

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: L2134269

Analyses/Method: Chlorinated Pesticides and Total Solids

Validation Level: Stage 2

AECOM Project

Number: 60566335 Task #2.12

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File Name: L2134269 DVR

SUMMARY

The data quality review of 6 subsurface sediment samples collected on July 20, 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 L2134269:

Sample ID	Laboratory ID
PDI-SC-S036-0TO1.4	L2134269-1
PDI-SC-S036-1.4TO3.4	L2134269-2
PDI-SC-S036-3.4TO5.2	L2134269-3
PDI-SC-S022-0TO2	L2134269-4
PDI-SC-S022-2TO4	L2134269-5
PDI-SC-S022-4TO6	L2134269-6

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

Data Validation Report
Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling
Subsurface Sediment – Nearshore Core Stations
ALS Lab Group: L2134269

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 except as noted below:

The percent recoveries for the following analytes and labeled compounds were outside the control limits of 75-125% and 70-130%, respectively.

Analysis Date and Time	Analyte	% Recovery
August 9, 2018	4,4'-DDT-13C12	51%
August 10, 2018	2,4'-DDD	138%
	4,4'-DDD-13C12	47%
	4,4'-DDT-13C12	31%

The results for 2,4'-DDD, 4,4'-DDD, 2,4'-DDT and 4,4'-DDT in PDI-SC-S036-0TO1.4, PDI-SC-S036-1.4TO3.4, PDI-SC-S036-3.4TO5.2, PDI-SC-S022-0TO2, PDI-SC-S022-2TO4, and PDI-SC-S022-4TO6 were qualified as estimated and flagged 'J' or 'UJ' based on these CCV results.

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. Sediment data were not qualified based on rinsate blank results.

4. Labeled compounds – Acceptable
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 these samples. Accuracy was assessed using the LCS results. Precision was not assessed.

8. Laboratory Duplicate

A laboratory duplicate was not performed using a sample from this laboratory group.



Data Validation Report

Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling

Subsurface Sediment – Nearshore Core Stations

ALS Lab Group: L2134269

9. 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 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. AECOM uses 30% solids (from the NFG pesticide guidance) as a benchmark to evaluate the percent solids content and professional judgment is used to determine the necessity to qualify data. Qualification on this basis was not required. The reporting limits do not exceed 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.

CONVENTIONAL ANALYSIS

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.

2. Laboratory Duplicate

A laboratory duplicate was not performed using a sample from this laboratory group.

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 L2134269 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Subsurface Sediment
ALS Burlington Laboratory Group: L2134269

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SC-S036-0TO1.4	L2134269-1	E1699M	2,4-DDD	0.860	ng/g	0.860 J	c
PDI-SC-S036-0TO1.4	L2134269-1	E1699M	2,4-DDT	2.68	ng/g	2.68 J	c
PDI-SC-S036-0TO1.4	L2134269-1	E1699M	4,4'-DDD	2.70	ng/g	2.70 J	c
PDI-SC-S036-0TO1.4	L2134269-1	E1699M	4,4'-DDT	4.63	ng/g	4.63 J	c
PDI-SC-S036-1.4TO3.4	L2134269-2	E1699M	2,4-DDD	3.03	ng/g	3.03 J	c
PDI-SC-S036-1.4TO3.4	L2134269-2	E1699M	2,4-DDT	0.162 M,J	ng/g	0.162 J	c
PDI-SC-S036-1.4TO3.4	L2134269-2	E1699M	4,4'-DDD	9.00 M	ng/g	9.00 J	c
PDI-SC-S036-1.4TO3.4	L2134269-2	E1699M	4,4'-DDT	0.955	ng/g	0.955 J	c
PDI-SC-S036-3.4TO5.2	L2134269-3	E1699M	2,4-DDD	25.2	ng/g	25.2 J	c
PDI-SC-S036-3.4TO5.2	L2134269-3	E1699M	2,4-DDT	1.72	ng/g	1.72 J	c
PDI-SC-S036-3.4TO5.2	L2134269-3	E1699M	4,4'-DDD	35.7	ng/g	35.7 J	c
PDI-SC-S036-3.4TO5.2	L2134269-3	E1699M	4,4'-DDT	12.3	ng/g	12.3 J	c
PDI-SC-S022-0TO2	L2134269-4	E1699M	2,4-DDD	0.012 U	ng/g	0.012 UJ	c
PDI-SC-S022-0TO2	L2134269-4	E1699M	2,4-DDT	0.021 U	ng/g	0.021 UJ	c
PDI-SC-S022-0TO2	L2134269-4	E1699M	4,4'-DDD	0.017 U	ng/g	0.017 UJ	c
PDI-SC-S022-0TO2	L2134269-4	E1699M	4,4'-DDE	0.015 M,J,R	ng/g	0.015 JN	k
PDI-SC-S022-0TO2	L2134269-4	E1699M	4,4'-DDT	0.037 U	ng/g	0.037 UJ	c
PDI-SC-S022-2TO4	L2134269-5	E1699M	2,4-DDD	0.010 U	ng/g	0.010 UJ	c
PDI-SC-S022-2TO4	L2134269-5	E1699M	2,4-DDT	0.014 U	ng/g	0.014 UJ	c
PDI-SC-S022-2TO4	L2134269-5	E1699M	4,4'-DDD	0.011 U	ng/g	0.011 UJ	c
PDI-SC-S022-2TO4	L2134269-5	E1699M	4,4'-DDT	0.045 U	ng/g	0.045 UJ	c
PDI-SC-S022-4TO6	L2134269-6	E1699M	2,4-DDD	0.028 U	ng/g	0.028 UJ	c
PDI-SC-S022-4TO6	L2134269-6	E1699M	2,4-DDT	0.018 U	ng/g	0.018 UJ	c
PDI-SC-S022-4TO6	L2134269-6	E1699M	4,4'-DDD	0.014 U	ng/g	0.014 UJ	c
PDI-SC-S022-4TO6	L2134269-6	E1699M	4,4'-DDE	0.030 M,J,R	ng/g	0.030 JN	k
PDI-SC-S022-4TO6	L2134269-6	E1699M	4,4'-DDT	0.053 U	ng/g	0.053 UJ	c

Notes:

- c - calibration issue
- 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
- U - Compound was analyzed for, but not detected above the value shown.
- UJ - reported quantitation limit is approximate