulcanized rubber suit for this project)
3. Emergency bailout air supply (BOB) 30cf - 50cf (3000psi Air)
4. 4-point harness (ADCI requirements).
5. Minimum 2 diving knifes.
6. Weight belt
7. Fins, as needed.
8. Miscellaneous personal equipment for diver's preference.

Tenders Personal Protective Equipment (PPE)

1. Safety Glasses
2. Face shield
3. Rubber gloves or appropriate gloves for the task at hand
4. Tyvek Suit
5. Proper footwear
6. Life jacket

Divers will wear AUS provide vulcanized rubber suit for project. The divers Helmet will be mated to the vulcanized rubber suit, gloves will also be connected to the suit to insure there is no exposed skin. The top side crew will wear proper PPE to handle diving equipment in and out of the contaminated and decontamination zone. Personal flotation is required to be properly worn always, on an AUS diving Vessel. *Note 2 Emergency oxygen (Jumbo 'D' Cylinder 647 Liter Capacity (90min)) will be capable of providing two none breathing divers oxygen simultaneously.

1.9 Lock Out / Tag Out Procedures

Safe clearances will be obtained. Safe clearance procedures shall comply with Associated Underwater Services safe work manual. If a vessel is within one hundred yards of the

diving location **NO** operations shall be initiated until the diving supervisor has verified such procedures with any vessel. If a LOTO cannot be verified, diving operations will not commence. For this project no vessel is scheduled to be in the vicinity of diving operations.

1.10 Equipment & Training Certifications

Divers' equipment certifications, ADCI, HAZMAT, First Aid/CPR, DanO2 and Dive Medical exam is attached to this dive plan under employee certifications Attachment #1.

Air certification for breathing air compressors used on this project will be available for inspection. A copy of the air compressor's certificate of analysis showing the breathing air meets CGA grade E requirements will be available for review.

All on-site personnel will be trained in OSHA Regulation 29 CFR 1910.120 (HAZWOPER), with their 8-hour refresher course completed in the last 12 months. The site supervisors will have current ADCI Supervisor Cards. All crew members will have current first aid and CPR certificates.

Site-specific training will be accomplished by having on-site personnel read this Dive Safety Plan before work begins. The review will include a discussion of the chemical, physical, and biological hazards; protective equipment and safety procedures; and emergency procedures.

Daily safety meetings will be held to cover the work to be accomplished, hazards anticipated, PPE and procedures required to minimize site hazards, and emergency procedures. No work will be performed before the daily safety meeting has been held. The daily safety meeting must also be held prior to new tasks and repeated if new hazards are encountered.

All employees will be current in first aid, CPR and DAN O2. The Crew members have also received training regarding the precautions and protective equipment necessary to protect against exposure to blood-borne pathogens. The first aid and CPR training includes Automated External Defibrillator (AED) training and oxygen provider. One AED will be present and available on site as well as sufficient oxygen.

1.11 DECONTAMINATION OPERATIONS

Authorization to Enter.

Only personnel with the appropriate training and medical certifications (if respirators are required) will be allowed to work at the project site. See attachment #3 for employee certifications.

Site Orientation and Hazard Briefing

No person will be allowed in the work area during site operations without first being given a site orientation and hazard briefing. This orientation will be presented by the Health Safety Officer, or designee, and will consist of a review of this Dive and Safety Plan. This

review must cover the chemical, physical, and biological hazards; protective equipment; safe work procedures; and emergency procedures for the project. Following this initial meeting, safety meetings will be held each day before work begins.

Exclusion Zone (EZ)

An EZ may consist of a specific work area or may be the entire area of potential contamination. All employees entering an EZ must use the required PPE and must have the appropriate training and medical clearance for hazardous waste work. Because there are multiple project locations, the body of water and the underside of the boat and floating platform are the EZ for this project.

The floating equipment used for this job will include the AUS diving vessel Landing Craft. No smoking, eating, or drinking will be allowed in the EZ.

Contamination-Reduction Zone (CRZ)

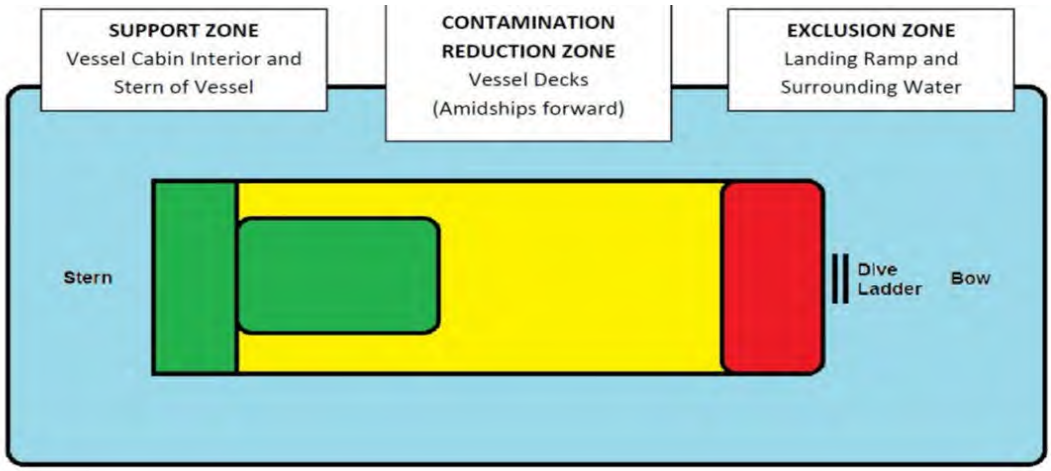
The CRZ or transition area will be established to perform decontamination of personnel and equipment. All personnel entering or leaving the EZ will pass through this area to prevent any cross-contamination. Tools, equipment, and machinery will be decontaminated in the CRZ, immediately outside the EZ. All personnel will be decontaminated on site adjacent to the EZ. Personal protective outer garments will be removed in the CRZ and prepared for cleaning or disposal. This zone is the only appropriate corridor between the EZ and the SZ.

For this specific project, the CRZ is initiated on the ladder and swim deck platform. All hand-held equipment will be passed to the tender, who will set everything in a labeled container for contaminated equipment. The diver decontamination process will be carried out on the platform where the diver will be sprayed down with water, and the dry suit will be removed from the diver. The diver will be considered to be in the SZ as soon as he is out of the dry suit and on the vessel deck.

AUS does not anticipate a need for containment of any runoff from the decontamination on the floating platform. The water will be allowed to flow back into the river after use.

Support Zone (SZ)

The SZ is a clean area outside the CRZ located to prevent employee exposure to hazardous substances. Eating and drinking will be permitted in the support area only after proper decontamination. For this project, the wheelhouse and back deck of the vessel will be SZ.



1.12 Decontamination

Topside Personnel Decontamination

All personnel wearing protective equipment in the EZ must undergo personal decontamination prior to entering the SZ. The diver will be rinsed off before any equipment is removed. Any equipment entering the water will be rinsed off before being taken to the next dive location.

The personnel decontamination area will consist of the following steps, at a minimum:
• Step 1 — Personnel leaving the contaminated zone will remove the gross contamination from their outer clothing and boots.
• Step 2 — Personnel will remove their outer garment and gloves and dispose of them in properly labeled containers. Personnel will then decontaminate their gear and boots with water.
• Step 3 — Personnel will thoroughly wash their hands, face and any exposed skin before leaving the CRZ.

Equipment Decontamination

All vehicles have entered the EZ will be decontaminated on shore. Since the level of contamination is low, decontamination will be limited to rinsing tires, wheel wells and the underside of the floating equipment (Vessel) with water.

PPE Decontamination

Where and whenever possible, single-use, external protective clothing will be used for work within the EZ or CRZ. This protective clothing will be disposed of in properly labeled containers. Reusable protective clothing will be rinsed on site with detergent and water. The contaminated rinse water will be collected for disposal.

Hazardous Waste Monitoring

AUS does not have employee medical monitoring for Hazardous Waste sites because our divers do not work greater than 30 days performing Hazardous Waste work. All divers have up to date annual ADCI physicals.

2.0 EMERGENCY MANAGEMENT PLAN

First aid supplies shall be provided and kept readily accessible at the work site. In addition, an American Red Cross standard first aid handbook or equivalent, medical O_2, and a bag type manual resuscitator and tubing shall be available at the dive location.

In the event of a decompression sickness incident, treatment shall be in accordance with the guidelines provided in the ADC Consensus Standards for Commercial Diving Operations.

The primary means of activating emergency services shall be to dial 911. Emergency contact numbers will be available and posted at the work site. A list of primary contact numbers is provided in this section.

In the event of the need for ambulance transport, the ambulance will be instructed of where to meet the crew.

Recommended procedures have been developed to deal with accidents and/or emergency situations should they occur. Table 2-1 provide a list of potential emergency situations that may arise and suggested actions to be taken in the event of an occurrence.

EMERGENCY SITUATION	RECOMMENDED ACTION
Entrapped or fouled diver	Provide diver a reasonable amount of time to clear himself. In the event he is unable to free himself, the standby diver will enter the water to assist. Once diver is free, if shaken or standby diver was required to go to his assistance, terminate dive.
Loss of breathing medium	The diving supervisor will switch to the standby supply at the dive manifold and immediately surface the diver on appropriate decompression schedule. Standby diver should be alerted immediately and stand ready to assist the diver as required. If diver is not receiving gas from the standby supply, he should be instructed to go to the bail out and be surfaced immediately using surface decompression schedules to satisfy any decompression commitments.

EMERGENCY SITUATION	RECOMMENDED ACTION
Loss of communication	Go to line pull signals and surface the diver on appropriate decompression table. If line pull signals cannot be established then standby diver will enter the water and swiftly advance following the primary diver's hose to aid the primary diver in his ascent to the surface.
Lost diver	Avoid panic and have diver review recent movements to ascertain general vicinity of diver. Have diver turn on mask free flow and look for bubbles to verify position. If position cannot be verified, have diver follow his hose back until he recognizes where he is.
Injured diver	Diver immediately informs topside of the nature and extent of injury. Dive is aborted and diver is surfaced either by himself or with the air of the standby diver. Proper decompression schedules should be followed except when the severity of the injury indicates a greater risk than omitting decompression. Monitor divers breathing during ascent. If breathing stops, overpressure divers regulator, if possible. Request medical assistance and emergency evacuation as required.
Equipment failure with diver in the water	Evaluate effect of failure on diver. Inform diver of plan of action. Alert standby diver and topside crew. When diver acknowledges he is ready, activate plan and terminate dive if required.
Fire in equipment or aboard dive platform	Extinguish fire and secure equipment. Determine damage and effect on diver. If required, terminate dive using appropriate decompression schedule.
Diver loss of consciousness.	Dive is aborted and diver is surfaced with the aid of the standby diver.

EMERGENCY SITUATION	RECOMMENDED ACTION
	Monitor diver's breathing during ascent. If breathing stops, overpressure diver's regulator, if possible. Utilize floating stokes litter to manually lift diver from water by crew. Utilize contractor's crane to lift litter onto dock if necessary. Request medical assistance and emergency evacuation as required. Notify emergency services
Dry suit/Helmet/Neck Leakage	Dive is aborted and diver is brought to the surface. Follow PPE and equipment decontamination procedures and wash diver's skin with a soap/water solution.

EMERGENCY CONTACT NUMBERS

Hyperbaric Facility

Virginia Mason Hospital (Seattle) ---------- (206) 583-6433
1100 9th Ave, Seattle, WA 98101

Providence Hospital (Portland)---------- (503) 215-6061
4805 NE Glisan St, Portland, OR 97213

Deaconess Hospital (Spokane)
Hyperbaric Unit---------- (509) 473-7005
24 Hour Emergency---------- (509) 473-7100

Hospital Facility

Legacy Emanuel Medical Cente---------- (503) 413-2000
2801 N Gantenbein Ave, Portland, OR 97227

Providence Hospital (Portland)---------- (503) 215-6061
4805 NE Glisan St, Portland, OR 97213

Providence Portland Medical Center---------- (503) 215-1111
4805 NE Glisan St, Portland, OR 97213

Helo Evacuation

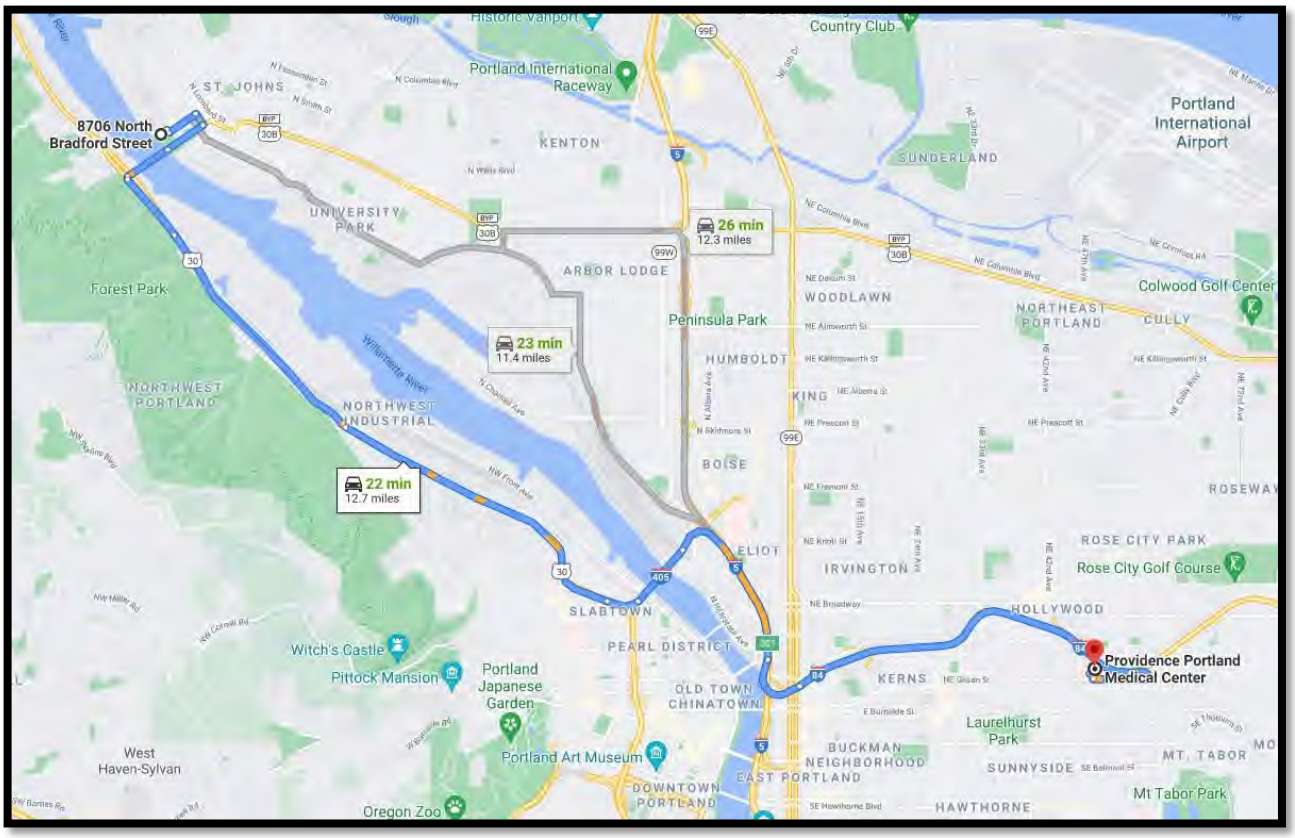
Air Evac International---------- (800) 854-2569
Critical Air (Tri-Cities)---------- (800) 247-8326
Air Lift Northwest---------- (800) 426-2430

Divers Alert Network (DAN) 24 Hours---------- (919) 684-9111
(919) 684-4326

US Coast Guard Marine Safety Office---------- (503) 240-9310

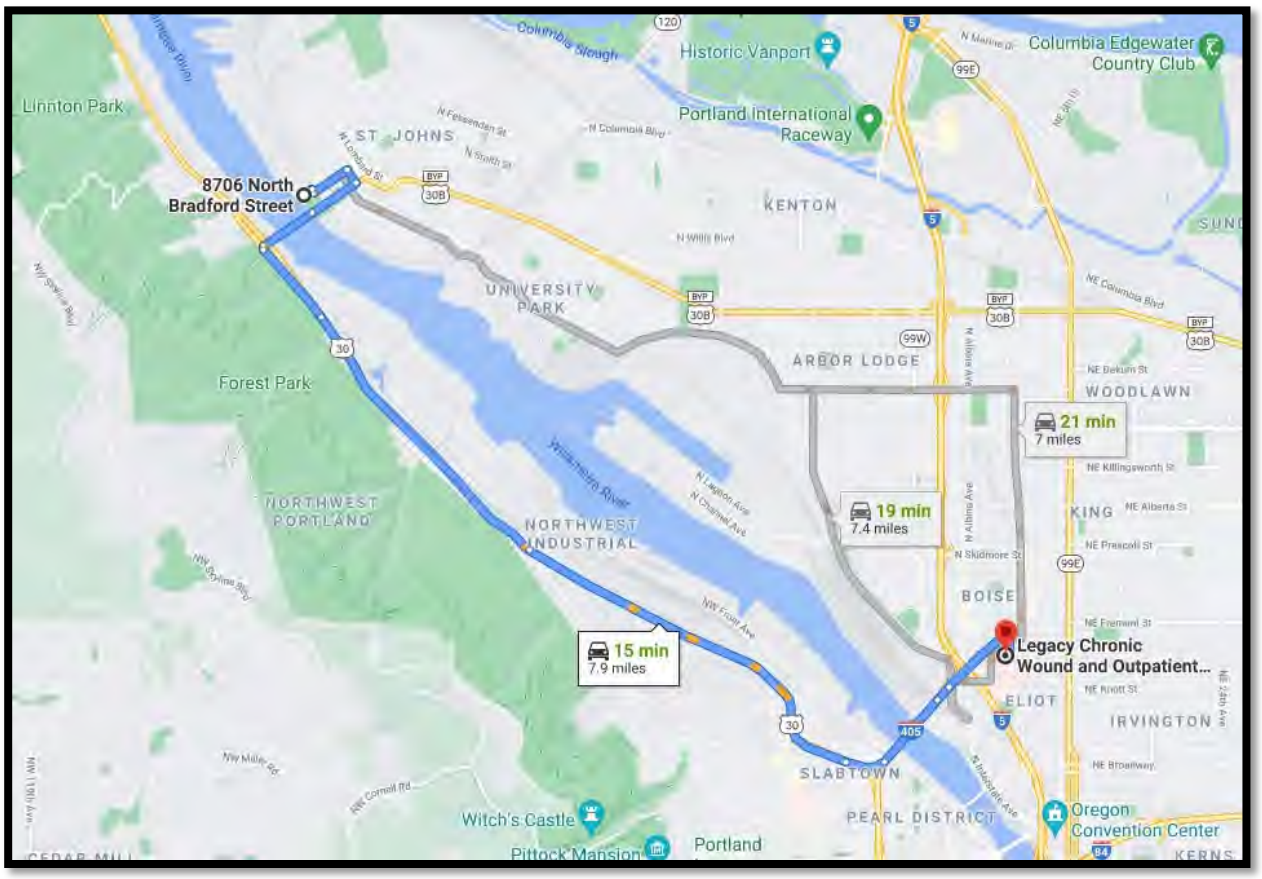
Associated Underwater Services, Inc.---------- (509) 533-6500
24 Hours Contacts:
Kerry Donohue ---------- (b) (6)
Dustin Audley ----------
Kirk Neumann ----------

Providence Portland Medical Center



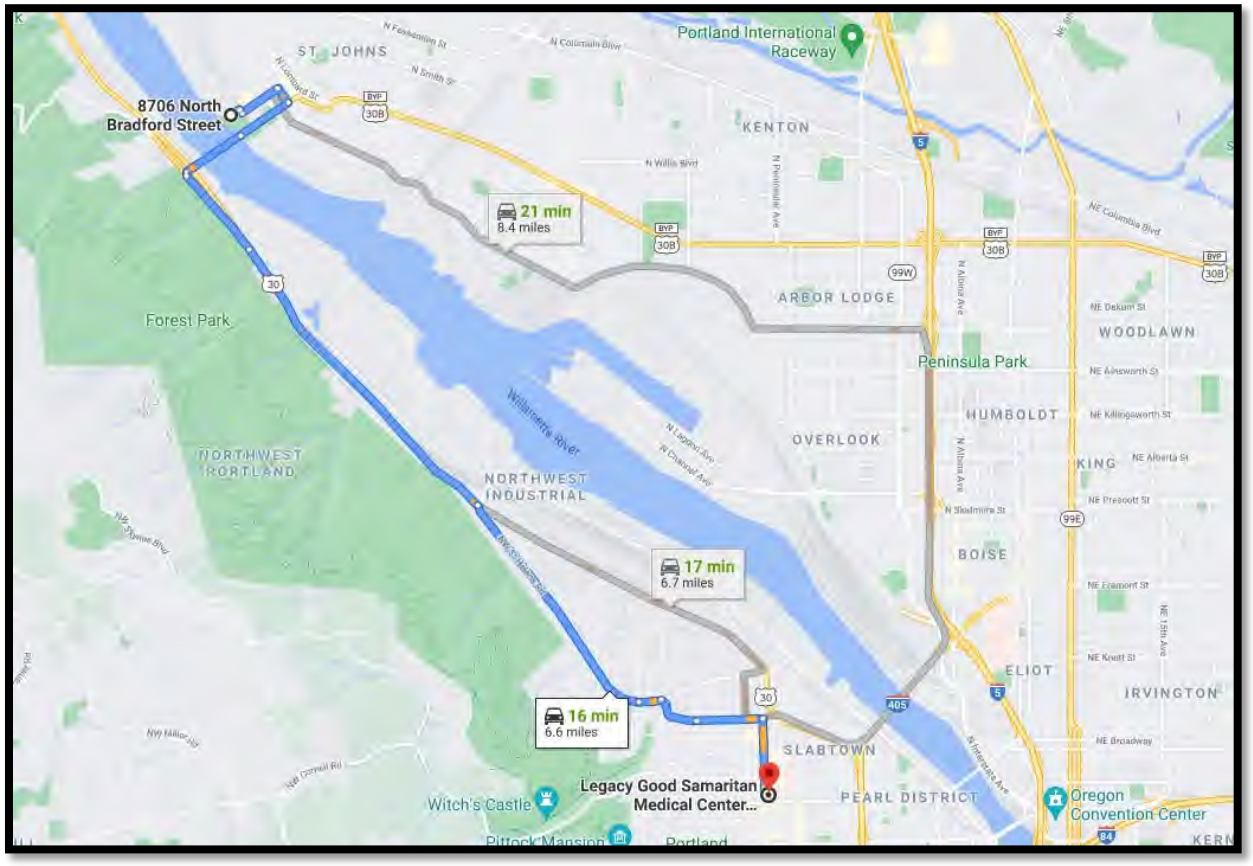
- 8706 N Bradford St Portland, OR 97203
- Take N Baltimore Ave to N Philadelphia Ave3 min (0.4 mi)
- Take NW St Helens Rd, US-30 E, I-5 S and I-84 E/US-30 E to NE Glisan St. Take exit 3 from I-84 E/US-30 E17 min (11.7 mi)
- Continue on NE Glisan St to your destination2 min (0.5 mi)
- Providence Portland Medical Center
  4805 NE Glisan St, Portland, OR 97213

Legacy Chronic Wound and Outpatient Burn Clinic



- 8706 N Bradford St Portland, OR 97203
- Take N Baltimore Ave to N Philadelphia Ave 3 min (0.4 mi)
- Take NW St Helens Rd and US-30 E to N Gantenbein Ave. Take the Kerby Ave exit from I-405 N/US-30 E 11 min (7.3 mi)
- Continue on N Gantenbein Ave to your destination 59 s (0.1 mi)
- Legacy Chronic Wound and Outpatient Burn Clinic
  3001 N Gantenbein Ave, Portland, OR 97227

Legacy Good Samaritan Medical Center Emergency Room



- 8706 N Bradford St Portland, OR 97203
- Take N Baltimore Ave to N Philadelphia Ave 3 min (0.4 mi)
- Continue to NW St Helens Rd 5 min (3.3 mi)
- Continue on NW St Helens Rd to your destination min (2.8 mi)
- Legacy Good Samaritan Medical Center Emergency Room
  1015 NW 22nd Ave, Portland, OR 97210

3.0 JOB SAFETY ANALYSIS

The Job Safety Analysis is an important tool used to identify and analyze all of the hazards associated with each task on a given project and then to come up with a safe working procedure to eliminate or alleviate situations before they become a serious problem.

The following table provides a complete list of hazards that can be assumed to exist on this project:

ACTIVITY HAZARDS ANALYSIS

ACTIVITY: ANALYZED BY/DATE: Samantha C 6/11/2021 REVIEWED BY/DATE: Dustin A 6/11/2021

PRINCIPAL STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS
Mobilization of Equipment	Rigging failure	Use only approved rigging in good condition. Use proper rigging techniques.
	Uncontrolled movement of equip.	Ensure all equipment is secured to deck for transport.
	Pinch Points	Personnel instructed in the hazards associated with working around cranes. All hands stand clear of equipment and other items on crane hook. Tag lines to be used to control loads on hook.
Fueling Equipment	Spillage of fuel	Ensure fuel containers and transfer procedures comply with USACE Safety and Health Requirements Manual. Adequate quantities of spill containment equipment will be stationed in the immediate area.
	Fire	Be aware of all sources of ignition. Personnel trained to be aware of inherent dangers. Fire extinguishing equipment will be on hand and ready for use to control fire.
	Inappropriate fuel into equipment	All containers and equipment to be well marked.
Operating LP Compressors	Rotating parts and release of compressed air	All personnel to be familiar with machinery onsite and instructed on safe starting procedures.
Use of HP Cylinders	Catastrophic failure of HP Bottles	All bottles onboard to be in good condition and with DOT specified hydrostatic testing criteria.
	Cylinder valve failure	Cylinder and valves in good condition. Ensure cylinders are current with hydrostatic testing requirements.
	Release of HP air	Cylinder and valves in good condition and properly connected. Protect face and head upon initial charging of valve.
	Freezing of valves on HP cylinders	Monitor manifolds during low temperatures for signs of icing. Protect and heat valves as required. In temperatures below 20°F, all HP bottles and connected valves shall be stored in a heated enclosure.
Underwater activities	Loss of voice communication, primary diver	Ensure communication equipment is in good condition and receives proper routine maintenance.

PRINCIPAL STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS
	Loss of primary air	Ensure proper maintenance on all life support equipment. Prior to diver entering water, check that all systems (primary and standby) are functional. For nitrox dives, alternative decompression schedules that may be required by a change of breathing medium (e.g. from nitrox to air) should be reviewed prior to diver entering water. Dives should be planned to that such an occurrence would not put the diver into an extreme decompression situation.
	Diver entanglement or trapped diver	Review dive plan to identify potential obstructions such as lines, cables, and structural appurtenances. If entanglement or a trapped diver situation arises, the diver will be given a reasonable amount of time to clear himself. However, in the event he is unable to free himself, the standby diver will enter the water to assist.
	Injured diver	Comprehensive safety meeting to take place at the beginning of each shift to discuss all potential hazards. Diver to follow all safety precautions.
	Hypothermia	Diver to be dressed out appropriately for underwater conditions. Dive supervisor shall be aware of symptoms and surface diver immediately upon signs of a hypothermic condition.
	Decompression sickness	All dives shall be in accordance with US Navy Decompression Tables. Any sign of decompression sickness will be treated with the appropriate USN treatment procedures.
	Vessel traffic	Dive flag will be posted at dive site prior to diver entering the water. In addition, a battery powered loud hailer will be on the dive station. If, in spite of attempted warnings, approaching vessel(s) do not acknowledge warning; diver will be told to return to stage until hazard is cleared.
	Heavy current	If a sudden, heavy current exists, diver will return to surface until flow is reduced to acceptable limits. Designated dive supervisor in conjunction with the diver shall determine the condition under which safe and effective work can be performed.

PRINCIPAL STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS
	Wind and waves Lighting	Diving Supervisor will determine if it is possible for a diver to safely enter and exit the water routinely and in an emergency. Movement and wave height also must be such that any in-water decompression stops are not compromised. Adequate lighting will be provided to insure a safe working condition.
Vulcanized Rubber dry suit, gloves, and diving helmet	Overinflation, leaks,	Ensure hat mates to suit and a proper seal can be made. Visually inspect suit for proper working condition and no damage Experienced and familiar with Dry Suit diving Inspection of valve, seals, o rings
Weather, temperature	Hyperthermia / hypothermia	Hyperthermia caused by heat stress, ensure diver and topside crew is well hydrated and proper PPE is worn for the current condition. Hypothermia Caused by cold. Ensure diver and crew have the proper PPE for the current condition in and out of the water. Wormer undergarments

EQUIPMENT TO BE USED	INSPECTION REQUIREMENTS	TRAINING REQUIREMENTS
LP Air Compressor	Visual inspection of moving parts. Verify filter maintenance is current.	General orientation and component location. Operating instructions.
Video System	Proper power and GFI connections	Connection and operating instructions
Rotary Compressor	Visual inspection of moving parts	General orientation and component locations. Operating instructions
Air Tools	Visual inspection of moving parts. Air connections made up properly and safety wire in place. Deck whips in good conditions. Proper air supply	General orientation and component function. Operating instructions. Work instructions.

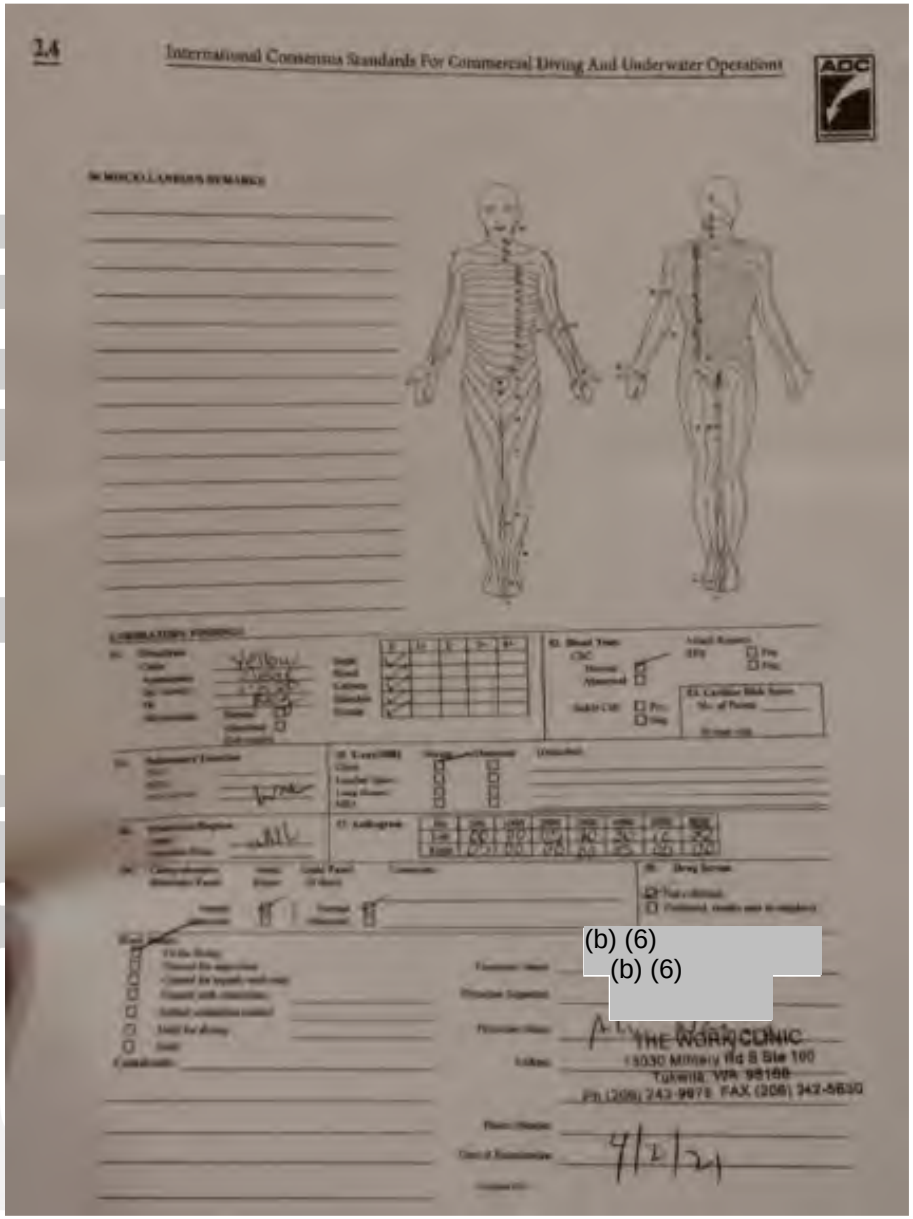
Work Activity Sequence (Identify the principal steps involved and the sequence of work activities)	**Potential Health and Safety Hazards** (Analyze each principal step for potential hazards)	**Hazard Controls** (Develop specific controls for each potential hazard)
Mobilize vehicle and boat trailer to the boat ramp.	Loose connection at trailer hitch. Non-working taillights on the boat trailer. Improper tire pressure in vehicle and trailer.	The connection at the hitch will be checked each and every time prior to tow; this includes the safety latch over the ball joint is secure and locked and that the safety chains are properly secured to the vehicle. Cable connections for the trailer lights will be checked and verified that they are working. Tire pressure will be visually inspected daily and checked with an air pressure instrument at each fuel fill-up. • Perform vessel inspection prior to deployment of craft and document.
Launch vessel	Vehicle driver unable to see all obstructions in view while backing trailer up. Truck/workers may slip on wet algae coated ramps, especially steeper ones. Interfering with public and other personal water crafts.	All workers will wear USCG approved Personal Floatation Devises while Launching the boat. This will be performed with at least two workers present. The second worker will act as a visual guide for the vehicle driver to point out all obstructions that the driver cannot see. Inspect boat ramps before using them to become familiar with their environment; use parking brake if needed, and take caution with footing to prevent slips, trips, and falls. Being patient around public boat ramps is necessary especially around the holiday weekend where overcrowding may result. Keep winch strap attached to the bow of the boat until boat just floats off of the trailer. Then connect a bow line to the bow of the boat and then remove the winch strap in order to ensure that control of boat is maintained at all times. Standing clear of the trailer and the boat have the vehicle slowly drive the trailer out from under the boat with the second worker maintaining control of the boat using the bow line.
Mobilize boat to dive locations.	Insufficient fuel. Unfamiliar waters. Non-communication.	Longer commutes will take place periodically and for this reason, fuel is an important factor. An extra reserve tank will be on board that will have a fuel line connection, and if needed, will be a simple change in plug connection from one tank to the other. Re-fueling must be performed when the engine is off, and in a well-ventilated area. Always be aware of fuel levels and know that your destination is only the halfway point and that much fuel is needed to get back. Do not drive at full throttle unless there is a 100% confidence level with the location. Speed should vary accordingly with familiarity. Always provide the daily float plan to the other workers on land. Walkie-talkie radios and /or cell phones should be on and charged. 30-minute communication checks will take place between the two parties.

Work Activity Sequence (Identify the principal steps involved and the sequence of work activities)	**Potential Health and Safety Hazards** (Analyze each principal step for potential hazards)	**Hazard Controls** (Develop specific controls for each potential hazard)
Vessel operations during diving and sampling	Lose focus of surroundings while paying more attention to sampling. Foul weather.	During data collection, focus will always be priority of other surroundings such as other watercrafts or obstructions in the water especially near the shoreline. Before each data collection stint begins, a full circle visual inspection will be performed of the surroundings. During the data collection time, constant visuals will take place. Review the weather report every day before entering the water. Sampling will not be planned if thunderstorms or lightening is in the forecast. If caught suddenly in foul weather while aboard the survey boat, work will be halted; all electronics will be turned off immediately and seek proper shelter.
Working onboard the vessel	Fall in water, drowning. Tripping hazard Back Injury Hand injury	• Always wear PFD and maintain footing while working from bow. • Keep boat free from tripping hazards. • Be aware of vessel position and movement and communicate with the operator. • Always maintain good center of gravity inside the boat. • Use caution when retrieving equipment and divers from the water, or when moving equipment about the vessel; use proper lifting and bending techniques when handling • Good Hygiene practices to be always used. • Use care and wear leather gloves if needed to prevent injuring finger or hand on sharp, heavy objects.
While on water.	Insect bites. Sun burn. Dehydration. Emergency plan.	Bug repellant spray and sunscreen will be onboard and used accordingly. A first aid kit will be onboard. Plenty of water and ice will be in a cooler, figure at minimum, one gallon of water per person. Water should be consumed at least every 15-20 minutes throughout the workday. A printout of the company Health and Safety Plan (HSP) will be onboard and reviewed. The HSP will have all emergency contacts listed. Refer to on protection from Biological Hazards Heat Stress.
While on water.	Falling into water. Heat/cold stress. Fire hazard from gas powered motor.	If a worker falls into the water, a life vest/ring should be thrown to this person immediately. If in the vicinity of the motor, it should be turned off immediately. The worker will be assisted getting back on board, work will be halted, and he/she will be taken back to the boat ramp. Proper clothing will be worn at all times and may vary depending on temperature conditions. Each

Work Activity Sequence (Identify the principal steps involved and the sequence of work activities)	**Potential Health and Safety Hazards** (Analyze each principal step for potential hazards)	**Hazard Controls** (Develop specific controls for each potential hazard)
		worker will be aware of each other and act on any heat/cold stress signs that are noticed. Proper type fire extinguisher will be on board in case of a fire hazard that could be caused from the gas-powered motor. Refer HSP to 2.1.3 Heat Stress.
Returning to the boat ramp, pulling boat out of the water, driving back to the hotel, securing equipment.	Tiredness at the end of the workday can cause mistakes.	All workers will wear USCG approved Personal Floatation Devises while pulling the survey boat out of the water. The same procedures will abide for these tasks as described above. However, at this point in the day, tiredness from being on a boat in the water for a full day can be multiplied. Maintain focus during this time and watching out for each other is extremely important to prevent accidents. Ensure that winch strap is reattached to the bow of the boat before the trailer and boat are pulled from the water.

Attachment 1

Association of Diving Contractors
International
ADCI
Cert. # (b) (6)
Expires 03/10/2026
MIXED GAS DIVER
DAVE SCHNEIDER I.D. (b) (6)
Commercial Diver Certification Card


24
International Consensus Standards For Commercial Diving And Underwater Operations
ADC
(b) (6)
(b) (6)
THE WORK CLINIC
Tukwila, WA 98188
4/1/24

Record of Completion

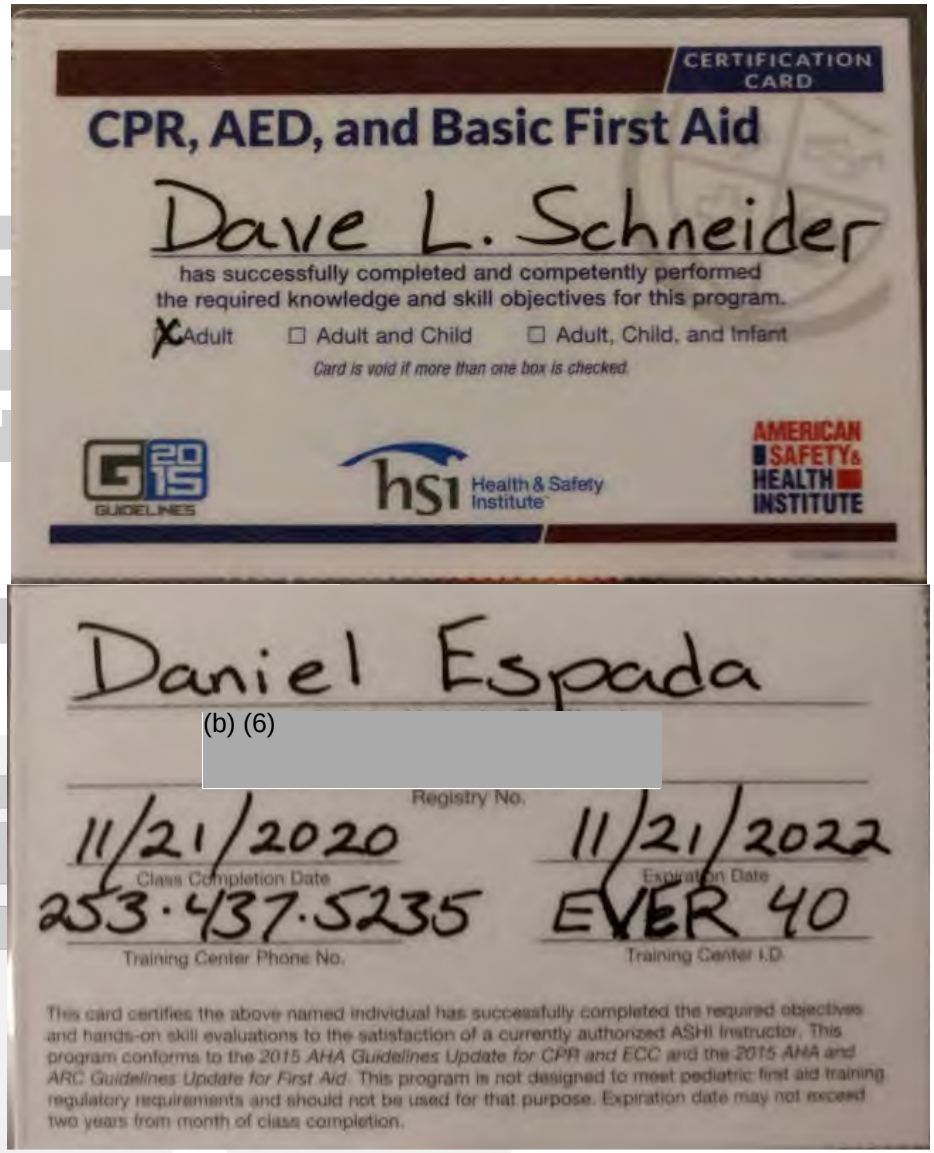
Dave Schneider

has successfully completed all the requirements for the course

HAZWOPER 8-Hour Refresher

Completion Date: 4/8/2020

VIVID learning systems



CERTIFICATION CARD

CPR, AED, and Basic First Aid

Dave L. Schneider

has successfully completed and competently performed the required knowledge and skill objectives for this program.

X Adult ☐ Adult and Child ☐ Adult, Child, and Infant

Card is void if more than one box is checked.

2015 GUIDELINES

hsi Health & Safety Institute

AMERICAN SAFETY & HEALTH INSTITUTE

Daniel Espada

(b) (6)

Registry No.

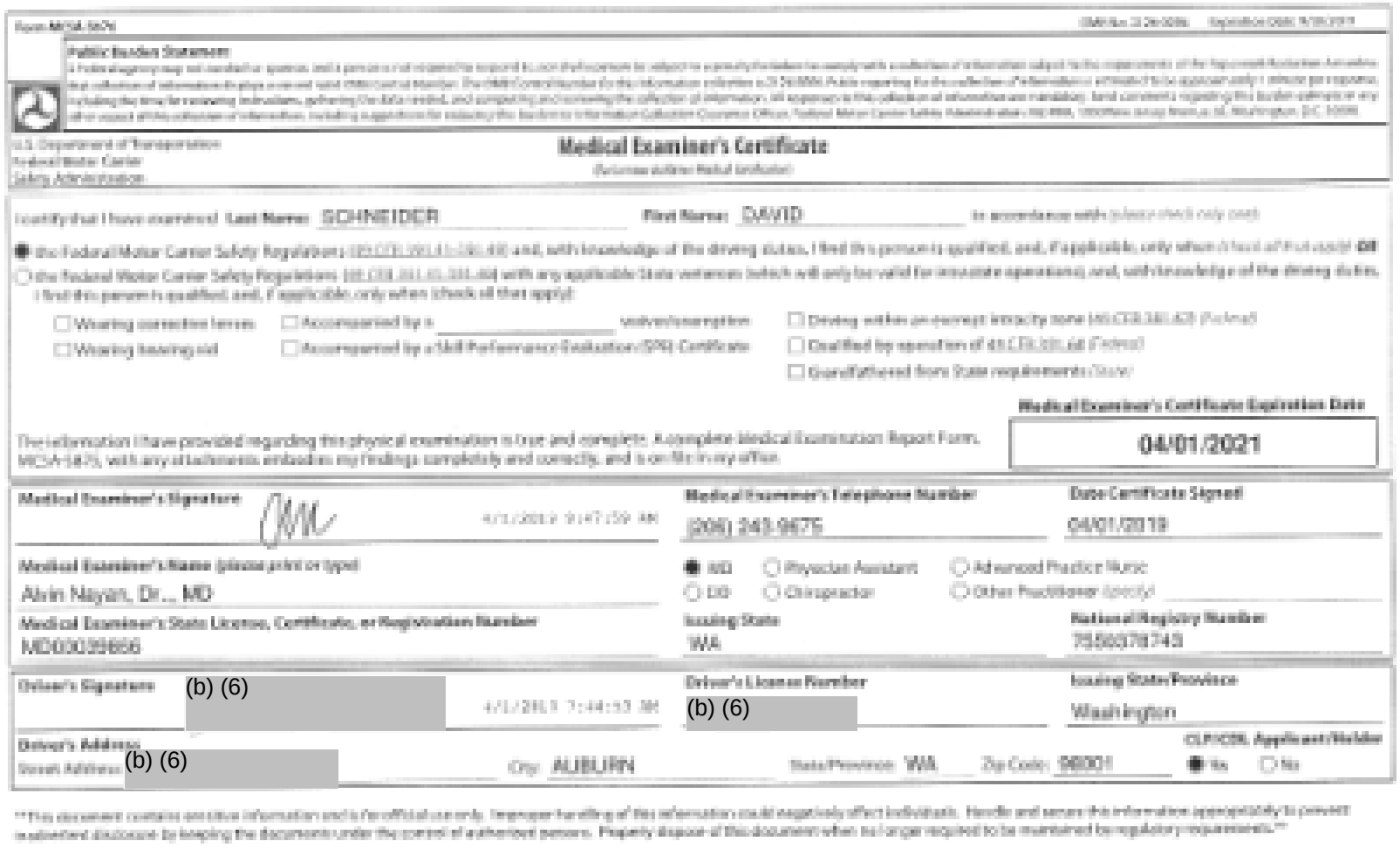
11/21/2020
Class Completion Date

11/21/2022
Expiration Date

253·437·5235
Training Center Phone No.

EVER 40
Training Center I.D.

This card certifies the above named individual has successfully completed the required objectives and hands-on skill evaluations to the satisfaction of a currently authorized ASHI Instructor. This program conforms to the *2015 AHA Guidelines Update for CPR and ECC* and the *2015 ANA and ARC Guidelines Update for First Aid*. This program is not designed to meet pediatric first aid training regulatory requirements and should not be used for that purpose. Expiration date may not exceed two years from month of class completion.



Form MCSA-5876 OMB No. 2126-0006 Expiration Date: 9/30/2019

Public Burden Statement

A Federal agency may not conduct or sponsor, and a person is not required to respond to, nor shall a person be subject to a penalty for failure to comply with a collection of information subject to the requirements of the Paperwork Reduction Act unless that collection of information displays a current valid OMB Control Number. The OMB Control Number for this information collection is 2126-0006. Public reporting for the collection of information is estimated to be approximately 1 minute per response, including the time for reviewing instructions, gathering the data needed, and completing and reviewing the collection of information. All responses to this collection of information are mandatory. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Information Collection Clearance Officer, Federal Motor Carrier Safety Administration, MC-RRA, 1200 New Jersey Avenue, SE, Washington, D.C. 20590.

U.S. Department of Transportation
Federal Motor Carrier
Safety Administration

Medical Examiner's Certificate

(Formerly Additional Medical Certificate)

I certify that I have examined **Last Name:** SCHNEIDER **First Name:** DAVID in accordance with (please check only one):

- ● the Federal Motor Carrier Safety Regulations (49 CFR 391.41-391.49) and, with knowledge of the driving duties, I find this person is qualified, and, if applicable, only when (check all that apply) **OR**
- ○ the Federal Motor Carrier Safety Regulations (49 CFR 391.41-391.49) with any applicable State variances (which will only be valid for intrastate operations), and, with knowledge of the driving duties, I find this person is qualified, and, if applicable, only when (check all that apply):

- ☐ Wearing corrective lenses
- ☐ Wearing hearing aid
- ☐ Accompanied by a _____ waiver/exemption
- ☐ Accompanied by a Skill Performance Evaluation (SPE) Certificate
- ☐ Driving within an except intracity zone (49 CFR 391.62) (Federal)
- ☐ Qualified by operation of 49 CFR 391.64 (Federal)
- ☐ Grandfathered from State requirements (State)

The information I have provided regarding this physical examination is true and complete. A complete Medical Examination Report Form, MCSA-5875, with any attachments embodies my findings completely and correctly, and is on file in my office.

Medical Examiner's Certificate Expiration Date: 04/01/2021

Medical Examiner's Signature: [signature] 4/1/2019 9:47:59 AM

Medical Examiner's Telephone Number: (206) 243-9675

Date Certificate Signed: 04/01/2019

Medical Examiner's Name (please print or type): Alvin Nayan, Dr., MD

- ● MD
- ○ DO
- ○ Physician Assistant
- ○ Chiropractor
- ○ Advanced Practice Nurse
- ○ Other Practitioner (specify)

Medical Examiner's State License, Certificate, or Registration Number: MD00039656

Issuing State: WA

National Registry Number: 7550378743

Driver's Signature: (b) (6) [redacted] 4/1/2019 7:44:53 AM

Driver's License Number: (b) (6) [redacted]

Issuing State/Province: Washington

Driver's Address:
Street Address: (b) (6) [redacted] City: AUBURN State/Province: WA Zip Code: 98001

CLP/CDL Applicant/Holder: ● Yes ○ No

This document contains sensitive information and is for official use only. Improper handling of this information could negatively affect individuals. Handle and secure this information appropriately to prevent inadvertent disclosure by keeping the documents under the control of authorized persons. Properly dispose of this document when no longer required to be maintained by regulatory requirements.

Helmet/Mask Inspection Certificate

CUSTOMER: Dave Schneider **DATE:** 1/24/2021

HELMET TYPE: _____ **COLOR:** _____ **S/N:** 00030K

Check for alteration or modification: _____

Verify Condition and/or Function of:

View Port: Good **Comms:** Fair/Clear

Neck Dam: Good **Clasp and Latch:** Good/Worn

Regulator: Good **Free Flow Valve:** Fair

Check Valve: Good **Emerg. Valve:** Fair

Exhaust Valves: Good **Rubber Parts:** Great

KMDSI Annual O-Ring Kit Installed: ✓

**"INSPECTED IN ACCORDANCE WITH KMDSI ANNUAL MAINTENANCE INSPECTION AND OVERHAUL CHECKLIST. FOR DETAILS, SEE KMDSI WEB SITE: www.kmdsi.com

Insert Test: YES ✓ **NO** _____ **(See Attached Sheet)**

Comments and Recommendations _____

NOTE: O-RINGS SHOULD BE LUBRICATED WITH SILICONE GREASE FOR MAXIMUM LIFE. THE DEMANDS OF YOUR WORKING ENVIRONMENT WILL DICTATE THE NEED TO REPLACE O-RINGS, AS WELL AS THE FREQUENCY OF YOUR DIVING. AT A MINIMUM, ALL O-RINGS SHOULD BE REPLACED ONCE A YEAR, OR EVERY 400 OPERATING HOURS, WHICHEVER COMES FIRST. EXPOSURE TO EXCESSIVE HEAT OR OZONE WILL CAUSE O-RINGS TO AGE PREMATURELY. HELMETS AND MASKS WHICH ARE USED IN HEAVY OIL ENVIRONMENTS, JETTING OPERATIONS, OR CAUSTIC CHEMICALS WILL REQUIRE MORE FREQUENT O-RING REPLACEMENTS. ALL OTHER RESPIRING PARTS WHICH ARE SUBJECT TO THE SAME CONDITIONS WILL REQUIRE MORE FREQUENT REPLACEMENT.

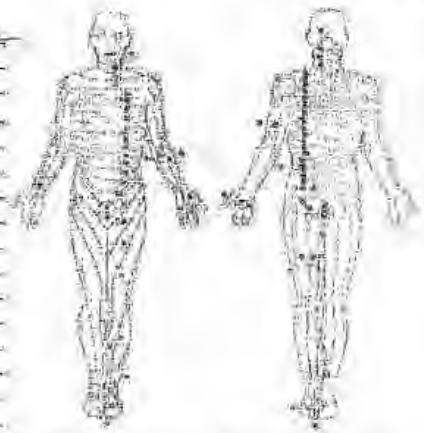
Technician Signature: Dave Schneider **Date** 1/24/2021

UNDERWATER SPORTS
YEAR OF VISUAL
MONTH OF VISUAL
TUMBLED
BURST DISC
VALVE O/H
Argon
Air
2021 Bailout
FEB Visual
Sales Person: 34 Jaiden
Sub-Total: 27.00
Total: 29.73
Visa 02/05/21: 29.73
Total Paid: 29.73
Amount Due: 0.00
Become our Facebook Fan
Underwater Sports Federal Way

Schneider	5/14/2021	1958	Outfall Inspection	City of Everett / Outfall	19	Diver	:	N
Schneider	1/21/2021	2201	Burn of king piles 86.3, 87.1, 67.2	Orion / T-5	61	Diver	:117	N
Schneider	1/22/2021	2201	Burned piles 67.1 & 67.3	Orion / T-5	64	Diver	:119	N
Schneider	1/20/2021	2201	Dive to burn king piles at elevation 66.6	Orion / T-5	61	Diver	:120	N
Schneider	1/15/2021	2201	Diver assisted with pulling piles	Orion / T-5	37	Diver	:125	N
Schneider	1/2/2021	2201	Assisted Orion with Mile ups	Orion / T-5	63	Diver	:177	N
Schneider	1/16/2021	2201	Diver marked and started cutting piles	Orion / T-5	57	Diver	:209	N
Schneider	1/14/2021	2201	Assisted the client with spile survey	Orion / T-5	48	Diver	:228	N
Schneider	12/28/2020	2201	Helping to Align H Piles & Sloet pilars	Orion / T-5	59	Diver	:267	N
Schneider	1/2/2021	2379	Wheel Job	Brusco Tug	10	Diver	:35	N
Schneider	1/19/2021	2201	Marked and burned King pile	Orion / T-5	56	Diver	:98	N
Schneider	1/25/2021	2201	Burn & Remove monster pile	Orion / T-5	60	Diver	:124	N
Schneider	1/12/2021	2201	Freshed King Pile	Orion / T-5	63	Diver	:40	N
Schneider	1/26/2021	2201	Burn Monster pile	Orion / T-5	60	Diver	:42	N
Schneider	1/13/2021	2201	Inspection /Burning / Webbing	Orion / T-5		Diver		N
Schneider	8/4/2020	2114	Pressure wash piles / prep to wrap	PPM Coleman Docks / Pier 62	Air / 8'	Diver	:302	N
Schneider	8/14/2020	2312	Dive to weld out final 25% of pile #4	P66 - Anodes	Air / 30'	Diver	:245	N
Schneider	9/3/2020	2231	Survey bottom to locate debris	Schnitzer Steel / Bottom Survey	Air /49'	Diver	:664	N
Schneider	9/8/2020	2330	Mag Partical test existing welds, weld patch onto pit area, test new weld area w mag	P66 / 250X 25 Fire Tank	Air / 40'	Diver	:318	N
Schneider	11/13/2020	2255	Dive to rig and relocate construction boom secition	Ceccanti-Boing / Boom Relocation	Air / 15'	Diver	:515	N
Schneider	11/17/2020	2255	019 Outfall inspection, T-70 West Ramp pile sweep inspection	Boeing/ Ceccanti	Air / 15'	Diver	:190	N
Schneider	11/20/2020	2255	Outfall Survey, T-21 / T-71 / Short Well spectional	Boeing/ Ceccanti	Air / 06'	Diver	:411	N
Schneider	12/14/2020	2255	burn pipe burn pipe collar and red heads on vault /Surveyed and cleaned ramp	Ceccanti /Boeing	Air /6'	Diver	:119	N
Schneider	8/12/2020	2312	Place anodes on plumb pile 1 75% weld out on pile 2	P66 - Anodes	Air / 33'	Diver	:211	N
Schneider	8/13/2020	2312	pleace 1ea anode on plumb pile #7 place 2 each anodes on plumb pile #5	P66 - Anodes	Air / 32'	Diver	:267	N
Schneider	8/17/2020	2201	Inspect Pile	Orion / T-5	Air / 36'	Diver	:203	N
Schneider	11/25/2020	2255	Chip concrete at outfall vault, slide sand collar into void	Boeing / Ceccanti	Air / 13'	Diver	:572	N
Schneider	12/2/2020	2201	20 bents weres swept, vibro hammer, 5 timber piles, inspect sheetpile	Orion / T-5	Air /51'	Diver	:310	N
Schneider	12/4/2020	2255	Pluged pipe with grout,	Ceccanti - Boing	Air /50'	Diver	:600	N
Schneider	12/28/2020	2201	Helping to Align H Piles & Sloet pilars	Orion / T-5	Air	59	Diver	:267
Schneider	8/5/2020	2114	Pressure wash pilings, prep for denso wrap	PPM Coleman Docks/Pier 62	Air / 8'	Diver	:347	N
Schneider	8/7/2020	2114	Denso wrap piles 50% overlap then installing pile jackets	PPM Coleman Docks	Air / 20'	Diver	:310	N
Schneider	1/9/2020	2177	Burn holes in plate and install bulk head rail	Garney Mudd Mountain	Air/10'	Diver	:97	N

Schneider	1/10/2020	2177	Exra burn steel plate and guid drill inwater holes	Garney Mudd Mountain	Air/10'	Diver	:27	N
Schneider	1/15/2020	2177	Forming around plate and bulkhead work	Garney Mudd Mountain	Air/10'	Diver	:223	N
Schneider	1/20/2020	2177	Fit steel plate to bulk head gate	Garney Mudd Mountain	Air/10'	Diver	:159	N
Schneider	1/28/2020	2177	Remove Forms	Garney Mudd Mountain	Air/10'	Diver	:221	N
Schneider	1/29/2020	2114	Removal 12 timber piles, 3 steel piles, survey bottom	PPM Coleman	Air/45'	Diver	:60	N
Schneider	2/11/2020	2272	Prepped piles for anode placement	Port of Newport NOAA Anodes	Air/31'	Diver	:228	N
Schneider	2/12/2020	2272	Prepped piles for anode placement	Port of Newport NOAA Anodes	Air/24'	Diver	:350	N
Schneider	2/14/2020	2272	Completed Install of cable for new anodes	Port of Newport NOAA Anodes	Air/24'	Diver	:230	N
Schneider	2/26/2020	2114	Dive to burn/cut piles and remove debris	PPM Coleman	Air/35'	Diver	:136	N
Schneider	2/27/2020	2114	Dive to burn/cut piles and remove debris	PPM Coleman	Air/36'	Diver	:170	N

Association of Diving Contractors International
ADCI
Cert. # (b) (6)
Expires 12/07/2025
SURFACE-SUPPLIED AIR DIVING SUPERVISOR
KRIS HUNTER I.D. (b) (6)
Commercial Diver Certification Card



(b) (6)

R.W. Hunter

2117 Union Ave.

North Bend OR 97459

541-808-9000

6/19/20

Hazmat/ OSHA – Will have by Monday 06/14/2021

HEARTSAVER

Heartsaver®
First Aid CPR AED

American Heart Association.

Kris Hunter

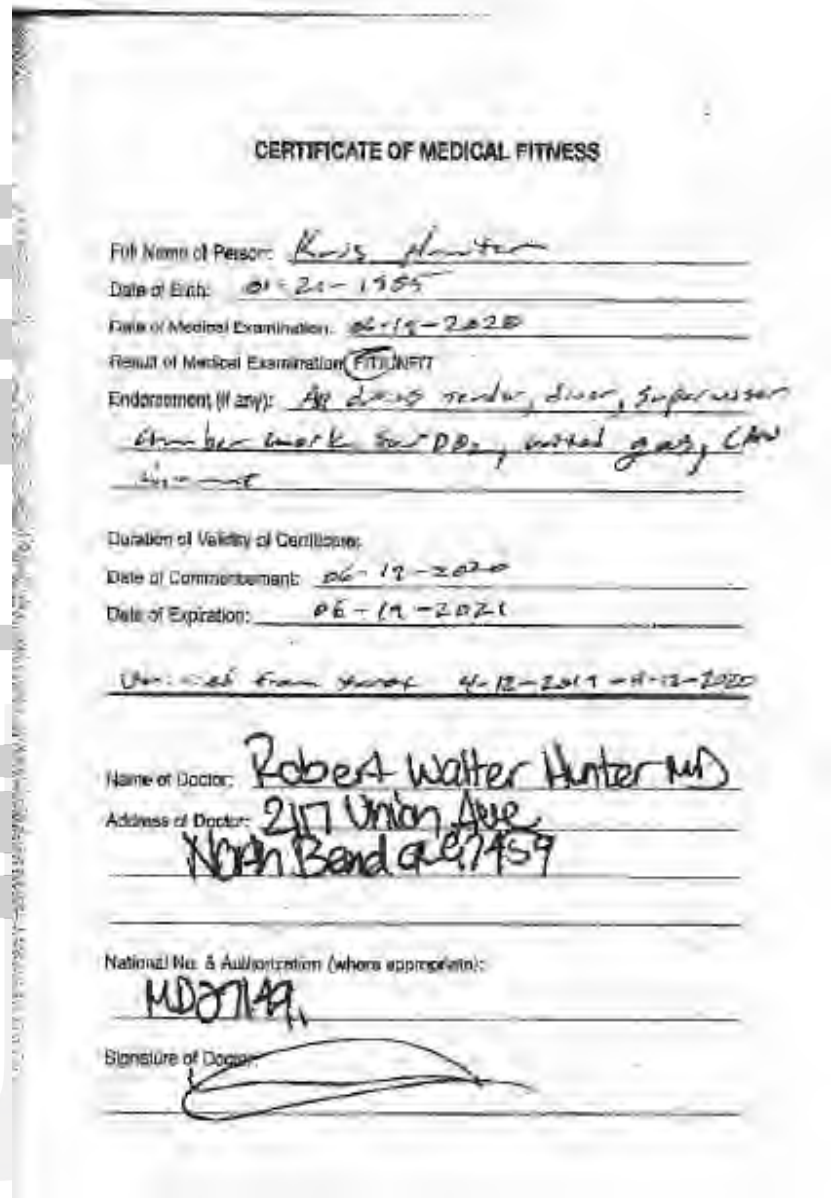
The above individual has successfully completed the cognitive and skills evaluations in accordance with the curriculum of the American Heart Association Heartsaver First Aid CPR AED Program.

Optional modules completed:

Issue Date	Recommended Renewal Date
12/19/2019	12/2021

HEARTSAVER

Training Center Name	EMT Associates
Training Center ID	OR20773
TC Address	2275 Rocky Lane Eugene OR 97401 USA
TC Phone	(541) 430-7149
Instructor Name	Brian Conley
Instructor ID	(b) (6)



CERTIFICATE OF MEDICAL FITNESS

Full Name of Person: Kris Hunter

Date of Birth: 01-21-1985

Date of Medical Examination: 06-19-2020

Result of Medical Examination: FIT/UNFIT

Endorsement (if any): All dives tender, diver, supervisor chamber work for DO_2, mixed gas, CAN [illegible]

Duration of Validity of Certificate:

Date of Commencement: 06-19-2020

Date of Expiration: 06-19-2021

Unrestricted from [illegible] 4-12-2019 - 4-12-2020

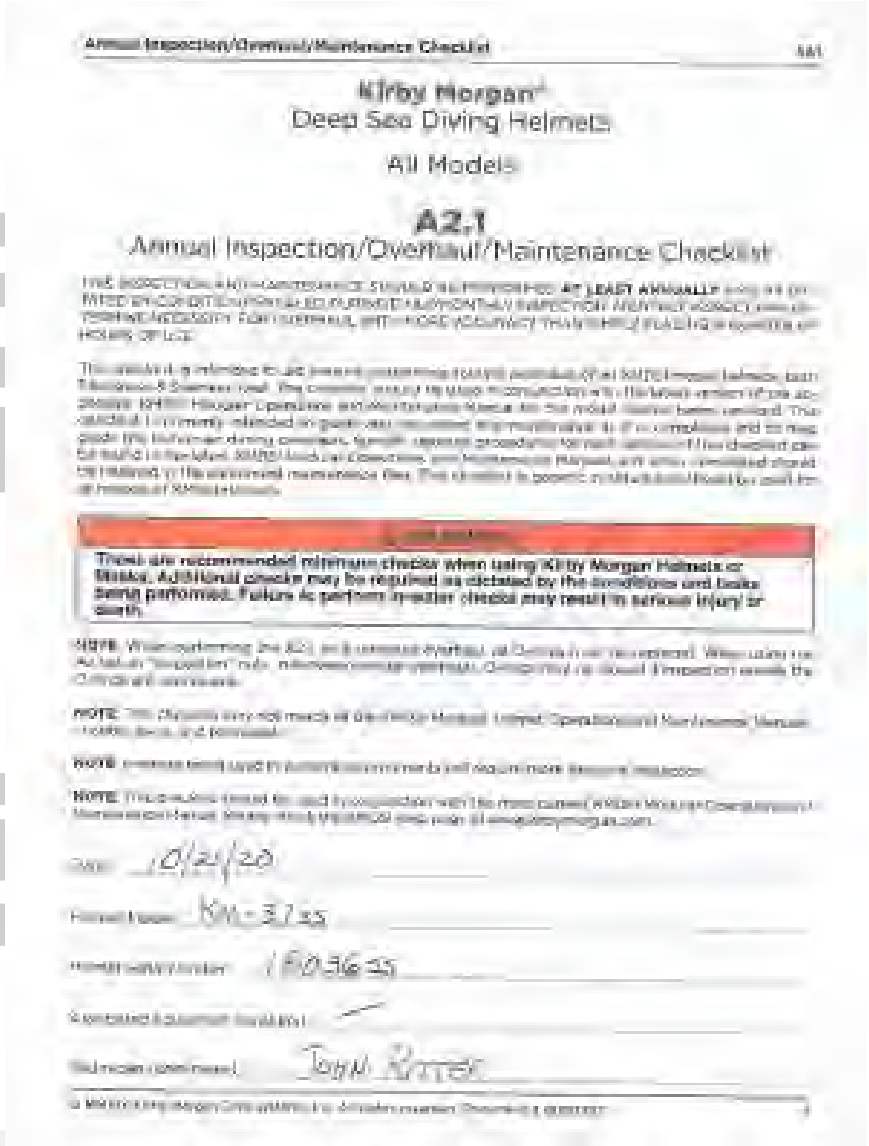
Name of Doctor: Robert Walter Hunter MD

Address of Doctor: 2117 Union Ave
North Bend OR 97459

National No. & Authorization (where appropriate):
MD27149

Signature of Doctor:

Dive Commercial International, Inc.
AUS
SERVICE
N/A
Repair Estimate/Authorization Sheet



Annual Inspection/Overhaul/Maintenance Checklist

Kirby Morgan®

Deep Sea Diving Helmets

All Models

A2.1

Annual Inspection/Overhaul/Maintenance Checklist

THE INSPECTION AND MAINTENANCE SHOULD BE PERFORMED **AT LEAST ANNUALLY** AND AT OR [illegible] RATED SATURATION OR SURFACE-SUPPLIED AIR/NITROX DIVING EQUIPMENT. MORE FREQUENT INSPECTION AND/OR MAINTENANCE MAY BE NECESSARY FOR OVERHAUL, BUT NOT LESS FREQUENTLY THAN EVERY [illegible] HOURS OF USE.

This checklist is a reference to aid personnel performing routine annual (Part 56/70) inspection, both Manufacturer & Owner/User. The checklist is only to be used in conjunction with the latest version of the applicable Kirby Morgan Operations and Maintenance Manual for the model helmet serviced. This checklist is currently intended for participants completing maintenance to their own helmets and to help guide the helmet owner during overhaul, specific required procedures for each helmet/model should be referred to the latest Kirby Morgan Service Bulletins, and Maintenance Manuals and when completed should be retained in the equipment maintenance files. This checklist is generic in nature and should not be used for all models of Kirby Morgan helmets.

There are recommended minimum checks when using Kirby Morgan Helmets or Masks. Additional checks may be required as dictated by the conditions and tasks being performed. Failure to perform in-water checks may result in serious injury or death.

NOTE: While performing the ADI, any [illegible] shall be recorded. When using the Actuation "Inspection" note, [illegible] Completing the Annual Inspection avoids the Overhaul requirements.

NOTE: This checklist may not apply to [illegible] Helmet Operational Manual/Owners Manual. Refer to [illegible] and procedures.

NOTE: [illegible] used in current recommended maintenance requirements.

NOTE: This checklist should be used in conjunction with the most current Kirby Morgan Operations & Maintenance Manual. Kirby Morgan [illegible] are [illegible] at www.kirbymorgan.com.

Date: 10/21/20

Helmet Model: KM-37 SS

Helmet Serial Number: 10036 SS

Completed Assembly Installed: ✓

Technician (print name): John Ritter

© KIRBY MORGAN DIVE SYSTEMS, INC. All rights reserved. Document 2 [illegible]

Annual Inspection/Overhaul/Maintenance Checklist A21

Procedures	Initials
(1) Perform a leak check of all EGS components and fittings using water in a pressurized condition. Repair/replace them as necessary.	N/A
(2) Inspect the [illegible] assembly for signs of wear and/or damage. Repair/replace as necessary.	N/A

Recorded in [illegible] for [illegible] and EGS System [illegible] is [illegible]? ☐ Yes ☒ No

Recorded [illegible] for [illegible] ☐ Yes ☒ No

I JOHN RITTER hereby certify that I have performed the work required in this A21 and I AM a certified KMDSI / Dive Lab technician.

Print name: JOHN RITTER

Signature: John Ritter Date: 10/21/20

ID #: DI-12156 Dessert Certification: 6/14/16

I _____ hereby certify that I have performed the work required in this A21 and **I AM NOT** a certified KMDSI / Dive Lab technician.

Technician/Owner Print Name: _____

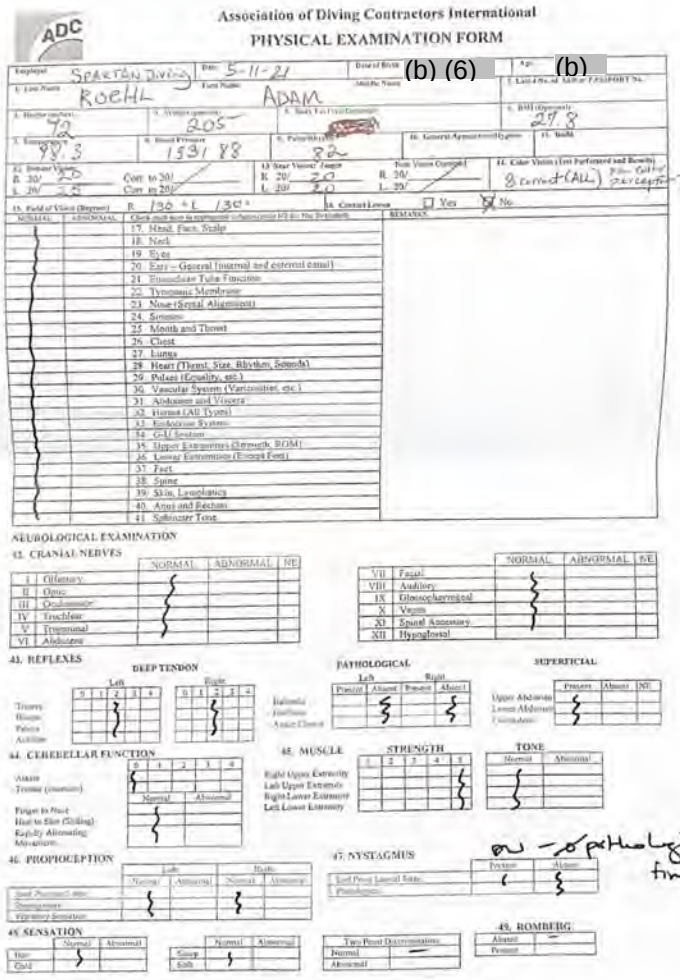
Signature: _____ Date: _____

© [illegible] Kirby Hansen Dive Systems, Inc. All rights reserved. Document # [illegible] TT

VISUAL INSPECTION
CERTIFICATE
JAN
FEB
MAR
APR
MAY
JUN
JUL
AUG
SEP
OCT
NOV
THIS CYLINDER HAS BEEN
VISUALLY INSPECTED TO OSHA
PSI/PCI AND CGA STANDARDS
THIS CYLINDER HAS PASSED
A VISUAL INSPECTION
ON THE DATE WHICH IS PUNCHED
2018
2019
2021
V.I. PLUS

Hunte	2/8/2021	2201	Dive and burn, remove king piles (78.1)	Orion / T-5	61	Diver	:114	N
Hunter	1/25/2021	2201	Burning on 70.1/ moved to 68.3 due to overhead	Orion / T-5	60	Diver	:68	N
Hunter	1/26/2021	2201	Burn 70.1 pulled out /location 62.3 & 63.3	Orion / T-5	59	Diver	:89	N
Hunter	1/27/2021	2114	26 timeer piles were cut -100 ft of piping rem	Colman Dock / PPM	20	Diver	:75	N
Hunter	2/2/2021	2045	D/D to Inspect Stop Log Slots	Wells Dam/Douglass County	64	Diver	:111	N
Hunter	2/9/2021	2201	Burned off King pile 74.2and started 74.4	Orion / T-5	59	Diver	:135	N
Hunter	2/10/2021	2201	Burnt off king piles 74.3,75.1,77.1	Orion / T-5	61	Diver	:147	N
Hunter	4/1/2021	2201	Pile Jacket Installation	Orion / T-5	15	Diver	:180	N
Hunter	4/14/2021	2170	Fix Buoy	King County Bouy	36	Diver	:131	N
Hunter	4/15/2021	2415	Install whalers on bulkhead replace mont	Quigg / Foss Lrhead	18	Diver	:194	N
Hunter	4/17/2021	2415	Install Walers on new bulkhead	Quigg / Foss Lrhead	21	Diver	:236	N
Hunter	4/19/2021	2415	Install Walers on new Bulk Head	Quigg /Foss Bulk Head Repair	21	Diver	:239	N
Hunter	4/20/2021	2415	Install Walers on new Bulk Head	Quigg /Foss Bulk Head Repair	21	Diver	:282	N
Hunter	4/22/2021	2415	Install Walers on new Bulk Head	Quigg /Foss Bulk Head Repair	21	Diver	:238	N
Hunter	4/23/2021	2415	Install Walers on new Bulk Head	Quigg /Foss Bulk Head Repair	21	Diver	:216	N
Hunter	4/26/2021	2415	Install walers on weld spacer plate, new bulk	Quigg Bros / Foss Bulkhead	21	Diver	:239	N
Hunter	4/27/2021	2415	Install spacer plates on Foss bulkhead	Quigg Bros / Foss Bulkhead	21	Diver	:231	N
Hunter	4/28/2021	2415	Install spacer plates on Foss bulkhead	Quigg Bros / Foss Bulkhead	21	Diver	:118	N
Hunter	5/5/2021	2431	Clean underside of dock, video inspection	Squamish Tribe / Dock Cleaning	6	Diver	:200	N
Hunter	5/13/2021	2063	ROV inspection & Trash rack install	Seattle City Light / Tolt Reservoir	132	Diver	:11	N

Association of Diving Contractors
International
Cert. # (b) (6)
Expires 10/10/2021
ADCI
INTERNATIONAL ENDORSEMENT
ENTRY LEVEL TENDER/DIVER
ADAM K. ROEHL I.D. (b) (6)
Commercial Diver Certification Card



Association of Diving Contractors International

PHYSICAL EXAMINATION FORM

Employer: SPARTAN DIVING | Date: 5-11-21 | Date of Birth: (b) (6) | Age: (b)

1. Last Name: ROEHL | First Name: ADAM | Middle Name:

2. Height (inches): 72 | 3. Weight (pounds): 205 | 4. Social Security Number: [redacted] | 5. BMI (Optional): 27.8

7. Temperature: 98.3 | 8. Blood Pressure: 153/88 | 9. Pulse Rate: 82 | 10. General Appearance/Hygiene: | 11. Build:

12. Distant Vision: R. 20/ 20 L. 20/ 25 | Corr. to 20/ | Corr. to 20/ | 13. Near Vision/ Jaeger: R. 20/ 20 L. 20/ 20 | Near Vision Corrected: R. 20/ L. 20/ | 14. Color Vision (Test Performed and Results): Ishihara Testing: 8 correct (ALL) EXCEPT 5 (5 is the 2nd digit)

15. Field of Vision (Degrees): R. 130 + L. 130 = | 16. Contact Lenses: ☐ Yes ☒ No

NORMAL	ABNORMAL	Check each item to be examined (check boxes for N/E in the Remarks)	REMARKS
		17. Head, Face, Scalp	
		18. Neck	
		19. Eyes	
		20. Ears – General (internal and external canal)	
		21. Eustachian Tube Function	
		22. Tympanic Membrane	
		23. Nose (Septal Alignment)	
		24. Sinuses	
		25. Mouth and Throat	
		26. Chest	
		27. Lungs	
		28. Heart (Thrmt, Size, Rhythm, Sounds)	
		29. Pulses (Equality, etc.)	
		30. Vascular System (Varicosities, etc.)	
		31. Abdomen and Viscera	
		32. Hernia (All Types)	
		33. Endocrine System	
		34. GU System	
		35. Upper Extremities (Strength, ROM)	
		36. Lower Extremities (Except Feet)	
		37. Feet	
		38. Spine	
		39. Skin, Lymphatics	
		40. Anus and Rectum	
		41. Sphincter Tone	

NEUROLOGICAL EXAMINATION

42. CRANIAL NERVES

		NORMAL	ABNORMAL	N/E
I	Olfactory			
II	Optic			
III	Oculomotor			
IV	Trochlear			
V	Trigeminal			
VI	Abducens			
VII	Facial			
VIII	Auditory			
IX	Glossopharyngeal			
X	Vagus			
XI	Spinal Accessory			
XII	Hypoglossal			

43. REFLEXES

DEEP TENDON

	Left					Right				
	0	1	2	3	4	0	1	2	3	4
Triceps										
Biceps										
Patella										
Achilles										

PATHOLOGICAL

	Left		Right	
	Present	Absent	Present	Absent
Babinski				
Hoffman				
Ankle Clonus				

SUPERFICIAL

	Present	Absent	N/E
Upper Abdominal			
Lower Abdominal			
Cremasteric			

44. CEREBELLAR FUNCTION

	0	1	2	3	4
Ataxia					
Tremor (nystagmus)					

	Normal	Abnormal
Finger to Nose		
Heel to Shin (Gliding)		
Rapidly Alternating Movements		

45. MUSCLE STRENGTH

	1	2	3	4	5
Right Upper Extremity					
Left Upper Extremity					
Right Lower Extremity					
Left Lower Extremity					

MUSCLE TONE

Normal	Abnormal

46. PROPIOCEPTION

	Left		Right	
	Normal	Abnormal	Normal	Abnormal
Joint Position Sense				
Stereognosis				
Vibratory Sensation				

47. NYSTAGMUS

	Present	Absent
End Point Lateral Gaze		
Pendular		

ON – 2 pathological findings

48. SENSATION

	Normal	Abnormal
Hot		
Cold		

	Normal	Abnormal
Sharp		
Soft		

Two Point Discrimination	
Normal	—
Abnormal	

49. ROMBERG

Absent	—
Present	

15

50. MISCELLANEOUS REMARKS

Unremarkable
Exam

LABORATORY FINDINGS

51. Urinalysis
Color: Yellow
Appearance: Clear
Sp. Gravity: 1.025
Ph: 6.0
Microscopic: Normal ☐ Abnormal(s) ☐ (See report)

	0	1+	2+	3+	4+
Sugar	✓				
Blood	✓				
Ketones	✓				
Bilirubin					
Protein					

52. Blood Tests — Attach Reports
CBC: Normal ☐ Abnormal ☐
RPR: ☐ Pos ☐ Neg
Sickle Cell: ☐ Pos ☐ Neg

53. Cardiac Risk Score
No. of Points
10 year risk

54. Pulmonary Function
FVC
FEV1
FEV1/FVC

55. X-ray/MRI — Normal / Abnormal / (Describe)
Chest: ☑ ☐
Lumbar Spine: ☐ ☐
Long Bones: ☐ ☐
MRI: ☐ ☐

56. Electrocardiogram
Static
Exercise Stress

57. Audiogram

	Hz	500	1000	2000	3000	4000	6000	8000
Left		10a	0a	5a	0a	0a	20a	50
Right		10a	0a	0a	0a	5a	15a	0a

58. Comprehensive Metabolic Panel — Attach Report
Normal ☐ Abnormal ☐

Lipid Panel (if done)
Normal ☐ Abnormal ☐

Comments:

59. Drug Screen
☒ Not collected
☐ Collected, results sent to employer

Work Status:
☑ Fit for diving
☐ Cleared for supervisor
☐ Cleared for topside work only
☐ Cleared with restrictions:
☐ Further evaluation needed
☐ Unfit for diving
☐ Unfit

Comments:

Examinee Name
Physician Signature (b) (6)
Physician Name (b) (6)
Address
Phone Number
Date of Examination

Urgent Care
Galveston, Texas 77551
P:(409)744-9800 F:(409)744-8844

Revised 2015

Hazmat / OSHA

Divers Alert Network

Certificate of Training

Adam Roehl

has successfully completed

Diving First Aid for the Professional Diver Provider (CPR:HCP)
First Aid and CPR (Blended) (DANDAN-875)

Approved by the United States Coast Guard to meet the training requirements of 46 CFR 11.302(a)(3), 46 CFR 12.602(a)(3), 46 CFR 11.20(i)(1) and STCW Code Table A-VI/1-3

April 23, 2019
Date

DIT 103-19 DFA Pro
Location

Steve Coleman
Instructor

Director of Training

CERTIFICATE OF DIVER MEDIC QUALIFICATIONS

Recognized by

The International Marine Contractors Association (IMCA)

ISSUED BY: DIVERS INSTITUTE OF TECHNOLOGY
DIVER MEDIC TECHNICIAN COURSE
1341 N. NORTHLAKE WAY, STE. 150
SEATTLE, WA 98103
USA

CERTIFICATE NUMBER: (b) (6)

DATE OF ISSUE: 28 MAY 2021

ISSUED TO: Adam Roehl

DATE OF BIRTH: (b) (6)

OFFSHORE DIVING CERTIFICATE ISSUER & #: (b) (6)

DATE ON WHICH DMT CERTIFICATE CEASES TO BE VALID UNLESS FURTHER TRAINING IS UNDERTAKEN:

28 MAY 2023

CERTIFICATE HOLDER PHOTOGRAPH

SEAL OF ISSUING BODY:

DIVERS INSTITUTE OF TECHNOLOGY
OFFICIAL DOCUMENT

SIGNED:
NAME: BRADLEY W PETERSON
DIVER MEDIC COURSE DIRECTOR

SIGNED:
NAME: DANIEL MATTHES
DIRECTOR OF TRAINING

KIRBY MORGAN®

1430 Jason Way
Santa Maria, CA 93455 USA
www.kirbymorgan.com

KM97 SN: (b) (6)

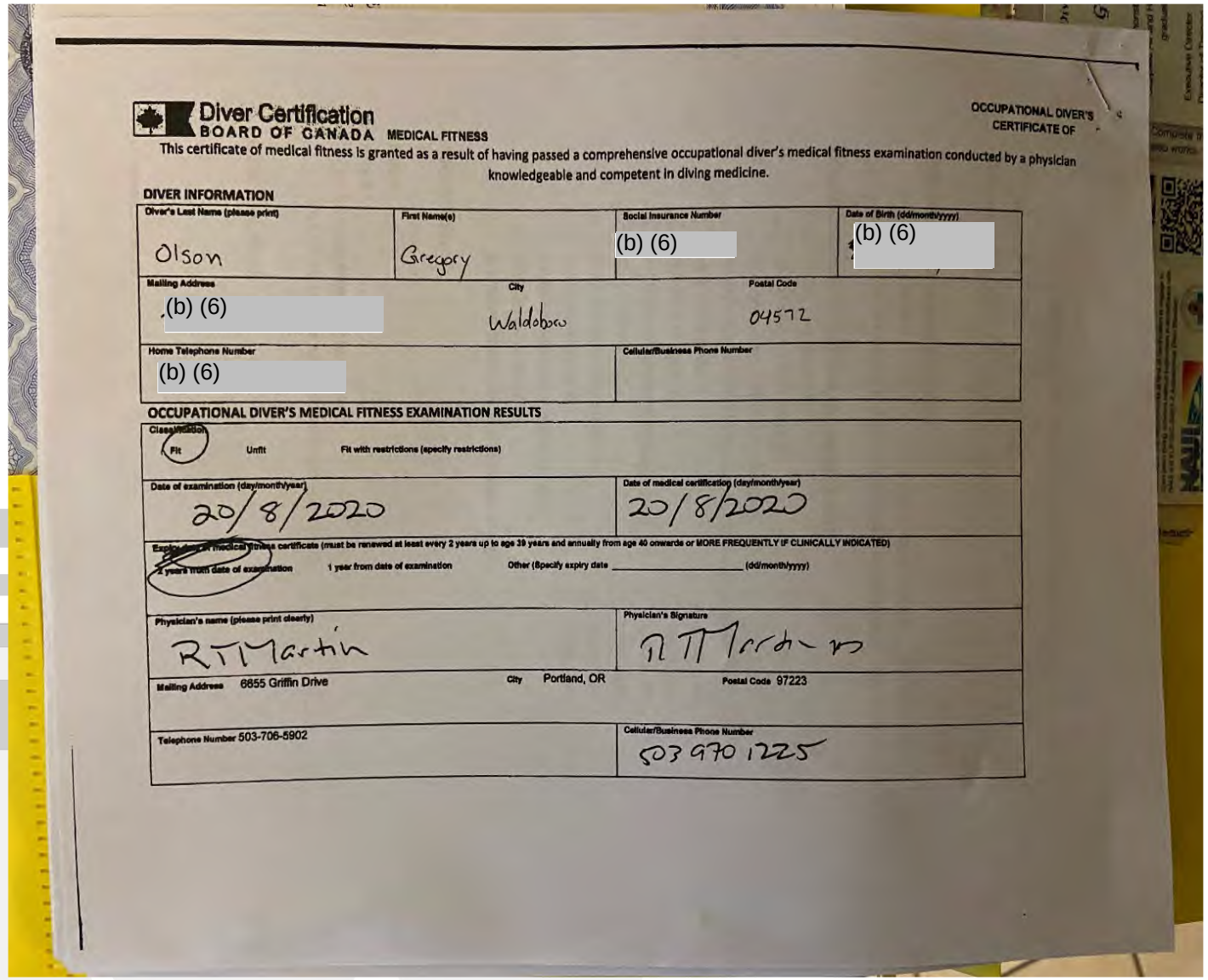
Inspected/Verified By: _____

Date: 5-6-21

J F M A J J A S O N D
EQUIPMENT INSPECTION
Performed on Date Punched
DECA DIVING
Santa Maria, California • USA • Phone (805) 928-4500
2017 2018 2019 2020 2022 2023 2024
DSI®
CE
WARNING

Dive Logs

Association of Diving Contractors International
Cert. # (b) (6)
ADCI
INTERNATIONAL ENDORSEMENT
GREGORY B. OLSON I.D. (b) (6)



Diver Certification
BOARD OF CANADA MEDICAL FITNESS

OCCUPATIONAL DIVER'S CERTIFICATE OF

This certificate of medical fitness is granted as a result of having passed a comprehensive occupational diver's medical fitness examination conducted by a physician knowledgeable and competent in diving medicine.

DIVER INFORMATION

Diver's Last Name (please print)	First Name(s)	Social Insurance Number	Date of Birth (dd/month/yyyy)
Olson	Gregory	(b) (6)	(b) (6)

Mailing Address	City	Postal Code
(b) (6)	Waldoboro	04572

Home Telephone Number	Cellular/Business Phone Number
(b) (6)	

OCCUPATIONAL DIVER'S MEDICAL FITNESS EXAMINATION RESULTS

Classification: (Fit) Unfit Fit with restrictions (specify restrictions)

Date of examination (day/month/year)	Date of medical certification (day/month/year)
20/8/2020	20/8/2020

Expiry date of medical fitness certificate (must be renewed at least every 2 years up to age 39 years and annually from age 40 onwards or MORE FREQUENTLY IF CLINICALLY INDICATED)
(2 years from date of examination) 1 year from date of examination Other (Specify expiry date _____ (dd/month/yyyy)

Physician's name (please print clearly)	Physician's Signature
RT Martin	RT Martin MD

Mailing Address	City	Postal Code
6855 Griffin Drive	Portland, OR	97223

Telephone Number	Cellular/Business Phone Number
503-706-5902	503 970 1225

This is to attest that

Gregory Olson

Has satisfactorily completed 40 Hours of Training in Health & Safety at Hazardous Waste Sites in compliance with Federal Occupational Safety & Health Administration (OSHA) 29 CFR 1910.120, and has successfully met the 80 hours Training requirement for Washington Administrative Code (WAC) 296-62-3040 Including 20 hours Contaminated Water Diving Procedures.

Presented by: DIVERS INSTITUTE OF TECHNOLOGY, Seattle, WA

Given this 11th day of March, 2021

Britt Coates
Instructor

Daniel Matthes
Director of Training

John Paul Johnston
Executive Director

ACCSC

Divers Alert Network

Certificate of Training

Gregory Olson

has successfully completed

Diving First Aid for the Professional Diver Provider (CPR:HCP)
First Aid and CPR (Blended) (DANDAN-875)

Approved by the United States Coast Guard to meet the training requirements of 46 CFR 11.302(a)(3), 46 CFR 12.602(a)(3), 46 CFR 11.20(b)(1) and STCW Code Table A-VI/1-3

Gordon K. Smith
Diver

Lord Cutler
Instructor

Lord Cutler
Instructor

Director of Training

Hat
Bailout
Dive logs

Association of Diving Contractors International

Cert. # (b) (6)

Expires 03/24/2026

ADCI

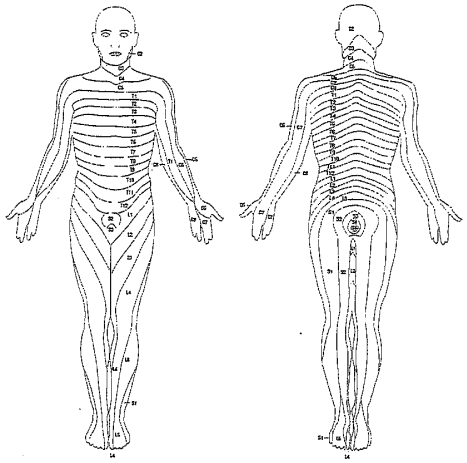
SURFACE-SUPPLIED AIR DIVER

VINCENT O'NEAL I.D. (b) (6)

Commercial Diver Certification Card

51. MISCELLANEOUS REMARKS

NORMAL EXAM



LABORATORY FINDINGS

52. Urinalysis

Color	Amber
Appearance	Clear
Sp. Gravity	1.010
Ph	5.0

	0	1+	2+	3+	4+
Sugar	/				
Blood			/		
Ketones	//				
Bilirubin	//				
Protein	//				

53. Blood Tests — Attach Reports

CBC: Normal ☒ Abnormal ☐ — RPR: ☐ Pos ☐ Neg

Sickle Cell: ☐ Pos ☐ Neg — HIV: ☐ Pos ☐ Neg

54. Pulmonary Function

FVC	NOT DONE
FEV1	
FEV1/FVC	2° TO COVID

55. X-rays

	Normal	Abnormal
Chest	☐	☐
Lumbar Spine	☐	☐
Long Bone Series	☐	☐
Other	☐	☐

(Describe)

56. Electrocardiogram

Static	N/A
Exercise Stress	

57. Audiogram

Hz	500	1000	2000	3000	4000	6000	8000
Left	0	0	0	5	0	0	0
Right	0	0	0	0	0	0	0

58. Comprehensive Metabolic Panel — Attach Report

Normal ☒ Abnormal ☐

Lipid Panel (if done)

Normal ☐ Abnormal ☐

Comments:

59. Drug Screen

☐ Not collected
☐ Collected, results sent to employer

Work Status:

- ☒ Fit for diving
- ☐ Cleared for supervisor
- ☐ Cleared for topside work only
- ☐ Cleared with restrictions:
- ☐ Further evaluation needed:
- ☐ Unfit for diving :
- ☐ Unfit

Comments:

Examinee Signature: (b) (6)

Examinee Name: (b) (6)

Physician Signature: James Harris MD

Physician Name:

Address: James Harris, MD, LLC
9370 SW Greenburg Rd, #101
Portland, OR 97223

Phone Number: (503) 246-7030

Date of Examination: 5/3/2021.

This is to attest that

Vincent L. O'Neal

[illegible]

Presented by DIVERS INSTITUTE OF TECHNOLOGY, [illegible]

[illegible] 26th day of March, 2019

[illegible] | [illegible] | [illegible]

ACCSC

Certificate of Completion

360training

This Certifies That

[redacted]

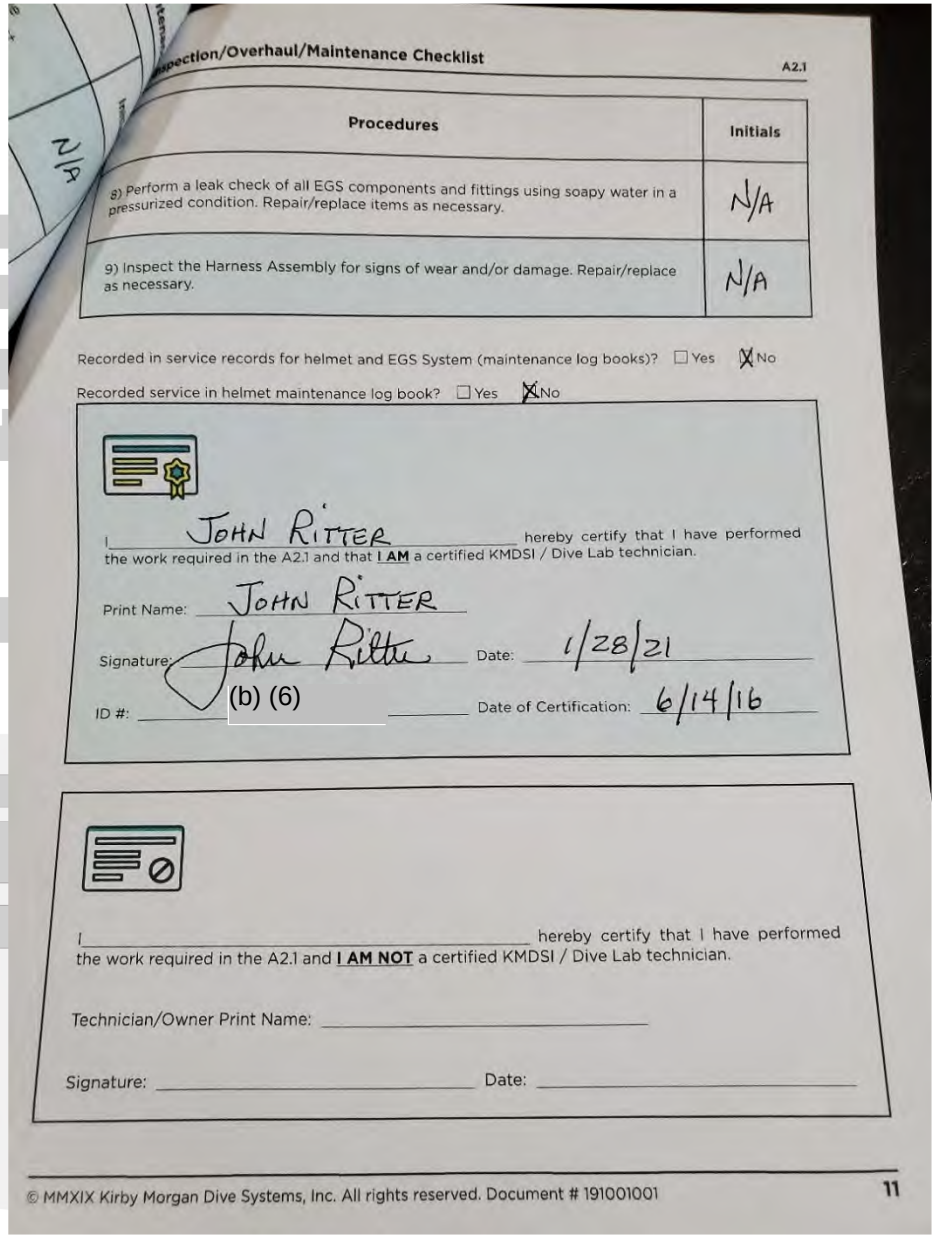
is awarded this certificate for

OSHA 10 Hour Outreach Training Program - Construction

Credit Hours: 10.00

Completion Date: 10/24/2019

First Aid Ed.
509-628-4826
www.firstaided.com
(b) (6)
Certification demonstrates proficiency.
First Aid Ed.
- CPR Adult/Child/Infant
- AED
- Oxygen Provider
- Near Drowning
(b) (6)
ISSUE DATE
STUDENT SIGNATURE



Inspection/Overhaul/Maintenance Checklist A2.1

Procedures	Initials
8) Perform a leak check of all EGS components and fittings using soapy water in a pressurized condition. Repair/replace items as necessary.	N/A
9) Inspect the Harness Assembly for signs of wear and/or damage. Repair/replace as necessary.	N/A

Recorded in service records for helmet and EGS System (maintenance log books)? ☐ Yes ☒ No

Recorded service in helmet maintenance log book? ☐ Yes ☒ No

I JOHN RITTER hereby certify that I have performed the work required in the A2.1 and that **I AM** a certified KMDSI / Dive Lab technician.

Print Name: JOHN RITTER

Signature: John Ritter Date: 1/28/21

ID #: (b) (6) Date of Certification: 6/14/16

I _____ hereby certify that I have performed the work required in the A2.1 and **I AM NOT** a certified KMDSI / Dive Lab technician.

Technician/Owner Print Name: _____

Signature: _____ Date: _____

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Bailout

O'Neal	1/5/2021	2218	Dry Dock -Elizabeth	Foss / Haul out	33	Diver	:85	N
O'Neal	1/12/2021	2218	Assist with haul out logs	Foss / Haul out	15	Diver	:75	N
O'Neal	1/16/2021	2201	Diver marked and started cutting piles	Orion / T-5	60	Diver	:107	N
O'Neal	1/20/2021	2201	Burned on 87.2 & 87.4	Orion / T-5	55	Diver	:141	N
O'Neal	1/22/2021	2201	King pile burning-70.1 and 70.4	Orion / T-5	58	Diver	:25	N
O'Neal	1/25/2021	2351	Dove on N & S structure /removed debris survey	Orion / Pier 59	45	Diver	:50	N
O'Neal	2/2/2021	2218	drydock 2 host tugs	Foss /	16	Diver	:118	N
O'Neal	2/9/2021	2218	Dry Dock -PO 4500287337	Foss / Arctic Bear	15	Diver	:39	N
O'Neal	2/10/2021	2351	Survey /remove debris from inspection foot	Pier 58 / Orion	20	Diver	:236	N
O'Neal	2/23/2021	2390	Dive to remove rocks and debris from Spillway	Bonneville Dam	62	Diver	:126	N
O'Neal	3/19/2021	2284	Remove caisson plug and replace supersacks	Slayden Consist	12	Diver	:361	N
O'Neal	3/16/2021	2409	Outfall Inspection	City of Granger/ Outfall	4	Diver	:30	N
O'Neal	3/24/2021	2322	Inspect Bridge, Pier, Pilings	Ceccanti / Sammamish	10	Diver	:167	N
O'Neal	4/8/2021	2170	Fix Buoy	King County Buoy	21	Diver	:21	N
O'Neal	4/13/2021	2218	Hou lout Hudson on Dry Dock #3	Foss / Haul out	15	Diver	:86	N
O'Neal	5/6/2021	2218	Haul out Northern Patriots	Foss / Haul out	15	Diver	:60	N
O'Neal	5/10/2021	2218	Dry Docking	Foss / Haul out	21	Diver	:67	N
O'Neal	5/11/2021	2255	Inspect Sheet fire wall T-95	Boeing	15	Diver	:85	N
O'Neal	0519/2021	2436		Kormarine / M/V Athos	37	Diver	:303	N
O'Neal	5/20/2021	2218	Dry Docking Boat Flicka	Foss / Drydock	20	Diver	:172	N

O'Neal	1/11/2020	2114	Recover sunken skiff	PPM Coleman Docks	Air/36'	Diver	:40	N
O'Neal	1/15/2020	2114	Cut timber piles, survey, and remove debris	PPM Coleman Docks	Air/32'	Diver	:210	N
O'Neal	1/16/2020	2114	Cut timber piles, survey, and remove debris	PPM Coleman Docks	Air/32'	Diver	:210	N
O'Neal	1/21/2020	2251	Swim nets in cells 14-16, 5, and diesel	Hanford		Diver	:442	N
O'Neal	1/24/2020	2268	Survey and remove debris	Coleman Dock	Air/45'	Diver	:86	N
O'Neal	2/13/2020	2275	Jamie AAA Inspection	Nichols Brothers Dry Dock	Air/8'	Diver	:50	N
O'Neal	2/27/2020	2270	Install 2 6K Lift Bags and 3 2K lift bags	DNR Lady Grace Salvage	Air/24'	Diver	:190	N
O'Neal	4/30/2020	2201	Install Bulkhead	Orion T5	Air/8'	Diver	:240	N
O'Neal	5/1/2020	2201	Install Bulkhead	Orion T5	Air/8'	Diver	:123	N
O'Neal	5/13/2020	1958	Inspect WPCF Sump Station and Intake	City of Everett WPCF	Air/38'	Diver	:115	N
O'Neal	6/2/2020	2218	Haul out Henry	Foss 4500276937	Air/21'	Diver	:90	N
O'Neal	6/5/2020	2302	Hull Cleaning	Motive Power	Air/15'	Diver	:200	N
O'neal	6/29/2020	2218	Haul out drydock Bountiful	Foss Bountiful	Air/20'	Diver	:92	N
O'Neal	7/8/2020	2218	Dry Dock 4500278724	Foss Enterprise	Air/20'	Diver	:180	N
O'neal	7/20/2020	2218	Set Tug into Dry Dock	Dry Dock / Foss	Air / 15'	Diver	:76	N
O'neal	8/3/2020	2218	Assist with Haul out	Foss/Pacific Titian	Air / 20'	Diver	:28	N

O'neal	8/5/2020	2323	Install plug in tank - Remove plug from tank	Oceanside Const/ Skagit WT	Air / 72'	Diver	:7	N
O'neal	8/10/2020	2341	Pile Wrap	PPM Hoffman / Pier 62	Air / 15'	Diver	:216	N
O'neal	8/11/2020	2341	Pile Wrap	PPM Hoffman / Pier 62	Air / 18'	Diver	:399	N
O'neal	8/18/2020	2114	Debris Cleanup / stub pile	PPM Hoffman / Colman Dock	Air / 15'	Diver	:390	N
O'neal	8/21/2020	2338	Inspect and epoxy repair water tank	Roslyn Clearwater / Crry Roslyn	Air / 32	Diver	:190	N
O'neal	9/2/2020	2255	Piling inspection for the central ramp area	Ceccanti / Central ramp inspection	Air / 10'	Diver	:280	N
O'neal	9/3/2020	2231	Survey bottom to locate debris	Schnitzer Steel / Bottom Survey	Air / 40'	Diver	:129	N
O'neal	9/4/2020	2231	Survey bottom to locate debris	Schnitzer Steel / Bottom Survey	Air / 41'	Diver	:192	N
O'neal	9/9/2020	2114	Stubbing Pile & Debris remover	PPM / Colman Dock	Air / 32'	Diver	:524	N
O'neal	9/10/2020	2114	Stubbing Pile & Debris Remover	PPM / Colman Dock	Air / 32'	Diver	:210	N
O'neal	9/11/2020	2114	Stubbing Pile & Debris Remover	PPM / Colman Dock	Air / 32'	Diver	:318	N
O'neal	9/15/2020	2114	Rig & out timer pilings, Rig & burn steel pilings	PPM / Colman Dock	Air /24'	Diver	:171	N
O'neal	9/16/2020	2063	MLPP inspection and cap removal	SPU / Chester Mores	Air / 30'	Diver	:277	N
O'neal	9/17/2020	2231	Inshore inspection - Bottom Sweep	Schnitzer Steel / General Metals	Air / 32'	Diver	:183	N
O'neal	9/18/2020	2114	Dive to burn and remove piers piles in Multi sections, piles floated out w lift bags	PPM / Colman Dock	Air / 25'	Diver	:295	N
O'neal	9/21/2020	2114	Cut and remove piles from under Colman dock	PPM / Colman Dock	Air / 37'	Diver	:363	N

O'neal	9/23/2020	2114	Burn Steel piles in 10-15 float on lift bag tow to shore, pull out water	PPM / Colman Dock	Air /33'	Diver	:413	N
O'neal	9/28/2020	2359	Inspect Pier, Piling and pile cap, begin debris removal	City of Kenmore / Park Insp	Air / 10'	Diver	:394	N
O'neal	10/1/2020	2231	Old dock bottom cleans up	Schnitzer Steel / Bottom Sweep	Air / 26'	Diver	:330	N
O'neal	10/8/2020	2361	Inspect Caissons, bents and bottom, Survey direction of Hamilton Construction Repress	Hamilton /Scottsburg Caisson OR	Air / 11'	Diver	:303	N
O'neal	10/14/2020	2114	Rig to pile stubs w crane, removal of debris to clean up bottom	PPM / Colman Dock	Air / 35'	Diver	:112	N
O'neal	10/19/2020	2218	Dry Dok F/V " Alaskan Defender " DDX #3	Foss / DD Alasken Def	Air / 17'	Diver	:233	N
O'neal	10/22/2020	2361	Perform inspection at Carisson 05, Install sandbags at base	Hamilton /Scottsburg Caisson OR	Air / 9'	Diver	:205	N
O'neal	10/23/2020	2361	Sandbag around Caisson 04	Hamilton /Scottsburg Caisson OR	Air / 10'	Diver	:207	N
O'neal	11/2/2020	2218	Foss Hail out PO 4500283385	Foss / Dry Docking	Air / 15'	Diver	:95	N
O'neal	11/10/2020	2368	Dry Dock M/V Wenatcher at Everett ship repair dry dock "Faithful servant"	WSF Wenatchee / Everett ship	Air / 24'	Diver	:316	N
O'neal	11/18/2020	2255	Outfall Survey, T-21 / T-71 / Short Well inspection	Boeing/ Ceccanti	Air / 15'	Diver	:385	N
Oneal	9/16/2020	2231	Bottom sweeps and debris clean up	Schnitzer Steel / General Metals	Air / 44'	Diver	:195	N
O'Neal	11/24/2020	2255	Assist w sand collar placement, chip concrete from vault to fit Sand Collar	Boeing / Ceccanti	Air / 16'	Diver	:570	N
O'Neal	12/1/2020	2356	Mob, survey on Bents 55-40	WSP/USCG Kodiak	Air /39'	Diver	:122	N
O'Neal	12/2/2020	2356	Magnetometer Survey of USCG Cargo Pier	WSP/USCG Kodiak	Air /38'	Diver	:165	N
O'Neal	12/14/2020	2218	Dry Dock #4	Foss / Gladys M	Air / 20'	Diver	:285	N

O'Neal	12/16/2020	2114	Dive to perfom deris sweep and remove concrete pile spices	Colman Dock / PPM	Air /32'	Diver	:300	N
O'Neal	12/22/2020	2201	inspect kink piling	Orion / T-5	Air /45'	Diver	:18	N
O'Neal	12/23/2020	2218	set vessel into Drydock	Foss / Gladys M	Air /20'	Diver	:35	N

Association of Diving Contractors International

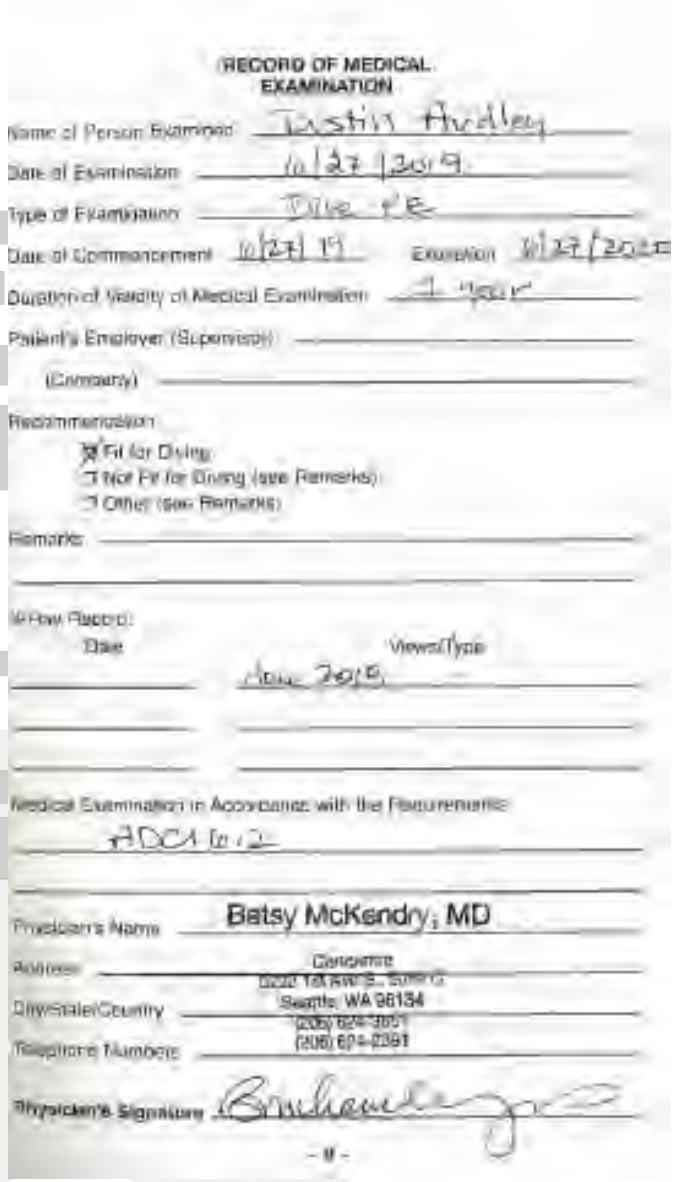
Cert. # (b) (6)

Expires 03/23/2025

SURFACE-SUPPLIED AIR DIVING SUPERVISOR

DUSTIN J. AUDLEY I.D. (b) (6)

Commercial Diver Certification Card



RECORD OF MEDICAL EXAMINATION

Name of Person Examined: Justin Hudley

Date of Examination: 10/27/2019

Type of Examination: Dive PE

Date of Commencement: 10/27/19 Expiration: 10/27/2020

Duration of Validity of Medical Examination: 1 year

Patient's Employer (Supervisor): _____

(Company): _____

Recommendation:

- [x] Fit for Diving
- [ ] Not Fit for Diving (see Remarks)
- [ ] Other (see Remarks)

Remarks: _____

If Pow Record:

Date	Views/Type
	June 2019

Medical Examination in Accordance with the Requirements:

ADC1 6.2

Physician's Name: Betsy McKendry, MD

Address: Concentra
5232 1st Ave S., Suite C
Seattle, WA 98134

City/State/Country: (206) 624-3651

Telephone Number: (206) 624-2391

Physician's Signature: [signature]

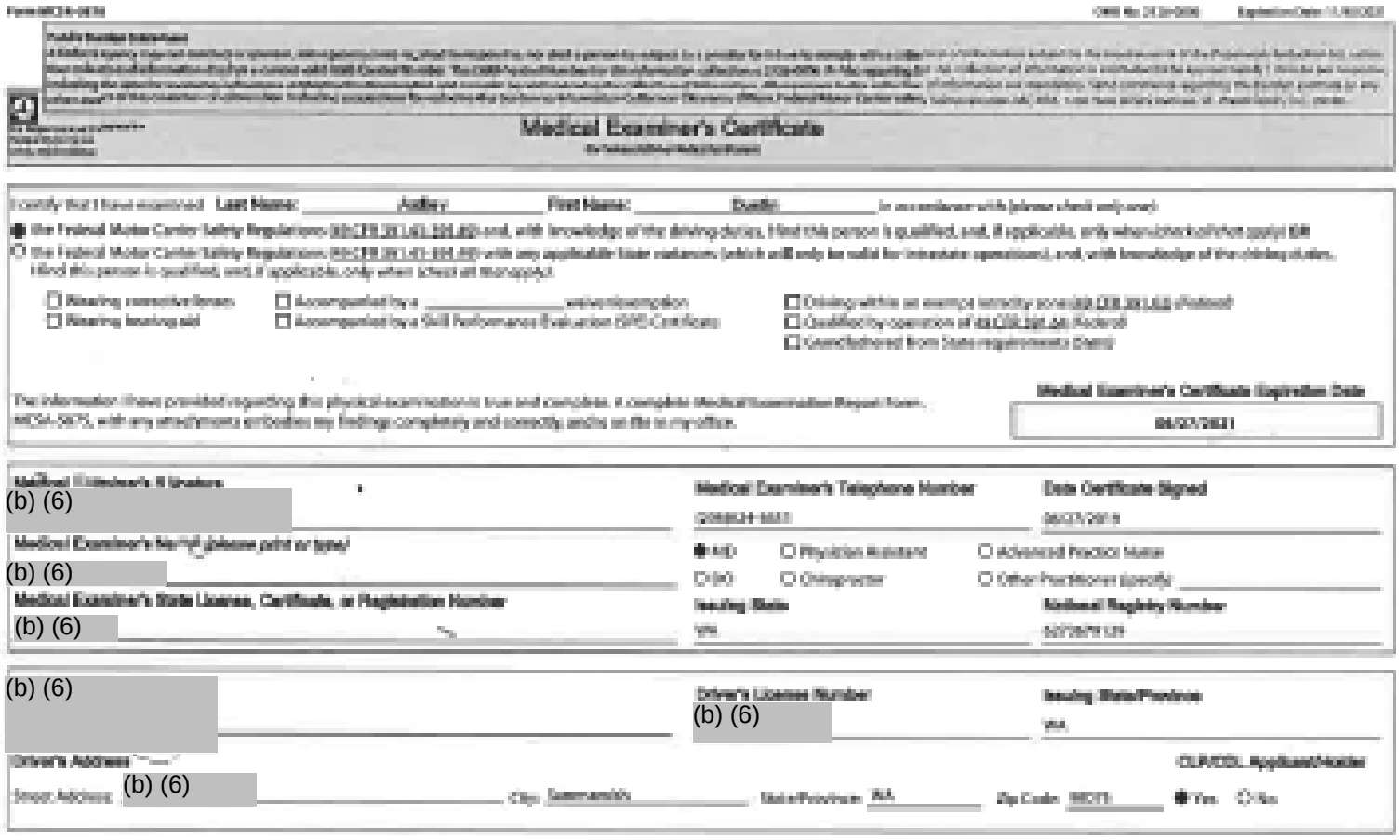
- 8 -

Record of Completion
Audley Dustin
has successfully completed all the requirements for the course
HAZWOPER 8-Hour Refresher
Completion Date: 4/10/2020

Form MCSA-5875 OMB No. 2120-0006 Expiration Date: 11/30/2021

Public Burden Statement

A Federal agency may not conduct or sponsor, and a person is not required to respond to, nor shall a person be subject to a penalty for failure to comply with a collection of information subject to the requirements of the Paperwork Reduction Act, unless that collection of information displays a current valid OMB Control Number. The OMB Control Number for this information collection is 2120-0006. In this reporting form, the collection of information is instructed to be approximately 1 minute per response, including the specific revisions, adjustments, and/or modifications, and consider by which the accuracy of the collection of information and mandatory. Send comments regarding the burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Information Collection Clearance Officer, Federal Motor Carrier Safety Administration, Washington, DC, USA. Use these letters, Mr. Paperwork Reduction Act, 2020.



Medical Examiner's Certificate

For Interstate Motor Carrier Safety

I certify that I have examined **Last Name:** Audley **First Name:** Dustin in accordance with (please check only one):

- ● the Federal Motor Carrier Safety Regulations (49 CFR 391.41-391.49) and, with knowledge of the driving duties, I find this person is qualified, and, if applicable, only when (check all that apply) OR
- ○ the Federal Motor Carrier Safety Regulations (49 CFR 391.41-391.49) with any applicable State variances (which will only be valid for intrastate operations), and, with knowledge of the driving duties, I find this person is qualified, and, if applicable, only when (check all that apply):

- ☐ Wearing corrective lenses
- ☐ Wearing hearing aid
- ☐ Accompanied by a _____ waiver/exemption
- ☐ Accompanied by a Skill Performance Evaluation (SPE) Certificate
- ☐ Driving within an exempt intracity zone (49 CFR 391.62) (Federal)
- ☐ Qualified by operation of 49 CFR 391.64 (Federal)
- ☐ Grandfathered from State requirements (State)

The information I have provided regarding this physical examination is true and complete. A complete Medical Examination Report Form, MCSA-5875, with any attachments embodies my findings completely and correctly, and is on file in my office.

Medical Examiner's Certificate Expiration Date

04/27/2021

Medical Examiner's Signature

(b) (6) [redacted]

Medical Examiner's Telephone Number

[illegible]

Date Certificate Signed

06/27/2019

Medical Examiner's Name (please print or type)

(b) (6) [redacted]

● MD ○ Physician Assistant ○ Advanced Practice Nurse

○ DO ○ Chiropractor ○ Other Practitioner (specify) _____

Medical Examiner's State License, Certificate, or Registration Number

(b) (6) [redacted]

Issuing State

WA

National Registry Number

[illegible]

(b) (6) [redacted]

Driver's License Number

(b) (6) [redacted]

Issuing State/Province

WA

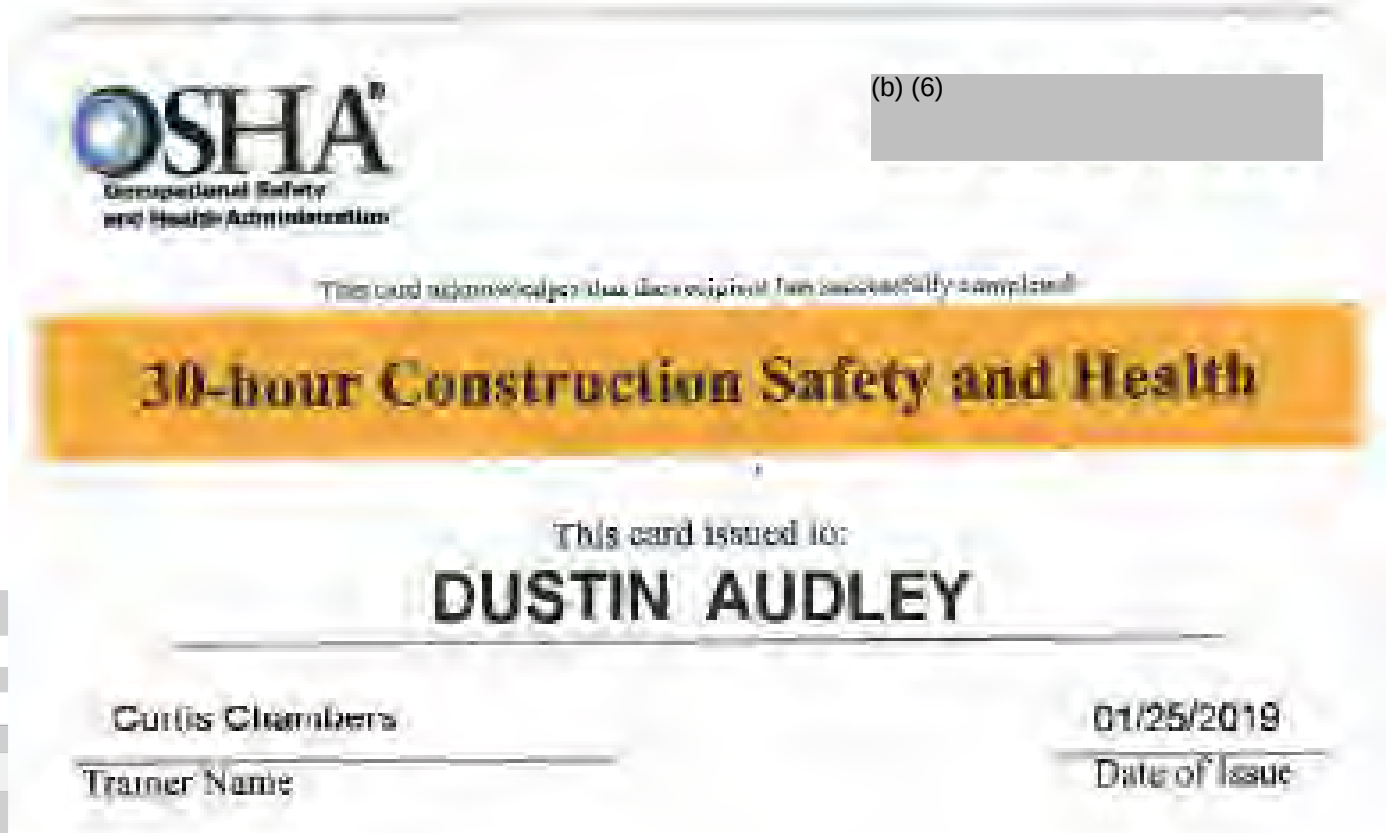
Driver's Address

Street Address: (b) (6) [redacted] City: Summerside State/Province: WA Zip Code: [illegible]

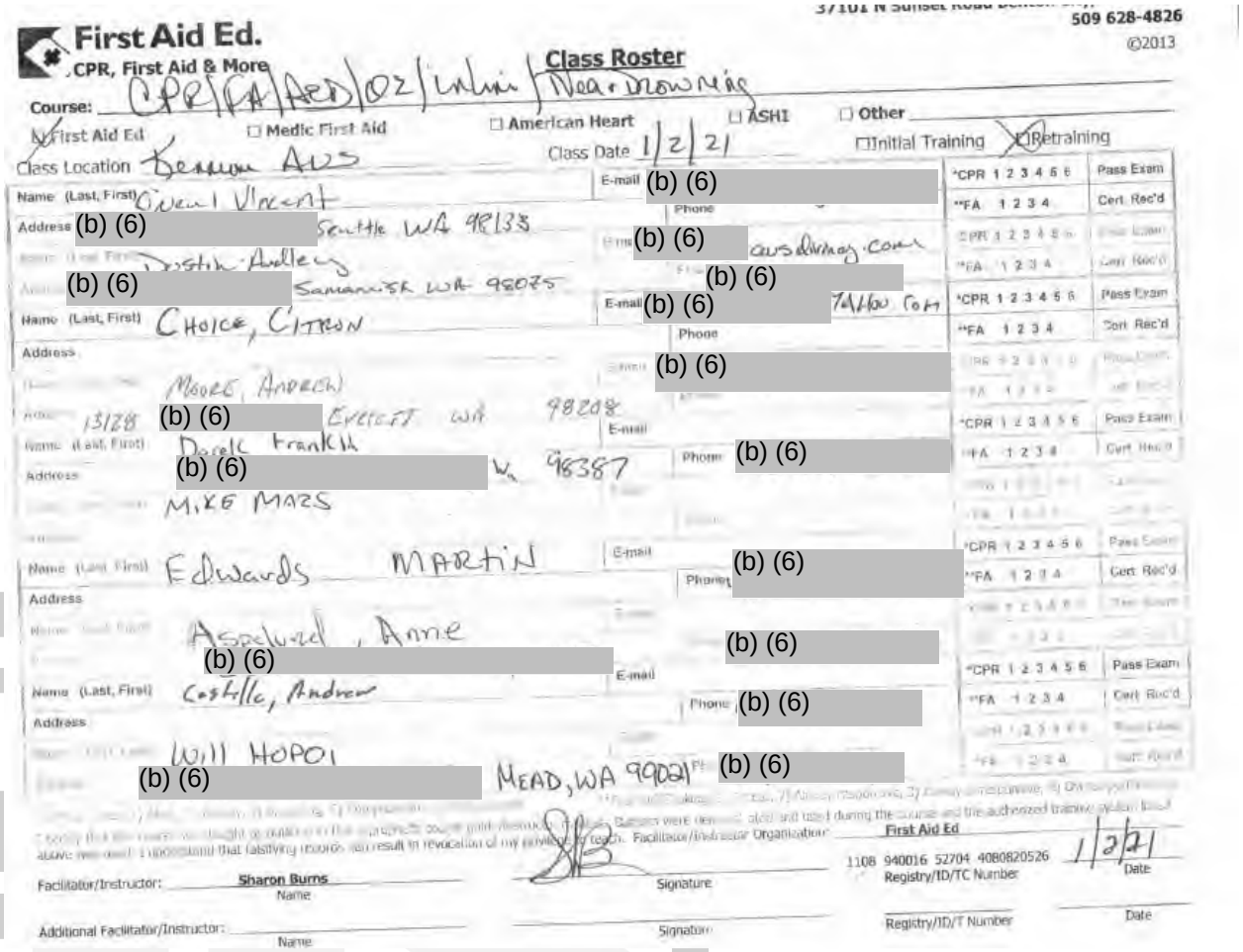
CLP/CDL Applicant/Holder

● Yes ○ No

This document contains sensitive information and is for official use only. Improper handling of this information could negatively affect individuals. Handle and secure this information appropriately to prevent inadvertent disclosure by keeping the documents under the control of authorized persons. Properly dispose of this document when no longer required to be maintained by regulatory requirements.


OSHA
Occupational Safety and Health Administration
(b) (6)
30-hour Construction Safety and Health
This card issued to:
DUSTIN AUDLEY
Curtis Chambers
Trainer Name
01/25/2019
Date of Issue

37101 N Sunset Road Denton Bay
509 628-4826
©2013



First Aid Ed.

CPR, First Aid & More

Class Roster

Course: CPR/FA/AED/O2/Infant/Near Drowning

☒ First Aid Ed ☐ Medic First Aid ☐ American Heart ☐ ASHI ☐ Other

Class Location Kenmore AUS Class Date 1/2/21 ☐ Initial Training ☒ Retraining

Name (Last, First)	Address	E-mail	Phone		
Cirenl Vincent	(b) (6) Seattle WA 98133	(b) (6)	(b) (6)	*CPR 1 2 3 4 5 6	Pass Exam
				**FA 1 2 3 4	Cert. Rec'd
Dustin Audley	(b) (6) Samamish WA 98075	(b) (6)	(b) (6)	*CPR 1 2 3 4 5 6	Pass Exam
				**FA 1 2 3 4	Cert. Rec'd
Choice, Citron		(b) (6)	74Hou Com	*CPR 1 2 3 4 5 6	Pass Exam
				**FA 1 2 3 4	Cert. Rec'd
Moore, Andrew	13128 (b) (6) Everett WA 98208	(b) (6)		*CPR 1 2 3 4 5 6	Pass Exam
				**FA 1 2 3 4	Cert. Rec'd
Derek Franklin	(b) (6) WA 98387		(b) (6)	*CPR 1 2 3 4 5 6	Pass Exam
				**FA 1 2 3 4	Cert. Rec'd
Mike Mazs					
Edwards Martin			(b) (6)	*CPR 1 2 3 4 5 6	Pass Exam
				**FA 1 2 3 4	Cert. Rec'd
Aspelund, Anne	(b) (6)		(b) (6)	*CPR 1 2 3 4 5 6	Pass Exam
				**FA 1 2 3 4	Cert. Rec'd
Castillo, Andrew			(b) (6)	*CPR 1 2 3 4 5 6	Pass Exam
				**FA 1 2 3 4	Cert. Rec'd
Will Hopoi	(b) (6) Mead, WA 99021		(b) (6)	*CPR 1 2 3 4 5 6	Pass Exam
				**FA 1 2 3 4	Cert. Rec'd

[illegible] Facilitator/Instructor Organization: First Aid Ed

Facilitator/Instructor: Sharon Burns (Name) Signature 1108 940016 52704 4080820526 Registry/ID/TC Number 1/2/21 Date

Additional Facilitator/Instructor: Name Signature Registry/ID/T Number Date

AUS

Dive Commercial International, Inc.

Diving Equipment Supply & Consultation

CUSTOMER: DUSTIN AUDLEY
HELMET S/N: KM-57 #6293KM
TECHNICIAN: JOHN RITTER
DATE: 11/6/20

SERVICE
- WELD LENS
- NO HEAD CUSHION

525-217 - SOFT GOODS KIT
510-010 - O-RING

Repair Estimate/Authorization Sheet

Dive Commercial Int'l.
Sales ✦ Service ✦ Consultation

Visual Inspection Certificate
JAN FEB MAR APR MAY JUN JUL AUG SEP OCT NOV

Attachment 2

Associated Underwater Services, Inc.

Contaminated Diving & Decontamination Procedure.

Portland Harbor Sediment Sampling.

Northwest Industrial.
5924 NW Front Ave Portland, OR 97210
June 29th. 2021
Jacods Engineering

PART 1 GENERAL

A. This section specifies the requirements for disinfecting and disposal of contaminated materials & equipment.

B. The River bottom soil / sediment at this jobsite is presumed to be contaminated with chemicals, heavy metals and is classified as a superfund site. This soil, sediment and water may or may not contain chemicals; however, it is assumed the water does not contain hazardous materials. AUS has taken necessary safety measures to protect the divers and crew. The divers will wear AUS provide vulcanized rubber suit for project. The divers Helmet will be mated to the vulcanized rubber suit, gloves will also be connected to the suit to insure there is no exposed skin. The top side crew will wear proper PPE to handle diving equipment in and out of the contaminated and decontamination zone. The vessel has been designated with three zones Support Zone (GREEN), Contamination Reduction Zone (YELLOW) and Exclusion Zone (RED).

- The Support Zone (Green) is for crew support, no contaminated materials are permitted in this zone. clean zone no possibility of contamination. The support zone is shown as green in the attached diagram and is designated for the topside support crew only.
- The Contamination Reduction Zone (Yellow) is the neutral zone, equipment and personal have been cleared of contaminates and safe to enter the Support Zone. The contamination Reduction Zone is for the removal and collection of equipment that has been cleared of all hazards and contaminates. This zone is yellow in the attached diagram and is designated for removal of diver's equipment and collection of materials after being deemed safe and decontaminated.

- The Exclusion Zone is the contaminated zone. Qualified personal only permitted in this zone. Qualified personals decontaminate the diver and equipment in this zone. Everything in this zone is considered contaminated and will not permitted to enter the reduction zone until deemed safe. After diver has been cleared from the exclusion zone the diver and crew are able to proceed to the contamination reduction zone where the diver can remove their equipment.

*Note: The vessel will be anchored with the support Zone facing down wind of the Exclusion Zone. wind direction will be always monitored ensuring that the wind is *NOT* blowing toward the Support Zone.

1. Divers will be equipped with a Kirby Morgan diving helmet that will mate to a vulcanized rubber dry suit for ultimate protection. Gloves will also mate to the vulcanized rubber dry suit for ultimate protection.

2. Equipment used in the water that may encounter soil / sediment and debris. Sediment sample equipment, Diver, Dive hose and necessary hand tools for moving material and sediment.

PART 2 PRODUCTS

The following steps shall be taken every time a boat, equipment or gear is moved between contaminated waters to avoid transporting toxins, invasive species and/or pathogens from one water source to another.

- **Inspect** and **remove** aquatic plants, soil, animals, and mud from boat, trailer, equipment, and gear.

- **Drain** all water from boat, motor, live well, bilge, transom wells, as well as from equipment and gear, including but not limited to tracked vehicles, barges, silt or turbidity curtain, hoses, sheet pile and pumps.

- **Dispose** of unwanted aquatic plants and animals in an appropriate way.

- **Disinfect** boat, equipment and gear by either:

- **Washing** with ~>104º F water (steam clean), OR

- **Drying** thoroughly for 5 days after cleaning with soap and water and/or high-pressure water.

- **Disinfecting** with either 200 ppm (0.5 oz per gallon or 1 Tablespoon per gallon) Chlorine (Hasachlor) for 10-minute contact time or 1:100 solution (38 grams per gallon) of Virkon Aquatic for 20- to 30-minute contact time.

Note: Virkon is not registered to kill zebra mussel veligers nor invertebrates like spiny water flea. Therefore, this disinfect should be used in conjunction with a hot water (>104º F) application.

Safety Precautions for Disinfectant Use: Virkon-A:

1. Receive and be required to read a copy of the Virkon-A Materials Safety Data Sheet (MSDS) for the product.

2. Wear chemical splash goggles.

3. Wear a face shield where the possibility exists for face contact due to splashing or spraying of the material.

4. Wear impervious clothing to prevent contact with skin. (Gloves, pants, jacket, hood, and boots) or a Tyvek style full body suit.
In addition, all employees who handle or mix Virkon-A in powder form and prefer to wear a dust mask respirator when handling powder.

Bleach:
Follow precautions 2, 3, and 4 (above).

- Chlorine (Hasachlor) Wear eye protection, rain gear, gloves if spraying. Stay upwind of the spray. Will break down in sunlight and when in contact with organic material. Is corrosive to metal and rubber. Is toxic to fish at these concentrations so rinse well after disinfection or neutralize with sodium. thiosulfate. For neutralizing chlorine, spray sodium thiosulfate in an 800 ppm solution (3 grams per gallon of water) on all surfaces after the disinfection period is over. Rinse with water from the next lake to remove any remaining sodium thiosulfate.

- Virkon Aquatic This is a disinfectant in the peroxygen (hydrogen peroxide) family. It is a powder. It is 99.9% biodegradable and breaks down to water and oxygen and is not corrosive at the working dilution. Wear dust mask if mixing powder and eye protection, rain gear and gloves if spraying. Stay upwind of spray.

Sources of disinfectants.

Chlorine (Hasachlor) - Household bleach (5.25% chlorine) can be purchased from a grocery or convenience store. HTH is granular chlorine (70% calcium hypochlorite) and can be purchased from a pool supply company.

Sodium Thiosulfate - Commonly used to neutralize chlorine and iodine. It should be available at a pool supply company or from a chemical supply company.

Virkon Aquatic is available from Western Chemical. It is the same formulation, but without the perfume and dye, and the label addresses specific fish pathogens. Their phone is 1-800-283-5292.

Disinfection measures must be taken prior to moving boats, equipment, and other gear from one waterbody to another. They are not needed daily when sampling the same waterbody or for law enforcement equipment in emergency situations. In cases where boats and gear return to state hatcheries or potable water reservoirs disinfection should be done in a location away from ponds and water supplies to prevent disinfectant or untreated water from entering those areas. Every effort should be made to keep the disinfection solution and rinse water out of surface waters.

To the extent practicable, equipment and gear used on waters known to be infested with invasive species and viruses should not be used on other non-infested waters. The following are some helpful hints to consider when planning your work in water.

- Organize your tooling / equipment so the work in infested waters is always done last

- If a high percentage of your work is done in waters with invasive species, consider dedicating certain gear to be used only in those waters.

The following methods are provided to assist staff when disinfecting equipment and gear commonly used by diving staff.

Personal protective gear, (PPE) including rain jacket and pants, Rubber gloves, boots/waders, dry suits and or Tyvek suits, Safety Glasses and or face shield.

Scrub personal protective gear with the disinfection solution. After scrubbing, the gear should be kept wet with the disinfection solution for the appropriate contact time. Rinse with clean water or water that is known not to be contaminated. Alternatively, personal gear may be steam cleaned or dried thoroughly for five days after cleaning with soap and water.

Tools

Remove any organic material from the tools and equipment. There are several options for disinfecting tooling.

- Option one: The tooling can be sprayed with the disinfection solution and a wet surface maintained for the appropriate contact time. The tooling should be rinsed with clean water or water from the next waterbody before it is used again.

- Option two: Fill a tub with disinfection solution and place all equipment in the tub for the appropriate contact time. The tooling should be rinsed with clean water or water from the next waterbody before it is used again.

- Option three: Use a completely new set of gear for each waterbody during the workday and disinfect all tooling at the end of the day using option one or two.

- Option four: Clean off any organic debris, wash with soap and water and let the tooling sit in the dry (not a damp environment) for a minimum of 5 days.

Boats, trailers, and live wells

Remove organic material from boats, trailers, and live wells. Drain water from live wells, bilges, and pumps. The outside and inside of the boat, trailer, live wells, bilges, and pumps should be sprayed with the disinfection solution and left wet for the appropriate contact time. The inside of the live wells, bilges and pumps should be made to contact the solution for the appropriate contact time as well. Run pumps so they take in the disinfection solution and make sure that the solution meets all parts of the pump and hose. The boat, trailer, bilges, live well, and pumps should be rinsed with clean water or water from the next waterbody after the appropriate contact time. *Every effort should be made to keep the disinfection solution and rinse water out of surface*

waters. Pull the boat and trailer off the ramp and onto a level area and away from street drains to minimize potential runoff into surface waters.

Motors

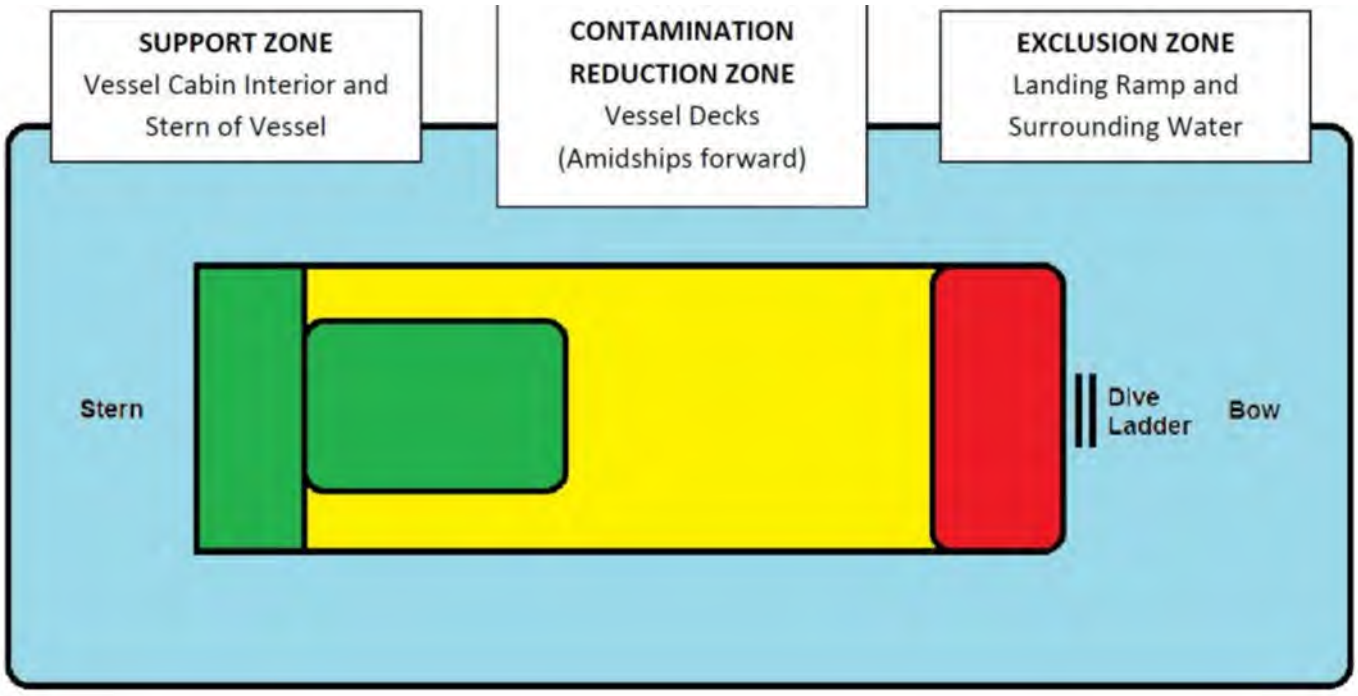
After removing from the water, tip the motor to the down position and start the motor for several seconds or turn motor over several times to dispel water from the cooling system. Alternatively, and especially for motors moored in water for several days or more, emerge the lower unit in a bucket of disinfectant and run the motor to ensure contact with all internal parts and allow for the appropriate contact time. Or rig up a short (6-foot) piece of garden hose to lower unit muffs. A pail of the disinfectant can be set in the back of the boat and gravity fed to the lower unit to run the disinfectant through the motor.

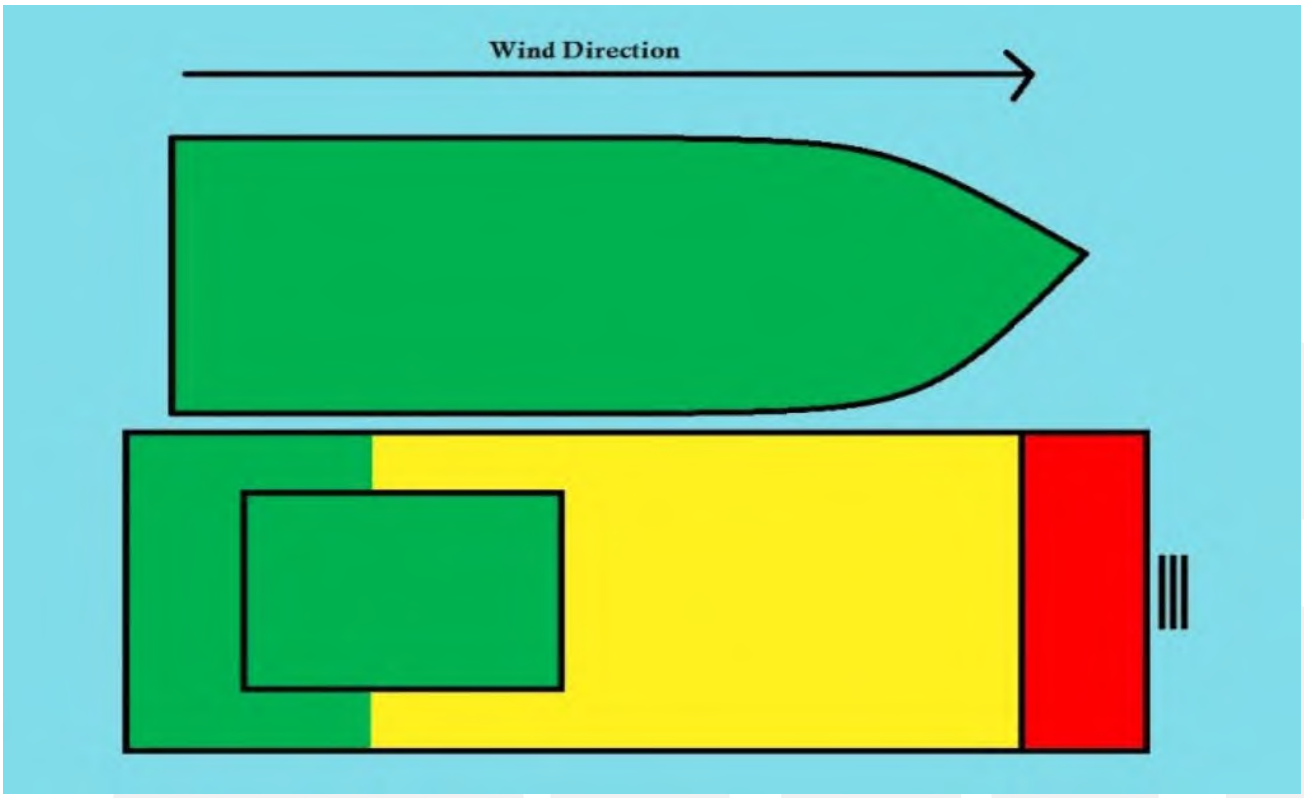
Allow solution to remain in motor for the appropriate contact time. The hose will need to be primed to start the gravity flow because the lower unit does not create enough suction to prime the hose. A non-corrosive (Virkon Aquatic) is recommended for use to protect the impeller. Rinse with clean water or water from the next waterbody.

Heavy Equipment

For heavy equipment steam-cleaning is an effective method of disinfection.

AUS


SUPPORT ZONE
Vessel Cabin Interior and
Stern of Vessel
CONTAMINATION
REDUCTION ZONE
Vessel Decks
(Amidships forward)
EXCLUSION ZONE
Landing Ramp and
Surrounding Water
Stern
Dive
Ladder
Bow


Wind Direction

AUS
ASSOCIATED UNDERWATER SERVICES

CERTIFICATE OF DISINFECTION

Pursuant to AUS equipment disinfection procedures the following equipment has been deemed clean and free of invasive species, viruses, pathogens and contaminates.

Equipment and Description:

- Dive Hose # _____
- Stand by Hose # _____
- Divers Helmet #_____
- Divers Suit# _____
- Vessel _____
- Trailer Plate # _____
- Sediment sampling equipment
- Bucket of hand tools with tape measure and scrapers
- Random lengths of line Disinfection Procedure

✓ *All washed in the exclusion zone of vessel then all diving gear will be collected and soaked with designated solution.*

I certify that the above equipment has been cleaned in accordance with AUS disinfection procedures and is free of debris, invasive species, pathogens, viruses & contaminates.

_____ _____

AUS

Attachment 3

ASSOCIATED UNDERWATER SERVICES

DIVE HOSE MAINTENANCE RECORD

I.D. # 48 LENGTH 320' TYPE Gates with Video

DATE WORK STARTED 4/21/20 WORK DONE BY Dustin Axelle

(SIGN & PRINT NAME)

A – DIVING HOSE

TEST PRESSURE 300 P.S.I. TIME ON TEST 10 MIN.

① FITTINGS	☑ O.K.	☐ REPLACE
② HOSE CONDITION	☑ O.K.	☐ REPLACE
③ D-RING & LIFELINE	☑ O.K.	☐ REPLACE

PULL TEST 200 LB TIME ON TEST 10 MIN.

B – PNEUMO HOSE

① FITTINGS	☑ O.K.	☐ REPLACE
② HOSE CONDITION	☑ O.K.	☐ REPLACE

C – COMMUNICATION CABLE

① CONTINUITY	☑ O.K.	☐ REPLACE

D – GENERAL CONDITION

NEEDS TAPING?, I.D. TAG?, GREASY?, ETC. _____

LIST WORK PERFORMED & ALL PARTS REPLACED

Retaped hose added new video cable.

REMARKS _____

DATE COMPLETED 4/21/20 APPROVED BY _____

(SUPERVISOR)

LOCATION Seattle

(VESSEL)

AUS DIVE HOSE MAINTENANCE RECORD

I.D. # 46 LENGTH 360 TYPE Gates with Video & Hw

DATE WORK STARTED 4/21/20 WORK DONE BY Destin Dudley

(SIGN & PRINT NAME)

A – DIVING HOSE

TEST PRESSURE _____ P.S.I TIME ON TEST _____ MIN.

① FITTINGS	☑ O.K.	☐ REPLACE
② HOSE CONDITION	☑ O.K.	☐ REPLACE
③ D-RING & LIFELINE	☑ O.K.	☐ REPLACE

PULL TEST _____ LB TIME ON TEST _____ MIN.

B – PNEUMO HOSE

① FITTINGS	☑ O.K.	☐ REPLACE
② HOSE CONDITION	☑ O.K.	☐ REPLACE

C – COMMUNICATION CABLE

① CONTINUITY	☑ O.K.	☐ REPLACE

D – GENERAL CONDITION

NEEDS TAPING? I.D. TAG?, GREASY?, ETC. _____

LIST WORK PERFORMED & ALL PARTS REPLACED Patched hose added new video cable.

REMARKS _____

DATE COMPLETED 4/21/20 APPROVED BY _____ (SUPERVISOR)

LOCATION Seattle (VESSEL)

Environmental Health Laboratory
Department of Environmental and Occupational Health Sciences
University of Washington

7/1/2020

To Kerry Donohue
kerry@ausdiving.com
Associated Underwater Services, Inc
6706 NE 175th Street, Unit D
Kenmore, WA 98028-4811

Samantha Crumpton
samantha@ausdiving.com

From Christine Olson

Subject Test Report For EHL#: 12006003

Dates	Receipt	Analysis
	6/22/2020	7/1/2020

Method EHLSOP-02: Analysis of Compressed Breathing Air

Results

		Client ID	Comp 16	Comp 17	Comp 1	Comp 20	Comp 21	Comp 22
		Sampling Date	06/11/20	06/11/20	05/28/20	06/11/20	06/22/20	06/22/20
Parameter	Measurement Method	Acceptance Range for WAC-CD	Result	Result	Result	Result	Result	Result
Carbon Dioxide, ppm	FTIR	1000 (+ 21)	477	533	507	467	386	447
Carbon Monoxide, ppm	FTIR	10 (± 0.3)	≤ 2	≤ 2	≤ 2	≤ 2	≤ 2	≤ 2
Methane, ppm	FTIR	NA	2	2	2	2	3	2
Water, ppm	FTIR	NA	> 100	> 100	> 100	> 100	> 100	> 100
Oil Mist/Particulate Matter	Qualitative/Visual Observation	No visible discoloration (1)	None	None	None	None	None	None
Oxygen (%)	TDLAS	NA	21	21	21	21	21	21
Odor	Organoleptic	Qualitative (2)	Slight	Slight	OK	OK	Slight	Slight
Are all parameters within acceptance range?			Yes	Yes	Yes	Yes	Yes	Yes

Quality Assurance

Parameter	Oxygen	Carbon Dioxide	Carbon Monoxide	Methane	Water
R^2, Calibration	NA	0.9976	0.9993	0.9993	0.9949
Lower Reporting Limit, ppm	1 %	30	2	1	14

Please arrange to have your sample tank(s) picked up at your earliest convenience.

Please Note:

1. The regulatory limit is < 5mg/m3. We don't perform quantitative gravimetric analysis for oil mist/particulate matter. A "Not passing" grade will be automatically issued to the sample if visible dust/oil mist is observed.
2. A "Not passing" grade will be automatically issued to the sample if a "pronounced" odor is detected.
3. Results apply only to the samples tested.
4. A background spectrum of the cell purged with purified nitrogen is subtracted from each sample spectrum.

Sample condition was acceptable unless otherwise noted.

Reviewed By: _____ 7-2-20

QAC Designee — Blair Sawyer PhD, Director, EH Laboratory/TOAC, Phone: 206-543-3263, E-mail: @uw.edu — Date

This test report shall not be reproduced except in full without written approval of the laboratory.

Department of Labor and Industries
Specialty Compliance Services
P.O. Box 44410, Olympia, WA 98504-4410

CERTIFICATE OF INSPECTION

INSPECTION DATE	EXPIRATION DATE
01/27/2021	01/29/2023
SPECIFIC PLANT LOCATION	STATE NUMBER
Volume Tank 21	01022-21W

LOCATION OF OBJECT	OBJECT INFORMATION	
Associated Underwater Services 6706 NE 175th St Unit D Kenmore, WA 98028-4811	Other #	291041
	NB #	
	MANUFACTURER	Samuel Pressure Vessel Group
	TYPE	AT
	USE	Storage
	SV SETTING	200
	YEAR BUILT	2020
	INSPECTOR	Stephan Osterbur
	INSP. EMPLOYER	State of Washington

Post this certificate in a conspicuous place in the room containing the object. It must be available for viewing. This certificate is not valid unless the invoice has been paid. The Object shall not be operated at a pressure in excess of that shown. Notify the State of Washington, Department of Labor and Industries at 360-902-6400 or your insurance carrier 60 days prior to the expiration date. It is a violation of WAC 296-104-701 to operate the object without a valid inspection certificate posted near the object.

AUS

DECK WHIP MAINTENANCE RECORD

I.D. # 010 LENGTH 35' TYPE #10 Dk Whip

DATE WORK STARTED 05-07-20 WORK DONE BY Chase Ross

(SIGN & PRINT NAME)

A – DIVING HOSE

TEST PRESSURE 300 P.S.I. TIME ON TEST 10 MIN.

① FITTINGS	☒ O.K.	☐ REPLACE
② HOSE CONDITION	☒ O.K.	☐ REPLACE

B – GENERAL CONDITION

NEEDS TAPING? I.D. TAG?, GREASY?, ETC. _____

LIST WORK PERFORMED & ALL PARTS REPLACED Annual Test

REMARKS _____

DATE COMPLETED 05/07/20 APPROVED BY _____ (SUPERVISOR)

LOCATION Spokane Shop (VESSEL)

AUS

DECK WHIP MAINTENANCE RECORD

I.D. # 75 LENGTH 25' TYPE Deck whip

DATE WORK STARTED 3/13/20 WORK DONE BY J O'Neal

Vincent O'Neal
(SIGN & PRINT NAME)

A – DIVING HOSE

TEST PRESSURE 300 P.S.I TIME ON TEST 10 MIN.

① FITTINGS ☑ O.K. ☐ REPLACE

② HOSE CONDITION ☑ O.K. ☐ REPLACE

B – GENERAL CONDITION

NEEDS TAPING? I.D. TAG?, GREASY?, ETC. _____

LIST WORK PERFORMED & ALL PARTS REPLACED Annual Pressure Test

REMARKS _____

DATE COMPLETED 3/13/20 APPROVED BY _____
(SUPERVISOR)

LOCATION Seattle.
(VESSEL)

Appendix E
Waste Management Plan

Jacobs

Portland Harbor Willbridge Cove Project Area
Portland, Oregon

Waste Management Plan

Final

June 10, 2021

Pre-Design Investigation Work Plan,
Willbridge Cove Project Area

Portland Harbor Willbridge Cove Project Area
Portland, Oregon

Project No:	D3372300
Document Title:	Waste Management Plan
Document No.:	
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Date:	May 2021
Client Name:	Willbridge Cove Group
Project Manager:	Marilyn Gauthier

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Acronyms and Abbreviations

Acronym	Definition
ARAR	Applicable or Relevant and Appropriate Requirement
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CFR	Code of Federal Regulations
DOT	U.S. Department of Transportation
EPA	U.S. Environmental Protection Agency
HSP	Health and Safety Plan
ID	identification
Jacobs	Jacobs Engineering Group Inc.
LDR	land disposal restriction
OAR	Oregon Administrative Rule
OSR	Off-Site Rule
PCB	polychlorinated biphenyl
PID/FID	photoionization detection/flame ionization detection
PPE	personal protective equipment
RCRA	Resource Conservation and Recovery Act of 1976
ROD	Record of Decision
SVOC	semi-volatile organic compound
TCLP	toxicity characteristic leaching procedure
TSCA	Toxic Substances Control Act
UN	United Nations
VOC	volatile organic compound
WCPA	Willbridge Cove Project Area

Note: If using a screen reader, adjustment to your default settings may be required.

1. Introduction

This Waste Management Plan contains procedures that will be implemented to maintain compliance, prevent pollution, and protect existing environmental resources during the investigation phase of work at the Willbridge Cove Project Area (WCPA) in Portland, Oregon. The WCPA is located on the Willamette River, between river mile 7.5 and 8 and is a part of the Portland Harbor Superfund Site.

The requirements outlined in this plan are intended to provide compliance with:

1) Resource Conservation and Recovery Act of 1976 (RCRA) 40 *Code of Federal Regulations* (CFR) 261 through 265 – Standards for waste characterization, classification, management, and treatment or disposal; Oregon has adopted and incorporates by reference the Federal RCRA hazardous waste management program
2) Oregon Administrative Rule (OAR) 340 Divisions 100 through 106; Oregon has adopted and incorporates by reference the Federal RCRA hazardous waste management program
3) Oregon State-only Waste Codes
4) 40 CFR Part 761.61(b) – Toxic Substances Control Act (TSCA) – Management of polychlorinated biphenyl (PCB) waste
5) Applicable or Relevant and Appropriate Requirements (ARARs) as provided in the Record of Decision (ROD) (EPA 2017)
6) U.S. Department of Transportation (DOT) shipping requirements as provided in 49 CFR 172

2. Transportation and Offsite Disposal Plan

The following subsections describe the CERCLA off-site rule and transportation requirements.

2.1 CERCLA Off-Site Rule Approval (40 CFR 300.440)

The CERCLA Off-Site Rule (OSR) 40 CFR 300.440 states that CERCLA wastes may only be placed in a facility operating in compliance with RCRA or other applicable federal or state requirements. It also applies to any CERCLA response action involving the offsite transfer of any hazardous substance, pollutant, or contaminant (CERCLA wastes).

Waste generated from in-water areas of the site will comply with the CERCLA OSR requirements. In-water is defined as the portion of the WCPA that contains all material from below the top of bank (approximately +30 feet North American Vertical Datum of 1988) and includes in-river sediments. All wastes associated with the predesign investigation will originate from site areas required to meet CERCLA OSR requirements.

The appropriate CERCLA OSR representative (based on the state where the disposal facility resides), will be contacted via email before disposal of the waste. Table E-2-1 provides contact information.

Table E-2-1. CERCLA Off-Site Rule Contacts

Waste Management Plan, Willbridge Cove Project Area, Portland, Oregon

EPA Region	Contact Name and Email Address	Telephone
1	Conor O'Brien (Obrien.Conor@epa.gov)	617.918.1769
2	Bonnie Hriczko (Region2_OSR@epa.gov)	732.321.6647
3	Claudia Scott (R3CERCLA_Offsite@epa.gov)	215.814.3240
4	Kayla Acosta (r4cercla_offsitecontact@epa.gov)	404.562.8451
5	William Damico (r5cercla.roc@epa.gov)	312.353.8207
6	Wilkin (Ron) Shannon (shannon.wilkin@epa.gov)	214.665.2282
7	Mike Martin (martin.mike@epa.gov)	913.551.7149
8	Linda Jacobson (jacobson.linda@epa.gov)	303.312.6503
9	Sharon Lin (lin.sharon@epa.gov)	415.972.3446
10	Cheryl Williams (williams.cherylb@epa.gov)	206.553.2137

Source: EPA 2020

The following information will be provided to the CERCLA OSR representative in the email:

1) The name of the facility or facilities to which the waste may be sent (for example, Clean Harbors Grassy Mountain facility)
2) The U.S. Environmental Protection Agency (EPA) identification (ID) number(s) or other unique identifying number(s) (e.g., UTD991301748)
3) The city and state in which each potential receiving facility is located (e.g., Clive, Utah)
4) WCPA – Portland Harbor Superfund Site, CERCLA Docket No. 10-2018-0236
5) The type of waste or wastes to be shipped (e.g., hazardous waste, lead contaminated soil)

6) The quantity for each waste to be sent (e.g., 100 cubic yards)
7) When the waste is to be shipped (e.g., September 2020)

A copy of the OSR approval record must be maintained in the project files.

2.2 Notification of Out-of-State Waste Receiving Facilities

Waste generated as part of predesign investigations in quantities greater than 10 cubic yards, and shipped to out-of-state treatment, storage, or disposal facilities require written notification to the appropriate state environmental officer and the EPA Project Coordinator. Written notification to the state and the EPA Project Coordinator will include the following information:

- Name and location of the waste receiving facility
- The type and quantity of waste being shipped
- Schedule for shipment of waste
- Method of waste transportation

Major changes made to the management of out-of-state shipped waste will also be communicated to the appropriate state compliance officer and the EPA Project Coordinator before the waste is shipped.

2.3 Transportation and Disposal Documentation

Before offsite disposal of waste, a waste designation memorandum (waste memo) will be prepared. This waste memo will include a waste profile, analytical summary table(s) applicable to the waste, a draft waste manifest, and any other applicable information necessary to complete review of the disposal package and provide signature as the generator.

The signed profile will then be submitted to the disposal facility for acceptance and approval. Once the approval letter is received from the disposal facility, transportation can be scheduled, and appropriate notifications will be made for out-of-state facilities.

Each load of waste will be manifested before leaving the site. Nonhazardous waste will be manifested on nonhazardous waste manifests. Hazardous waste must be shipped using EPA Hazardous Waste manifests. At a minimum, the manifest form will include the following information:

- Generator information, including name, address, contact, and phone number (EPA ID number will be added for hazardous waste)
- Transporter information, including name and EPA ID number for hazardous waste
- Facility information, including name, address, phone number, and EPA ID number
- Site name, including street address at a minimum and mailing address if available
- Type and number of container(s)
- Quantity of waste (volumetric estimate)
- Profile number
- 24-hour emergency phone number

The generator, or their designated representative, and the transporter must sign the manifest before the load of waste leaves the site. A copy of the manifest will be retained in the project file. The original signed manifest will be returned to the generator. To comply with applicable DOT regulations for shipping of

hazardous materials, paper manifests will accompany all waste shipments from the site to the receiving facility.

2.4 Transporter Requirements

Each transportation vehicle and load of waste will be inspected before leaving the site and the inspection will be documented. The quantities of waste leaving the site will be also be recorded on a waste tracking log. A transporter licensed for commercial transportation and who maintains a valid DOT number will transport nonhazardous wastes. If waste is classified as hazardous waste, the transporter will also have appropriate hazardous material certifications, and a valid EPA ID number.

A copy of the documentation indicating the selected transporter has appropriate licenses will be received and approved by Jacobs Engineering Group Inc. (Jacobs) before waste is transported.

The transporter will observe the following practices when hauling and transporting wastes offsite:

- Minimize impacts to general public traffic
- Verify that seals on trucks transporting liquids are in good condition
- Waste or material from other sites may not be combined with waste generated during the project

Personnel involved in offsite disposal activities will follow safety and spill response procedures outlined in the Health and Safety Plan (HSP).

Loads will be inspected to ensure that they are secure, proper placarding is provided as necessary, and shipping documentation is accurate.

2.5 Disposal Requirements

Offsite disposal facilities will use the waste profile and supporting documentation, such as analytical results, to determine if the facility will accept a waste. The disposal facility will be responsible for providing a copy of the final waste manifest and for a certificate of treatment or disposal for each load of waste received.

3. Onsite Waste Management

This section of the Waste Management Plan describes the potential waste streams that will be generated and how those wastes will be managed onsite.

3.1 Waste Management

Wastes generated during the predesign investigation will be managed in accordance with 40 CFR 261 Subpart C and OAR 340-101-0033. Onsite management and offsite disposal will also comply with ARARs as established in the Portland Harbor Superfund ROD (EPA 2017).

Table E-3-1 provides anticipated waste streams and management method for the predesign investigation-derived waste.

Table E-3-1. Project Wastes

Waste Management Plan, Willbridge Cove Project Area, Portland, Oregon

Waste Stream	Classification	Analysis Required[a]	Onsite Management Method	Final Disposition
Soil and sediment	Nonhazardous	Yes (up to full suite of analysis)	Placement at the site of origin or containerized in UN-rated new 55-gallon drums or UN-rated 5-gallon containers	Onsite or landfill
Decontamination water (processing area)	Nonhazardous	Yes (up to full suite of analysis)	Liquid contained in UN-rated new 55-gallon steel drums, UN-rated 5-gallon containers, or totes provided with secondary containment Sediment contained in UN-rated new 55-gallon steel drums or UN-rated 5-gallon containers	Offsite treatment facility
Sediment core liners	Nonhazardous	No, generator knowledge	Placed into containers with sediment	Offsite landfill
Nonaqueous phase liquid	Hazardous	Identify analytical requirements upon discovery	Liquid contained in new UN-rated 5-gallon container	Offsite treatment or incineration
Contaminated debris, including PPE plastic sheeting, disposable sampling equipment	Nonhazardous	No, generator knowledge	Trash bags	Offsite landfill

Notes:
a = The full suite of analysis is provided in Section 3.2.1 for soil and sediment, and Section 3.2.2 for liquid
PPE = personal protective equipment
UN = United Nations

All waste generated during the predesign investigation will be disposed of at CERCLA OSR-approved facilities only, as described in Section 2.1.

This Waste Management Plan will be reviewed before site remediation and updated with information acquired during the remedial design. The review will also verify the plan complies with applicable regulatory requirements and ARARs established in the ROD (EPA 2017).

3.2 Waste Characterization and Classification

Analytical results will be compared to the criteria in 40 CFR Part 261 Subpart C and Oregon Administrative OAR 340 Divisions 100 through 106 to characterize waste. PCB results will be compared to the TSCA limit of 50 parts per million to identify whether waste requires a TSCA-regulated disposal facility.

3.2.1 Sediment and Soil

Soil and sediment waste samples will be analyzed using the following methods as appropriate for characterization and the offsite disposal facility:

- Toxicity characteristic leaching procedure (TCLP) volatile organic compounds (VOCs) by 1311/8260B
- TCLP semi-volatile organic compounds (SVOCs) by 1311/8270C
- TCLP pesticides by 1311/8081A
- TCLP herbicides by 1311/8151A
- TCLP metals by 1311/6010B/7000
- Total petroleum hydrocarbons (if needed for state acceptance criteria)
- PCBs by 8082A
- Ignitability by 1010/1030
- Corrosivity by 9045C

3.2.2 Liquids

Liquid wastes will be analyzed for the following as appropriate for characterization and the offsite disposal facility:

- VOCs (8260B)
- SVOCs (8270C)
- Pesticides (8081A)
- Herbicides (8151A)
- Metals (6010B/7000)
- Corrosivity (9045C)
- Ignitability (1010)

3.2.3 Investigation-derived Waste Collection Details

The general procedures for collecting waste characterization samples are provided in the following subsections.

3.2.3.1 Solid Waste

Analysis will be run for every 10 drums/containers or every roll-off container.

Composite: One composite sample should be collected for every 10 drums – EXCEPT FOR VOCs. The composite sample will consist of an equal aliquot from each drum. If 10 drums are not generated, an equal aliquot from each drum will be used to prepare the composite.

Grab: One representative grab sample for VOCs should be collected for every 10 drums or 1 roll-off, biased toward the area with the highest photoionization detection/flame ionization detection (PID/FID) reading. If this information does not indicate a clear maximum, the grab sample should be collected at random.

3.2.3.2 Liquid Waste

Composite: One composite sample will be collected for every 10 drums of this waste stream. The composite sample will consist of an equal aliquot collected from each drum. The use of any other compositing technique is prohibited without approval from the Project Environmental Manager or Waste Coordinator.

Grab: One representative grab sample for VOCs should be collected for every 10 drums, biased toward the area with the highest PID/FID reading. If this information does not indicate a clear maximum, the grab sample should be collected at random.

3.3 Waste Profile

Waste characterization information will be documented on a waste profile form provided by the appropriate receiving waste management facility. The signed profile will then be submitted to the disposal facility for acceptance and approval. Supporting analytical data will be provided to facilitate acceptance.

The profile typically requires the following information:

- Generator information, including name, address, contact, and phone number
- Site name, including street/mailing address
- Process generating waste (e.g., soil from monitoring well installation)
- Source of contamination (e.g., groundwater contamination)
- Historical use for area
- Waste composition (e.g., 95 percent soil, 5 percent debris)
- Physical state of waste (e.g., solid or liquid)
- Applicable hazardous waste codes

3.4 Land Disposal Restrictions

If a waste is determined to be hazardous, the applicable RCRA land disposal restriction (LDR) standards for that waste will be identified. In general, hazardous wastes that will be land disposed of must meet treatment standards (either a specified technology or a specified concentration). If applicable, LDR documents will be prepared and submitted with the profile or with the first load of waste delivered to the facility.

3.5 General Waste Management Requirements

Appropriate emergency response equipment will be provided at waste staging areas. Spill control equipment, such as sorbent pads, will be available in the storage area, and where liquids are transferred from one vessel to another.

Hazardous wastes will be segregated from nonhazardous wastes to assist with disposal pickups by grouping hazardous waste containers together and nonhazardous waste containers together. Additionally, incompatible wastes (for example, flammable and corrosive wastes) will be segregated.

All containers will be inspected upon arrival for signs of contamination and/or deterioration. Any container(s) arriving with contents, contaminated, or in poor condition will be rejected and documented.

3.5.1 Containers

The following guidelines relate to drums and small containers:

- Liquids and wet soil waste must be placed into new, UN-approved, 55-gallon steel drums or other UN-approved containers, and properly labeled. All waste containers must be placed in an appropriate staging area.
- Appropriate drums for soil or water include 1A1 or 1A2 (steel open head or steel closed head drums). The waste coordinator should be contacted before using any other drum type.
- Smaller containers must be UN-approved with securely fastening lids.
- At least 8 to 10 inches of free board must be left in each drum/container to allow for contracting and expanding – they should not be filled to the top.
- All drums/containers must be wiped down on the outside after use.
- Waste in damaged containers will be transferred to undamaged containers.
- Pallets are not required but may be used at the discretion of the field team.
- Drums/containers containing liquid waste must be placed in secondary containment.
- Adequate aisle space (e.g., 30 inches) must be provided for containers such as the 55-gallon drums to allow the unobstructed movement of personnel and equipment. A row of drums should be no more than two drums wide.
- Containers must be compatible with the waste to be placed into them.
- Drums/containers must remain completely closed with lids, covers, bolts, and locking mechanisms engaged, as though ready for immediate transport, except when removing or adding waste to the drum.
- No waste container should be placed within 50 feet of a water course or storm drain.

The following guidelines relate to roll-off boxes:

- Roll-off boxes will be inspected and accepted by Jacobs prior to the driver leaving the site. Roll-off boxes with holes, gashes, remnant soil, or debris will not be accepted, and the driver will be required to remove the roll-off box and return with another box that is acceptable to Jacobs.
- Boxes for contaminated waste will be provided with covers, bows, and disposable liners. Liners will be disposed of as contaminated debris.
- When not adding or removing waste, covers will be properly secured.
- Covers will be properly secured at the end of each workday.
- Old labels will be removed, and each box will be provided with its own label, which will be placed in a visible location. Multiple labels may be used for full visibility.
- Boxes will be inspected by the transporter after removing the liner and decontaminated in the event of evidence of liner failure.
- Free liquids, other than an incidental amount of liquid, may not be added to waste in a roll-off box.
- Saturated soil will be placed in a sludge box with sealable containment or a container otherwise designed for free liquids (such as a dewatering box, sludge box, or vacuum box).

- Boxes will be filled until half-full, or otherwise monitored to verify that they will meet DOT weight restrictions.
- Boxes will not be located near a stormwater inlet or conveyance.
- Boxes that contain free liquids will be provided with secondary containment.
- Roll-off containers will be inspected by the transporter after removal of the liner and decontaminated in the event of evidence of liner failure.

3.5.2 Spill Kits

Spill response kits containing absorbent pads for oil and aqueous liquids will be maintained when sampling activities are performed so they can be easily accessed in the event of a leak from equipment or waste containers. Smaller kits will be maintained in construction support vehicles, if needed.

Spill kits will contain the following at a minimum:

- Absorbent pad(s)
- Appropriate PPE, including gloves and goggles
- Shovels
- 55-gallon drums, storage totes, or other containers for spill-related wastes

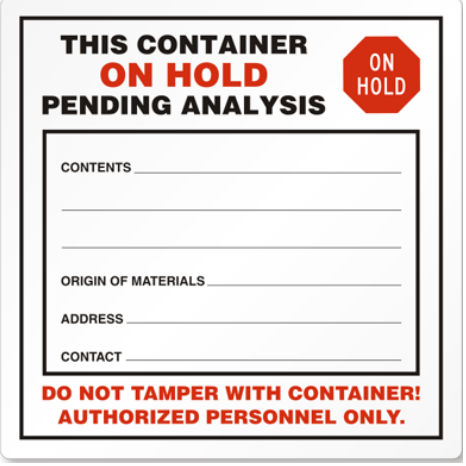
3.5.3 Waste Accumulation Time Limit

Nonhazardous waste will be removed from the site as soon as practical. Hazardous waste will be removed within 90 days of the accumulation start date. The accumulation start date for each container is the date that waste is first placed in the container.

3.5.4 Labels

Containers and portable tanks used to store/accumulate waste will initially be labeled as follows:

- **"Pending Analysis"** – This is a temporary label used until sediment and waste characterization analytical results are received and reviewed, and the waste characterized as hazardous or nonhazardous.



Once the waste is characterized, this label will be replaced with one of the following labels:

- **"Non-Hazardous Waste"** labels with the following information:
  - Generator name, contact name, and phone number
  - Waste-specific information (petroleum-contaminated soil or petroleum-contaminated water)

OR

- **"Hazardous Waste"** labels with the following information:
  - Accumulation start date (first date waste is put in drum)
  - EPA hazardous waste number(s)
  - Hazardous characteristics (ignitable, toxic, corrosive, or reactive)
  - Generator's name, address, and phone number
  - Generator's EPA ID number
  - Manifest tracking number
  - DOT proper shipping name
  - DOT ID number

3.5.5 Inspection of Waste Storage Areas

Waste storage areas will be inspected at least weekly for conditions that could result in a release of waste to the environment or the presence of hazardous wastes. Inspections will focus on conditions such as equipment malfunction, container or containment deterioration, and signs of leakage or discharge. Specifically, containers will be inspected for leaks, signs of corrosion, or signs of general deterioration.

Deficiencies observed or noted during inspection will be corrected immediately and documented. Appropriate measures may include transferring waste from a leaking container to a new container, replacing the liner or cover, or repairing the containment berm.

Inspections will be recorded in the project logbook and on a dedicated inspection form. Deficiencies and corrections will be noted and documented.

3.5.6 Training

Field personnel managing hazardous or potentially hazardous waste will meet the hazardous waste generator training requirements of 40 CFR 265.16 through the following:

- U.S. Occupational Safety and Health Administration 1910.120 Hazardous Waste Operations and Emergency Response training, including the Remediation Waste Management Module from the internal Jacobs class.
- On-the-job training that includes the following:
  - Site-specific HSP review – each site worker and guests must review and sign the plan
  - Activity hazard analysis and daily "tailgate" meetings
  - Project-specific review of this Waste Management Plan

3.6 Spill Prevention and Control

Per CERCLA Section 101(22), the definition of release includes "spilling, leaking, pumping, pouring, emitting, emptying, discharging, injecting, escaping, leaching, dumping, or disposing into the environment, including the abandonment or discarding of barrels, containers, and other closed containers" of a chemical, substance, and/or material. Procedures and controls regarding spills can be found in the site-specific HSP. Releases of wastes or chemicals will be reported in accordance with the emergency response plan, provided within the HSP.

Measures to contain spills and/or protect the ground surface will be deployed during the investigation as needed to minimize the possibility of environmental impacts from spills. These controls are required when the following occurs:

- To support equipment without built-in containment that is operating or idling (such as compressors and generators)
- Under heavy- and light-duty parked equipment operating or idling over 15 minutes (such as drilling rig, generators, forklifts, and trucks)
- For waste containers (drums, bins, tanks, and totes)

3.6.1 Spill Response

Spill response procedures include the following:

- Immediately warn nearby workers and shut down site operations
- Assess the spill area to determine if it is safe to respond
- Evacuate site personnel area if spill poses an immediate health endangerment; the site safety and health officer will designate an evacuation assembly point before beginning work
- Dial 911 if the size or nature of spill warrants professional emergency response
- Immediately eliminate nearby ignition sources
- Stop the source of the spill
- Establish site control for the spill area
- Contain and control spilled material through use of sorbent booms, pads, or other means
- Use proper PPE in responding to spills

3.6.2 Spill Cleanup and Removal

Spilled or released material will be contained immediately if safe and as appropriate for the spilled medium (liquid or solid), and the area cleaned up to the extent practicable. Containerized material will be

characterized and managed in accordance with waste management procedures (Section 3.1). Following spill cleanup, the cause of the spill will be investigated, and material storage and handling procedures will be reviewed and revised where appropriate.

3.6.3 Good Housekeeping

A clean and orderly work area reduces the possibility of accidental spills caused by mishandling chemicals and equipment and reduces safety hazards. The following practices will be incorporated into the field activities as part of good housekeeping practices:

- Garbage, waste materials, and construction debris will be regularly picked up and disposed of.
- The site will be maintained in an orderly condition.
- Materials will be kept in their original containers whenever possible, maintaining original labels and safety datasheets.
- Surplus materials will be properly disposed of.
- Equipment will be positioned so that items such as valves, tanks, and lines are protected from damage during ongoing operations.

3.6.4 Preventive Maintenance

Preventive maintenance involves the regular inspection and testing of equipment and operational systems. Breakdowns and failures can often be avoided by adjustment, repair, or replacement of equipment.

The following practices will be adhered to as part of preventive maintenance:

- Equipment will be inspected before it enters site and then daily thereafter. Inspections will focus on active or potential leaks, as well as faulty hoses and fittings. Inspections will be documented in the daily logs. Equipment requiring maintenance or repair will not be placed into service.
- Equipment being used will be maintained in accordance with the manufacturer's specifications. Maintenance will occur offsite if possible. Regular equipment maintenance will not be performed onsite.
- Before using or moving a vehicle or piece of equipment, a 360-degree walk-around will be performed. This review will include checking for the presence of leaks on the ground, under the vehicle or equipment, and in secondary containment associated with the vehicle or equipment.

4. Recordkeeping

The following records and documents will be maintained:

- A waste tracking log that contains the following minimum information for each container as it is generated will be kept separately from the project logbook:
  - A specific ID number corresponding to each container generated at the site (the container will be marked with the same ID)
  - The date that waste was first placed in the container (accumulation date)
  - The profile number associated with the waste
  - The date that the container was transported for final disposal and the name of the transporter moving the container
  - Manifest number (offsite disposal only)
- Transportation and offsite disposal records, including the following:
  - Signed profiles and associated characterization data
  - Initial manifests (generator and transporter)
  - Facility-signed manifests
  - Weight tickets
  - LDR documentation
  - Certificates of destruction
- Training records
- Inspection records

All records will be retained for 10 years following completion of the remedial action.

5. References

U.S. Environmental Protection Agency (EPA). 2017. *Record of Decision Portland Harbor Superfund Site, Portland Oregon*. Region 10. Seattle, Washington. January.

U.S. Environmental Protection Agency (EPA). 2020. *Off-Site Rule.* Superfund. https://www.epa.gov/superfund/site-rule.