

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: K1805746

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: K1805746 DVR

SUMMARY

The data quality review of 9 surface sediment samples and one rinsate blank collected between June 15 and June 17, 2018, has been completed. Samples were analyzed for chlorinated pesticides by EPA Method 1699-modified (GC/MS/MS), tributyltin by Unger 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. 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 Unger, MA et al., *Determination of Butyltins in Natural Waters by Flame Photometric Detection of Hexane Derivatives and Mass Spectrometric Confirmation*, *Chemosphere*, 1886,16(4):461-470. 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 K1805746:

Sample ID	Laboratory ID
PDI-SG-B301-BL1	K1805746-001
PDI-SG-B297-BL1	K1805746-002
PDI-SG-B293-BL1	K1805746-003
PDI-SG-B310-BL1	K1805746-004
PDI-SG-B309-BL1	K1805746-005
PDI-SG-B314-BL1	K1805746-006
PDI-SG-B030-BL1	K1805746-007
PDI-SG-B031-BL1	K1805746-008
PDI-SG-B042-BL1	K1805746-009
PDI-RB-VV-20180616 (rinsate blank)	K1805746-010

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*,



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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 temperatures were recorded. No discrepancies related to sample identification were noted by ALS. The coolers were received at temperatures below the EPA-recommended limits of greater than 0°C and less than or equal to 6°C at -0.3°C and -0.7°C. The laboratory did not indicate that any of the samples were frozen and all sample containers were intact; therefore, data were not qualified based on the low cooler temperatures.

ORGANIC ANALYSES

Sediment samples were analyzed for chlorinated pesticides and the rinsate blank was 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:

Chlorinated Pesticides by EPA Method 1699-modified – The sediment samples reported in this laboratory group were extracted 32-34 days past the method-recommended holding time of 14 days after sample collection. 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

PAHs by EPA Method 8270D-SIM – The percent differences (%Ds) for dibenz(a,h)anthracene (-21%) and benzo(g,h,i)perylene (-27%) were outside the control limits of $\pm 20\%$ in the CCV analyzed on June 20, 2018. The results for dibenz(a,h)anthracene and benzo(g,h,i)perylene in PDI-RB-VV-20180616 were qualified as estimated and flagged 'UJ' based on these CCV results.

3. Blanks – Acceptable except as noted below:

General – Two rinsate blanks were collected on June 16 and June 21, 2018, were reported with this laboratory group (ID K1805746-010) and laboratory group K1805921 (ID K1805921-004), and are applicable to the samples collected in this laboratory group. One or more analytes were detected in the rinsate blank collected on June 21, 2018. Refer to the associated data validation memorandum for further information.

The following analytes were detected at concentrations between the method detection limits (MDLs) and the reporting limits in the rinsate blank reported in this laboratory group.

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Analysis	Analyte	Result
Chlorinated Pesticides	4,4'-DDT	0.30 ng/L
	Heptachlor	0.18 ng/L
PAHs	Naphthalene	0.0020 ug/L
	Phenanthrene	0.0024 ug/L
	Pyrene	0.0013 ug/L
	Benz(a)anthracene	0.0024 ug/L
bis(2-Ethylhexyl)phthalate	bis(2-Ethylhexyl)phthalate	0.44 ug/L

The result for benz(a)anthracene in PDI-RB-VV-20180616 was qualified as not detected based on the method blank as described below. Sediment data were not qualified based on rinsate blank detections.

PAHs by EPA Method 8270D-SIM – Benz(a)anthracene (0.0022 ug/L) was detected at a concentration between the MDL and the reporting limit in the method blank extracted on June 19, 2018. The result for benz(a)anthracene in PDI-RB-VV-20180616 was reported at a concentration between the MDL and reporting limit, was qualified as not detected, and was flagged 'U' at the reporting limit based on this method blank result.

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/LCSD extracted on June 22, 2018, were outside the control limits:

Analyte	LCS	LCSD	Control Limits	RPD CL = 30%
2,4'-DDD	ok	78%	79-113%	ok
2,4'-DDE	59%	57%	75-117%	ok
4,4'-DDE	68%	68%	76-115%	ok
4,4'-DDT	ok	114%	85-113%	ok
Aldrin	ok	144%	81-113%	ok
cis-Nonachlor	172%	181%	59-138%	ok
Dieldrin	ok	ok	62-111%	38%

ok – acceptable

CL – control limits

The results for 2,4'-DDE and 4,4'-DDE in PDI-RB-VV-20180616 were qualified as estimated and flagged 'UJ' based on these LCS/LCSD results. All other chlorinated pesticides listed in the table above were either reported as not detected in the associated sample or two of the three quality control parameters (LCS, LCSD, and/or relative percent difference [RPD]) were acceptable; therefore, data were not qualified for these analytes based on these LCS/LCSD results.

The percent recoveries for cis-nonachlor (172%) and trans-nonachlor (144%) in the LCS extracted on August 2, 2018, were outside the control limits of 69-134% and 76-124%, respectively. cis-Nonachlor was not detected in the associated samples; therefore, sediment data were not qualified for cis-nonachlor based on this LCS result. The result for trans-

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nonachlor in PDI-SG-B293-BL1 was qualified as estimated and flagged 'J' based on this LCS result.

PAHs by EPA Method 8270D-SIM – The percent recoveries for the following PAHs were outside the control limits in the LCS/LCSD extracted on June 19, 2018:

Analyte	LCS	LCSD	Control Limits
Benz(a)anthracene	131%	ok	74-124%
Benzo(b)fluoranthene	141%	ok	73-136%
Benzo(k)fluoranthene	136%	ok	74-134%
Benzo(a)pyrene	139%	ok	75-131%

ok - acceptable

As the percent recoveries in the LCSD and the RPDs for the LCS/LCSD pair were acceptable, no data were qualified for the analytes listed in the table above based on the elevated LCS recoveries.

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

General – MS/MSDs were not performed in association with the rinsate blank sample. Precision and accuracy were assessed using the associated LCS/LCSD results.

Chlorinated Pesticides by EPA Method 1699-modified – An MS/MSD was performed using PDI-SG-B031-BL1. The percent recoveries for cis-nonachlor in the MS (167%) and the MSD (171%) were outside the control limits of 27-144%. cis-Nonachlor was not detected in PDI-SG-B031-BL1; therefore, data were not qualified based on these MS/MSD results.

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 multiple 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 reported in laboratory group K1805746.

CONVENTIONAL ANALYSIS

Soil 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-B031-BL1. Results were comparable.

3. Reporting Limits – Acceptable



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OVERALL ASSESSMENT OF DATA

The data reported in this laboratory group is considered usable for meeting project objectives. The completeness for laboratory group K1805746 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
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Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B293-BL1	K1805746-003	CWA1699M	trans-Nonachlor	0.30 J	ug/kg	0.30 J	I
PDI-RB-VV-20180616	K1805746-010	CWA1699M	2,4-DDE	0.50 U	ng/L	0.50 UJ	I
PDI-RB-VV-20180616	K1805746-010	CWA1699M	4,4'-DDE	0.50 U	ng/L	0.50 UJ	I
PDI-RB-VV-20180616	K1805746-010	SW8270DSIM	Benz(a)anthracene	0.0024 J	ug/L	0.020 U	bl
PDI-RB-VV-20180616	K1805746-010	SW8270DSIM	Benzo(g,h,i)perylene	0.020 U	ug/L	0.020 UJ	c
PDI-RB-VV-20180616	K1805746-010	SW8270DSIM	Dibenz(a,h)anthracene	0.020 U	ug/L	0.020 UJ	c

Notes:

bl - laboratory blank contamination

c - calibration issue

J - estimated value

I - laboratory control sample recovery

ng/L - nanogram per liter

ug/kg - microgram per kilogram

ug/L - microgram per liter

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

Note: Line items where the laboratory result contains a "J" and the final result contains a "U" with a data validation reason code "bl" indicate that the final result is reported as not detected ("U" flag) at the reporting limit.