

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: K1807194

Analyses/Method: Chlorinated Pesticides, Tributyltin, Polycyclic Aromatic Hydrocarbons (PAHs),
bis(2-Ethylhexyl)phthalate, and Total Solids

Validation Level: Stage 2A

AECOM Project

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File Name: K1807194 DVR

SUMMARY

The data quality review of 2 surface sediment samples collected on May 23 and June 19, 2018, has been completed. 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/or total solids by EPA Method 160.3-modified at ALS Environmental (ALS) located in Kelso, Washington as described in the cross-reference below. The analyses were performed in general accordance with the methods specified in EPA's *Test Methods for Evaluating Solid Waste (SW-846)*, *Method 1699: Pesticides in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS*, December 2007 (modified by ALS SOP SVM-PESTMS2), and *Methods for Chemical Analysis of Water and Wastes*, March 1983, and/or 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 K1807194:

Sample ID	Laboratory ID	Requested Analyses
PDI-SG-B420-BL1	K1807194-001	Chlorinated Pesticides, Tributyltin, PAHs, bis(2-Ethylhexyl)phthalate
PDI-SG-B423-BL1	K1807194-002	Chlorinated Pesticides

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 results reported in 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. The sample identification for PDI-SG-B423-BL1 was incorrectly recorded on the sample label; however, the correct identification was recorded on the

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sample container lid. The laboratory identified the sample using the identification on the sample lid through the process of elimination. The cooler was received at a temperature below the EPA-recommended limits of greater than 0°C and less than or equal to 6°C at -0.5°C. The laboratory did not indicate that any sample containers were received broken; therefore, data were not qualified based on the low cooler temperature. The samples in this laboratory group were frozen after sample collection and stored at the AECOM field warehouse until they were shipped to ALS on August 1, 2018.

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 – Samples analyzed for chlorinated pesticides, tributyltin, PAHs, and bis(2-ethylhexyl)phthalate were extracted between 31 and 71 days past the method-recommended holding time of 14 days after sample collection. The samples were frozen immediately after sample collection, and held in archive in the AECOM field warehouse before shipment to ALS. The samples were received by ALS on August 1, 2018. Per ALS-Kelso protocol, the samples were frozen in archive until extraction and were thawed for less than 14 days; therefore, the samples were not extracted outside the holding time.

2. Initial and Continuing Calibration Verifications – Acceptable

Tributyltin by Krone et al. – The percent difference (%D) for the surrogate tri-n-propyltin (-25.6%) on one of the analytical columns for the continuing calibration verification (CCV) analyzed on September 5, 2018, was below the method limits of $\pm 25\%$. Data were not qualified based on surrogate recoveries in CCVs.

3. Blanks – Acceptable

A rinsate blank was not submitted with this laboratory group. Associated rinsate blanks are reported under separate cover. Target compounds may have been detected in the rinsate blanks associated with these samples. Data were not qualified based on rinsate blank results.

4. Surrogates – Acceptable

5. Internal Standards – Acceptable where applicable

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

Chlorinated Pesticides by EPA Method 1699-modified – The percent recoveries for the following analytes in the LCS extracted on August 3, 2018, were outside the control limits:

Analyte	LCS	Control limit
2,4'-DDT	73%	77-118%
cis-Nonachlor	156%	69-134%

cis-Nonachlor was reported as not detected in the associated samples; therefore, no data

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were qualified for cis-nonachlor based on this LCS result. The results for 2,4'-DDT in PDI-SG-B420-BL1 and PDI-SG-B423-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-S228 (laboratory group K1807188, 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-S155 (laboratory group K1807188 discussed under separate cover). Results were acceptable.

PAHs by Method 8270D-SIM – An MS/MSD was performed using PDI-SG-S155 (laboratory group K1807188, 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 Method 8270D – An MS/MSD was performed using PDI-SG-B420-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 method detection limits (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 multiple samples were elevated due to lower extraction volume due to matrix interference. The reporting limits and MDLs for dieldrin exceeded the cleanup level in all sediment samples reported in laboratory group K1807194.

Tributyltin by Krone et al. – The reporting limits for tributyltin reported as not detected in multiple samples were elevated due to moisture content. The elevated reporting limits and MDLs do not exceed the cleanup level.

CONVENTIONAL ANALYSES

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

1. Holding Times – Acceptable

Samples PDI-SG-B420-BL1 and PDI-SG-B423-BL1 were frozen and held in archive at the AECOM storage facility immediately after collection. The frozen samples were submitted to ALS on August 1, 2018, and the total solids analyses were performed on August 2, 2018, 37 to 64 days past the 7-day holding time indicated for total solids in the QAPP. No data qualifiers were assigned based on the holding time exceedance.



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2. Laboratory Duplicate – Acceptable

A laboratory duplicate was performed using PDI-SG-B420-BL1. 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 K1807194 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Surface Sediment - Stratified Random
ALS Kelso Laboratory Group: K1807194

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B420-BL1	K1807194-001	CWA1699M	2,4-DDT	0.21 U	ug/kg	0.21 UJ	I
PDI-SG-B423-BL1	K1807194-002	CWA1699M	2,4-DDT	0.21 U	ug/kg	0.21 UJ	I

Notes:

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.