

# Data Validation Report

Project: Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling  
 Portland Harbor Superfund Site  
 Subsurface Sediment – Deep/Nearshore Cores

Laboratory: ALS Environmental, Burlington, Ontario, Canada

Laboratory Group: L2161800

Analyses/Method: Chlorinated Pesticides and Total Solids

Validation Level: Stage 2

AECOM Project

Number: 60566335 Task #2.12

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File Name: L2161800 DVR

## SUMMARY

The data quality review of 17 subsurface sediment samples, one field duplicate, and three rinsate blanks collected between September 6 and September 7, 2018, has been completed. Samples were analyzed for chlorinated pesticides by EPA Method 1699-modified (GC/HRMS) and/or total solids by American Society for Testing and Materials (ASTM) Method D-2974 at ALS Environmental (ALS) located in Burlington, Ontario, Canada. The analyses were performed in general accordance with the methods specified in *Method 1699: Pesticides in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS*, December 2007 (modified by ALS SOP BU-TM-1103 v07 OCP), and *Annual Book of ASTM Standards*, American Society for Testing & Materials (ASTM), Philadelphia, Pennsylvania. 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 L2161800:

| Sample ID              | Laboratory ID |
|------------------------|---------------|
| PDI-SC-S113C-0TO1.1    | L2161800-01   |
| PDI-SC-S113C-1.1TO3.1  | L2161800-02   |
| PDI-SC-S113C-3.1TO5.6  | L2161800-03   |
| PDI-SC-S113C-5.6TO6.6  | L2161800-04   |
| PDI-SC-S260-0TO1.3     | L2161800-05   |
| PDI-SC-S260-1.3TO2.6   | L2161800-06   |
| PDI-SC-S260-2.6TO4.2   | L2161800-07   |
| PDI-SC-S260-4.2TO6     | L2161800-08   |
| PDI-SC-S260-6TO7       | L2161800-09   |
| PDI-SC-S019-0TO2       | L2161800-10   |
| PDI-SC-S019-2TO4       | L2161800-11   |
| PDI-SC-S019-4TO6       | L2161800-12   |
| PDI-SC-S019-6TO8       | L2161800-13   |
| PDI-SC-S019-8TO10      | L2161800-14   |
| PDI-SC-S019-10TO12     | L2161800-15   |
| PDI-SC-S019-12TO13.7   | L2161800-16   |
| PDI-SC-S019-13.7TO14.7 | L2161800-17   |

**Data Validation Report**  
**Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling**  
**Subsurface Sediment – Deep/Nearshore Cores**  
**ALS Lab Group: L2161800**

| Sample ID  | Laboratory ID |
|--|---------------|
| PDI-SC-S019-10TO12D<br>(Duplicate of PDI-SC-S019-10TO12) | L2161800-18   |
| PDI-RB-SS-180906 (rinsate blank)                         | L2161800-19   |
| PDI-RB-LL-180907 (rinsate blank)                         | L2161800-20   |
| PDI-RB-AL-180907 (rinsate blank)                         | L2161800-21   |

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 High Resolution Superfund Methods Data Review*, April 2016, *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 ANALYSIS

Samples were analyzed for chlorinated pesticides by EPA Method 1699.

1. Holding Times – Acceptable
2. Initial and Continuing Calibration Verifications – Acceptable
3. Blanks – Acceptable
4. Labeled compounds – Acceptable except as noted below:

The percent recovery for 4,4'-DDE-13C12 (15%) was outside the control limits of 21-125% in the LCS extracted on September 20, 2018. Data were not qualified based on the labeled compound recovery in a QC sample.

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

The percent recovery for 2,4'-DDE (152%) exceeded the control limits of 50-120% in the LCS extracted on September 12, 2018. 2,4'-DDE was not detected in associated samples; therefore, data were not qualified based on this elevated LCS recovery.

7. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

An MS/MSD was not performed in association with the rinsate blanks. Accuracy was assessed using the LCS results. Precision was not assessed.

**Data Validation Report**  
**Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling**  
**Subsurface Sediment – Deep/Nearshore Cores**  
**ALS Lab Group: L2161800**

An MS/MSD was not performed in association with the sediment samples. Accuracy was assessed using the LCS results. Precision was assessed using the laboratory and field duplicate results.

8. Laboratory Duplicate – Acceptable

A laboratory duplicate was performed using PDI-SC-S113C-0TO1.1. Results greater than five times the reporting limits (RLs) were evaluated. Results were comparable.

9. Field Duplicate – Acceptable except as noted below

A field duplicate was submitted for PDI-SC-S019-10TO12 and identified as PDI-SC-S019-10TO12D. Results greater than five times the RLs were evaluated. Results were comparable with the following exceptions.

The relative percent difference (RPD) for 2,4'-DDD was greater than 50% for the PDI-SC-S019-10TO12 /PDI-SC-S019-10TO12D field duplicate pair. The results for 2,4'-DDD in PDI-SC-S019-10TO12 and PDI-SC-S019-10TO12D were qualified as estimated and flagged 'J' based on these field duplicate results.

10. Reporting Limits – Acceptable except as noted below:

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

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 do not exceeded the cleanup level.

Analytes with an ion abundance ratio outside the control limits of 1.56 +/- 25% were flagged 'R' by the laboratory indicating an 'EMPC' (estimated maximum possible concentration). These results were qualified as tentatively identified and flagged 'JN' based on this laboratory flag as identified in Table 1.

12. Other Items:

Samples PDI-SC-S260-2.6TO4.2 and PDI-SC-S260-4.2TO6 were re-analyzed due to potential instrument carryover. Results from the re-analysis were reported.

## CONVENTIONAL ANALYSIS

Sediment samples were analyzed for total solids by ASTM D-2974.

1. Holding Times – Acceptable except as noted below:

The sediment samples exceeded the 7-day holding time as indicated in the QAPP. No data qualifiers were assigned based on the holding time exceedance.



**Data Validation Report**

**Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling**

**Subsurface Sediment – Deep/Nearshore Cores**

**ALS Lab Group: L2161800**

2. Laboratory Duplicate – Acceptable

A laboratory duplicate was performed using PDI-SC-S113C-0TO1.1. Results were comparable.

3. Field Duplicate – Acceptable

A field duplicate was submitted for PDI-SC-S019-10TO12 and identified as PDI-SC-S019-10TO12D. Results were comparable.

4. 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 L2161800 is 100%.

**Table 1**  
**QA/QC Data Summary Review**  
**Portland Harbor**  
**Subsurface Sediment**  
**ALS Burlington Laboratory Group: L2161800**

| Sample ID             | Laboratory ID | Method | Analyte  | Laboratory Result | Units | Final Result | Reason Code |
|-----------------------|---------------|--------|----------|-------------------|-------|--------------|-------------|
| PDI-SC-S113C-5.6TO6.6 | L2161800-4    | E1699M | 2,4-DDD  | 0.057 J,R         | ng/g  | 0.057 JN     | k           |
| PDI-SC-S260-4.2TO6    | L2161800-8    | E1699M | 4,4'-DDE | 0.023 M,J,R       | ng/g  | 0.023 JN     | k           |
| PDI-SC-S019-4TO6      | L2161800-12   | E1699M | 2,4-DDT  | 0.11 J,R          | ng/g  | 0.11 JN      | k           |
| PDI-SC-S019-8TO10     | L2161800-14   | E1699M | 2,4-DDT  | 0.26 J,R          | ng/g  | 0.26 JN      | k           |
| PDI-SC-S019-10TO12    | L2161800-15   | E1699M | 2,4-DDD  | 8.42              | ng/g  | 8.42 J       | fd          |
| PDI-SC-S019-10TO12    | L2161800-15   | E1699M | 2,4-DDT  | 0.23 M,J,R        | ng/g  | 0.23 JN      | k           |
| PDI-SC-S019-10TO12D   | L2161800-18   | E1699M | 2,4-DDD  | 14.6              | ng/g  | 14.6 J       | fd          |

**Notes:**

fd - field duplicate RPD  
J - estimated value  
JN - tentatively identified analyte  
k - Estimated Maximum Possible Concentration (EMPC)  
M - manual integration by laboratory  
ng/g - nanogram per gram  
R - Ion abundance outside acceptance criterion  
RPD - relative percent difference