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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Portland Harbor Superfund Site Surface Sediment – Stratified Random
Laboratory:	ALS Environmental, Kelso, WA
Laboratory Group:	K1804531
Analyses/Method:	Chlorinated Pesticides, Tributyltin, Polycyclic Aromatic Hydrocarbons (PAHs), bis(2-Ethylhexyl)phthalate, and Total Solids
Validation Level:	Stage 2A
AECOM Project Number:	60566335 Task #2.12
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SUMMARY

The data quality review of 2 surface sediment samples collected on May 11 and May 13, 2018, has been completed. The samples were analyzed for chlorinated pesticides by EPA Method 1699-modified (GC/MS/MS), tributyltin by Krone et al., PAHs by EPA Method 8270D modified by selected ion monitoring (SIM), bis(2-ethylhexyl)phthalate by EPA Method 8270D, and total solids by EPA Method 160.3-modified at ALS Environmental (ALS) located in Kelso, Washington. The analyses were performed in general accordance with the methods specified in EPA's *Test Methods for Evaluating Solid Waste (SW-846)*, *Methods for Chemical Analysis of Water and Wastes*, March 1983, and *Method 1699: Pesticides in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS*, December 2007 (modified by ALS SOP SVM-PESTMS2), and Krone CA et al., *A Method for Analysis of Butyltin Species and Measurement of Butyltins in Sediment and English Sole Livers from Puget Sound*, Environmental Conservation Division, Northwest and Alaska Fisheries Center, National Marine Fisheries Service, NOAA, November 1988. The laboratory provided level 2 and level 4 data packages containing sample results and associated quality assurance (QA) and quality control (QC) data, preparation logs, and raw instrument outputs (where applicable). The following samples are associated with laboratory group K1804531:

Sample ID	Laboratory ID
PDI-SG-B077-BL1	K1804531-001
PDI-SG-B380-BL1	K1804531-002

Data validation is based on method performance criteria and QC criteria documented in the *Quality Assurance Project Plan (QAPP),* dated March 23, 2018, as amended. If data qualification was required, data were qualified based on the definitions and use of qualifying flags outlined in the EPA documents *USEPA National Functional Guidelines for Organic Superfund Methods Data Review*, January 2017, and *USEPA National Functional Guidelines for Inorganic Superfund Methods Data Review,* January 2017. Data qualifiers assigned to this sample set are included in Table 1.

SAMPLE RECEIPT

Upon receipt by ALS, the sample jar information was compared to the chain-of-custody (COC) and the cooler temperature was recorded. No discrepancies related to sample identification were noted by ALS, and the cooler was received at a temperature within the EPA-recommended limits of greater than 0°C and less than or equal to 6°C.

ORGANIC ANALYSES

Samples were analyzed for chlorinated pesticides, tributyltin, PAHs, and bis(2-ethylhexyl)phthalate, by the methods identified in the introduction to this report.

1. Holding Times – Acceptable

2. Initial and Continuing Calibration Verifications – Acceptable

   Chlorinated Pesticides by EPA Method 1699-modified – The percent difference for heptachlor (-46.1%) was below the control limit of ±25% in the continuing calibration verification (CCV) analyzed on June 24, 2018. Heptachlor was qualified as estimated and flagged 'UJ' in PDI-SG-B057-BL1 and PDI-SG-B380-BL1 based on this CCV result.

3. Blanks – Acceptable except as noted below:

   General – Two rinsate blanks were collected on May 11, and May 17, 2018, were reported with laboratory groups K1804532 (ID K1804532-031) and K1804768 (ID K1804768-003) and are applicable to the samples collected in this laboratory group. One or more analytes were detected in the rinsate blanks and one or more of these analytes may have been qualified as not detected based on the associated method blank results. Refer to the associated data validation memorandum for further information. Sediment data were not qualified based on rinsate blank detections.

4. Surrogates – Acceptable

5. Internal Standards – Acceptable where applicable

6. Laboratory Control Sample (LCS) – Acceptable except as noted below:

   Chlorinated Pesticides by EPA Method 1699-modified – The percent recoveries for cis-nonachlor (140%) and heptachlor (57%) were outside the control limits of 69-134% and 81-114%, respectively, in the LCS extracted on May 18, 2018. cis-Nonachlor was not detected in the associated samples; therefore, data were not qualified based on this LCS result. The results for heptachlor in the associated samples were qualified based on the associated CCV result as described in Section 2; therefore, no further qualification based on the LCS recovery was necessary.

7. Matrix Spike/Matrix Spike Duplicate (MS/MSD) – Acceptable except as noted below:

   Chlorinated Pesticides by EPA Method 1699-modified – An MS/MSD was performed using PDI-SG-S254-BL1 (reported in laboratory group K1804532, discussed under separate cover). Data in this laboratory group were not qualified based on these MS/MSD results. Qualification, if any, is discussed in the associated data validation report.

   Tributyltin by Krone et al. – An MS/MSD was performed using PDI-SG-B077-BL1. Results were acceptable.

   PAHs by EPA Method 8270D-SIM – An MS/MSD was performed using PDI-SG-S254-BL1 (reported in laboratory group K1804532, discussed under separate cover). Data in this

laboratory group were not qualified based on these MS/MSD results. Qualification, if any, is discussed in the associated data validation report.

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – An MS/MSD was not performed in association with this analysis due to insufficient sample volume. Accuracy was assessed using the LCS. Precision was not assessed for this analyte.

8. Reporting Limits– Acceptable except as noted below:

General – One or more results were flagged 'J' by the laboratory to indicate the reported concentrations were above the MDLs but below the reporting limits. Laboratory 'J'-flagged results are considered estimated. As the result is between the MDL and the reporting limit, there is a greater level of uncertainty associated with the numerical result.

Chlorinated Pesticides by EPA Method 1699-modified – The reporting limits for one or more pesticides reported as not detected in these samples were elevated due to the moisture content and/or dilution due to matrix interference. The reporting limits and MDLs for dieldrin exceeded the cleanup level in both sediment samples in laboratory group K1804531.

CONVENTIONAL ANALYSES

Sediment samples were analyzed for total solids by EPA Method 160.3-modified.

1. Holding Times – Acceptable

2. Laboratory Duplicate – Acceptable

A laboratory duplicate was performed using PDI-SG-B077-BL1. The result was comparable.

3. Reporting Limits – Acceptable

OVERALL ASSESSMENT OF DATA

The data reported in this laboratory group is considered usable for meeting project objectives. The completeness for laboratory group K1804531 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Surface Sediment - Stratified Random
ALS Kelso Laboratory Group: K1804531

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B077-BL1	K1804531-001	CWA1699M	Heptachlor	0.66 U	ug/kg	0.66 UJ	c
PDI-SG-B380-BL1	K1804531-002	CWA1699M	Heptachlor	0.46 U	ug/kg	0.46 UJ	c

c - calibration issue

J - estimated value

ug/kg - microgram per kilogram

U - Compound was analyzed for, but not detected above the value shown.