

Data Validation Report

Project: Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling
 Portland Harbor Superfund Site
 Subsurface Sediment – Nearshore Core Stations

Laboratory: ALS Environmental, Burlington, Ontario, Canada

Laboratory Group: L2179362

Analyses/Method: Chlorinated Pesticides and Total Solids

Validation Level: Stage 2

AECOM Project

Number: 60566335 Task #2.12

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File Name: L2179362 DVR

SUMMARY

The data quality review of 2 subsurface sediment samples collected on August 1, 2018, has been completed. Samples were analyzed for chlorinated pesticides by EPA Method 1699-modified (GC/HRMS) and 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 L2179362:

Sample ID	Laboratory ID
PDI-SC-S088-0TO2	L2179362-01
PDI-SC-S088-2TO3.3	L2179362-02

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.

The samples were collected on August 1, 2018 and were held frozen in the AECOM storage facility freezer until shipment to ALS-Burlington. The samples were received by the laboratory on October 11, 2018.



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ORGANIC ANALYSIS

Samples were analyzed for chlorinated pesticides by EPA Method 1699.

1. Holding Times – Acceptable

The samples were extracted 61 days past the method-recommended holding time of 14 days. As described above, the samples were frozen in archive until shipment; therefore, the samples were not extracted outside the holding time.

2. Initial and Continuing Calibration Verifications – Acceptable

3. Blanks – Acceptable

4. Labeled compounds – Acceptable except as noted below:

The percent recoveries for the following labeled compounds exceeded the control limits.

Sample	Labeled Compound	Percent Recovery	Control Limits
PDI-SC-S088-2TO3.3	4,4'-DDE-13C12	129%	21-125%
MB (Batch WG2899461)	4,4'-DDE-13C12	150%	21-125%
	4,4'-DDT-13C12	133%	5-120%

MB – method blank

Data were not qualified based on the labeled compound recovery in a QC sample. The results for 2,4'-DDE and 4,4'-DDE in PDI-SC-S088-2TO3.3 were qualified as estimated and flagged 'J' based on this labeled compound recovery.

5. Internal Standards – Acceptable

6. Laboratory Control Sample (LCS) – Acceptable

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

An MS/MSD was not performed in association with this laboratory group. Accuracy was assessed using the LCS results. Precision was not assessed.

8. Laboratory Duplicate

A laboratory duplicate was not performed in association with this laboratory group. Precision was not assessed.

9. Field Duplicate

A field duplicate was not collected in with this laboratory group. Precision was not assessed.



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10. Reporting Limits – Acceptable except as noted below:

Several results in PDI-SC-S088-2TO3.3 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.

2,4'-DDT in PDI-SC-S088-2TO3.3 had an ion abundance ratio outside the control limits of 1.56 +/- 25% and was flagged 'R' by the laboratory indicating an 'EMPC' (estimated maximum possible concentration). The result for 2,4'-DDT in PDI-SC-S088-2TO3.3 was qualified as tentatively identified and flagged 'JN' based on this ion abundance ratio.

CONVENTIONAL ANALYSIS

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

1. Holding Times – Acceptable

The 7-day holding time indicated in the QAPP was exceeded. Sample data was not qualified based on the holding time exceedance.

2. Laboratory Duplicate

A laboratory duplicate was not performed in association with this laboratory group. Precision was not assessed.

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 L2179362 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Subsurface Sediment
ALS Burlington Laboratory Group: L2179362

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SC-S088-2TO3.3	L2179362-2	E1699M	2,4-DDE	0.0551 J	ng/g	0.0551 J	lc
PDI-SC-S088-2TO3.3	L2179362-2	E1699M	4,4'-DDE	0.230 J	ng/g	0.230 J	lc
PDI-SC-S088-2TO3.3	L2179362-2	E1699M	2,4-DDT	0.061 J,R	ng/g	0.061 JN	k

Notes:

- J - estimated value
- JN - tentatively identified analyte
- k - Estimated Maximum Possible Concentration (EMPC)
- lc - labeled compound recovery
- ng/g - nanogram per gram
- R - Ion abundance outside acceptance criterion