

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: K1804982

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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File Name: K1804982 DVR

SUMMARY

The data quality review of 3 surface sediment samples collected between April 28 and April 30, 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 K1804982:

Sample ID	Laboratory ID
PDI-SG-B405-BL1	K1804982-001
PDI-SG-B409-BL1	K1804982-002
PDI-SG-B414-BL1	K1804982-003

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 cooler temperatures within the EPA-recommended limits of

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greater than 0°C and less than or equal to 6°C. All the samples reported in this laboratory group were frozen at the field warehouse after sampling until they were shipped to ALS on May 25, 2018. When they were received by the laboratory, the samples were kept frozen and placed on hold. On June 7, 2018, all samples were released by AECOM for analysis. The laboratory kept the samples frozen until extraction.

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 except as noted below:

General – The samples reported in this laboratory group were extracted several days past the method-recommended holding time of 14 days after sample collection. As described under Sample Receipt, the samples were frozen in archive at the AECOM warehouse prior to shipment to ALS-Kelso, and were frozen in archive upon receipt by the laboratory. The samples were thawed for less than 14 days; therefore, the samples were not extracted outside the holding times.

2. Initial and Continuing Calibration Verifications – Acceptable except as noted below:

Chlorinated Pesticides by EPA Method 1699-modified – The percent differences for cis-nonachlor (47.4%) and gamma-chlordane (26.5%) exceeded the control limit of $\pm 25\%$ in the continuing calibration verification (CCV) analyzed on July 17, 2018. cis-Nonachlor and gamma-chlordane were not detected in the associated sample; therefore, data were not qualified based on these CCV results.

3. Blanks – Acceptable except as noted below:

General – Two rinsate blanks were collected on April 29, and May 2, 2018, were reported with laboratory groups K1803975 (ID K1803975-035) and K1804181 (ID K1804181-028) 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 were qualified as not detected based on the associated method blank results. Refer to the associated data validation memoranda 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 the following analytes in the LCSs noted below were outside the control limits:

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Extraction Date	Analyte	LCS	Control limit
July 2, 2018	4,4'-DDT	120%	78-119%
	cis-Nonachlor	223%	69-134%
July 25, 2018	2,4'-DDT	76%	77-118%
	cis-Nonachlor	169%	69-134%

4,4'-DDT and cis-nonachlor were not detected in the sample associated with the LCS extracted on July 2, 2018; therefore, data were not qualified based on the LCS results.

cis-Nonachlor was not detected in the samples associated with the LCS extracted on July 25, 2018; therefore, data were not qualified based on this LCS result. The results for 2,4'-DDT in PDI-SG-B405-BL1 and PDI-SG-B414-BL1 were qualified as estimated and flagged 'UJ' based on this LCS result.

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-B403-BL1 (reported in laboratory group K1804798, 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-B409-BL1. Results were acceptable.

PAHs by EPA Method 8270D-SIM – An MS/MSD was performed using PDI-SG-S204 (reported in laboratory group K1804985, 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 performed using PDI-SG-B409-BL1. Results were acceptable.

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 all sediment samples in laboratory group K1804982.



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CONVENTIONAL ANALYSIS

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

1. Holding Times – Acceptable except as noted below:

The 7-day holding time indicated for total solids in the QAPP was exceeded by 52 to 54 days for samples PDI-SG-B405-BL1, PDI-SG-B409-BL1, and PDI-SG-B414-BL1. As described under Sample Receipt, the samples were frozen in archive at the AECOM warehouse prior to shipment to ALS-Kelso, and were frozen in archive upon receipt by the laboratory before analyses were released by AECOM for analysis. No data qualifiers were assigned based on the holding time exceedance.

2. Laboratory Duplicate – Acceptable

Laboratory duplicates were performed using PDI-SG-S027 (reported in laboratory group K1806088, reported under separate cover) and two samples from projects unrelated to Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling. Results were 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 K1804982 is 100%.

Table 1
QA/QC Data Summary Review
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Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B405-BL1	K1804982-001	CWA1699M	2,4-DDT	0.64 U	ug/kg	0.64 UJ	I
PDI-SG-B414-BL1	K1804982-003	CWA1699M	2,4-DDT	0.76 U	ug/kg	0.76 UJ	I

J - estimated value

I - laboratory control sample recovery

ug/kg - microgram per kilogram

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