

# Data Validation Report

Project: Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling  
 Portland Harbor Superfund Site  
 Sediment Traps

Laboratory: ALS Environmental, Kelso, WA

Laboratory Group: K1810683

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: K1810683 DVR

## SUMMARY

The data quality review of 4 sediment trap samples and one rinsate blank collected on October 31, 2018, has been completed. Samples were analyzed for chlorinated pesticides by EPA Method 1699-modified (GC/MS/MS), tributyltin by Krone et al. (sediments) or Unger et al. (water), 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 Krone, CA et. al., *A Method for Analysis of Butyltin Species and Measurement of Butyltins in Sediment and English Sole Livers from Puget Sound*, NOAA, November 1988, 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 K1810683:

| Sample ID                      | Laboratory ID |
|--------------------------------|---------------|
| PDI-ST-T06B-1810               | K1810683-001  |
| PDI-ST-T06A-1810               | K1810683-002  |
| PDI-ST-T07A-1810               | K1810683-003  |
| PDI-ST-T07B-1810               | K1810683-004  |
| PDI-RB-ST-1810 (rinsate blank) | K1810683-005  |

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.

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## 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 and the coolers were received at temperatures 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 (%D) for 4,4'-DDD (-29.5%) was outside the control limits of +20% in the CCV analyzed on November 21, 2018. The result for 4,4' DDD in PDI-RB-ST-1810 was qualified as estimated and flagged 'UJ' based on this CCV result.

Tributyltin by Krone et al. – The %D for the surrogate tri-n-propyltin (-31.0%) was outside the control limits of ±25% in the CCV analyzed on December 1, 2018. Data were not qualified based on surrogate %D results in a CCV.

3. Blanks – Acceptable except as noted below:

General – One rinsate blank (PDI-RB-ST-1810) was submitted with this laboratory group. One or more analytes were detected in this rinsate blank. Sediment data were not qualified based on rinsate blank detections.

Chlorinated Pesticides by EPA Method 1699-modified – 4,4'DDE (0.082 ng/L) was detected at a concentration between the method detection limit (MDL) and the reporting limit in the method blank extracted on November 7, 2018. 4,4'DDE was not detected in the associated sample (rinsate blank); therefore, data were not qualified based on this method blank result.

PAHs by EPA Method 8270D-SIM – Benz(a)anthracene (0.035 ug/kg) was detected at a concentration between the MDL and the reporting limit in the method blank associated with the sediment samples and extracted on November 7, 2018. Benz(a)anthracene was detected in the associated samples at concentrations above the reporting limits and significantly higher than the method blank concentration; therefore, data were not qualified based on this method blank result.

The following PAHs were detected at concentrations between the MDLs and the reporting limits in the method blank associated with the rinsate blank and analyzed on November 7, 2018:

| Analyte             | Result (ug/L) |
|---------------------|---------------|
| Naphthalene         | 0.0060        |
| 2-Methylnaphthalene | 0.0048        |
| Acenaphthene        | 0.0016        |

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| Analyte            | Result (ug/L) |
|--------------------|---------------|
| Dibenzofuran       | 0.0053        |
| Fluorene           | 0.0030        |
| Phenanthrene       | 0.0085        |
| Fluoranthene       | 0.0024        |
| Pyrene             | 0.0020        |
| Benzo(a)anthracene | 0.0023        |

The PAHs listed in the above table were reported between the MDLs and reporting limits in PDI-RB-ST-1810, qualified as not detected, and flagged 'U' at the reporting limits based on these method blank results.

4. Surrogates – Acceptable
5. Internal Standards – Acceptable where applicable
6. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) – Acceptable
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.

An MS/MSD was performed using PDI-ST-T06A-1810 for all sediment analyses. Results were acceptable except as noted below.

PAHs by EPA Method 8270D-SIM – The percent recoveries for the following analytes in the MS/MSD were outside the control limits of 70-130%:

| Analyte              | MS  | MSD |
|----------------------|-----|-----|
| Naphthalene          | 48% | 44% |
| 2-Methylnaphthalene  | 58% | 53% |
| Acenaphthylene       | 66% | 58% |
| Acenaphthene         | 66% | 58% |
| Dibenzofuran         | 66% | 59% |
| Fluorene             | ok  | 64% |
| Phenanthrene         | ok  | 62% |
| Anthracene           | ok  | 68% |
| Fluoranthene         | ok  | 58% |
| Pyrene               | ok  | 68% |
| Benzo(b)fluoranthene | ok  | 69% |
| Benzo(g,h,i)perylene | 69% | 63% |

ok – acceptable

The percent recoveries in the MS and the relative percent differences (RPDs) for the MS/MSD pair were acceptable for fluorene, phenanthrene, anthracene, fluoranthene, pyrene, and benzo(b)fluoranthene; therefore, data were not qualified for these analytes based on these MS/MSD results. The results for naphthalene, 2-methylnaphthalene, acenaphthylene, acenaphthene, dibenzofuran, and benzo(g,h,i)perylene in PDI-ST-T06A-1810 were qualified as estimated and flagged 'J' based on these MS/MSD results.



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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 K1810683.

PAHs by EPA Method 8270D-SIM – The results for acenaphthylene in PDI-ST-T06B-1810, PDI-ST-T06A-1810, PDI-ST-T07A-1810, and PDI-ST-T07B-1810 were flagged ‘X’ by the laboratory to indicate poor peak resolution. The result for acenaphthylene in PDI-ST-T06A-1810 was qualified as estimated based on MS/MSD results as described in Section 7; therefore, no further qualification based on the poor peak resolution was required. The results for acenaphthylene in PDI-ST-T06B-1810, PDI-ST-T07A-1810, and PDI-ST-T07B-1810 were qualified as estimated and flagged ‘J’ due to poor peak resolution.

**CONVENTIONAL ANALYSIS**

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-ST-T06A-1810. 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 K1810683 is 100%.

**Table 1**  
**QA/QC Data Summary Review**  
**Portland Harbor**  
**Sediment Traps**  
**ALS Kelso Laboratory Group: K1810683**

| Sample ID        | Laboratory ID | Method     | Analyte              | Laboratory Result | Units | Final Result | Reason Code |
|------------------|---------------|------------|----------------------|-------------------|-------|--------------|-------------|
| PDI-ST-T06B-1810 | K1810683-001  | SW8270DSIM | Acenaphthylene       | 2.0 X             | ug/kg | 2.0 J        | q           |
| PDI-ST-T06A-1810 | K1810683-002  | SW8270DSIM | Naphthalene          | 2.9               | ug/kg | 2.9 J        | m           |
| PDI-ST-T06A-1810 | K1810683-002  | SW8270DSIM | 2-Methylnaphthalene  | 1.9 J             | ug/kg | 1.9 J        | m           |
| PDI-ST-T06A-1810 | K1810683-002  | SW8270DSIM | Acenaphthylene       | 2.0 X             | ug/kg | 2.0 J        | m           |
| PDI-ST-T06A-1810 | K1810683-002  | SW8270DSIM | Acenaphthene         | 2.2               | ug/kg | 2.2 J        | m           |
| PDI-ST-T06A-1810 | K1810683-002  | SW8270DSIM | Dibenzofuran         | 2.0               | ug/kg | 2.0 J        | m           |
| PDI-ST-T06A-1810 | K1810683-002  | SW8270DSIM | Benzo(g,h,i)perylene | 18                | ug/kg | 18 J         | m           |
| PDI-ST-T07A-1810 | K1810683-003  | SW8270DSIM | Acenaphthylene       | 1.2 X             | ug/kg | 1.2 J        | q           |
| PDI-ST-T07B-1810 | K1810683-004  | SW8270DSIM | Acenaphthylene       | 1.9 X             | ug/kg | 1.9 J        | q           |
| PDI-RB-ST-1810   | K1810683-005  | CWA1699M   | 4,4'-DDD             | 0.50 U            | ng/L  | 0.50 UJ      | c           |
| PDI-RB-ST-1810   | K1810683-005  | SW8270DSIM | Naphthalene          | 0.0088 J          | ug/L  | 0.020 U      | bl          |
| PDI-RB-ST-1810   | K1810683-005  | SW8270DSIM | 2-Methylnaphthalene  | 0.0064 J          | ug/L  | 0.020 U      | bl          |
| PDI-RB-ST-1810   | K1810683-005  | SW8270DSIM | Acenaphthene         | 0.0026 J          | ug/L  | 0.020 U      | bl          |
| PDI-RB-ST-1810   | K1810683-005  | SW8270DSIM | Dibenzofuran         | 0.0063 J          | ug/L  | 0.020 U      | bl          |
| PDI-RB-ST-1810   | K1810683-005  | SW8270DSIM | Fluorene             | 0.0049 J          | ug/L  | 0.020 U      | bl          |
| PDI-RB-ST-1810   | K1810683-005  | SW8270DSIM | Phenanthrene         | 0.013 J           | ug/L  | 0.020 U      | bl          |
| PDI-RB-ST-1810   | K1810683-005  | SW8270DSIM | Fluoranthene         | 0.0020 J          | ug/L  | 0.020 U      | bl          |
| PDI-RB-ST-1810   | K1810683-005  | SW8270DSIM | Pyrene               | 0.0021 J          | ug/L  | 0.020 U      | bl          |
| PDI-RB-ST-1810   | K1810683-005  | SW8270DSIM | Benz(a)anthracene    | 0.0020 J          | ug/L  | 0.020 U      | bl          |

**Notes:**

- bl - laboratory blank contamination
- c - calibration issue
- J - estimated value
- m - matrix spike 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.
- q - quantitation issue

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.