

Data Validation Report

Project:	Portland Harbor
Laboratory:	Alpha Analytical Laboratory
Environmental Test Record (ETR):	1408048
Analyses/Method:	Polycyclic Aromatic Hydrocarbons (PAH), Petroleum Biomarkers, n-Alkanes and Total Petroleum Hydrocarbons (TPH), and Total Organic Carbon (TOC)

Summary

One tar ball sample was collected in Portland Harbor, Oregon on August 22, 2014. The sample was analyzed for polycyclic aromatic hydrocarbons (PAH) and petroleum biomarkers by EPA Method 8270D modified by selected ion monitoring mode (SIM), n-alkanes and total petroleum hydrocarbons (TPH) by EPA Method 8015D, and total organic carbon (TOC) by EPA Method 9060A by Alpha Analytical Laboratory located in Mansfield, Massachusetts. The laboratory provided Level 4 data packages containing samples results and associated quality assurance (QA) and quality control (QC) data, preparation logs, and raw instrument output. The following sample is associated with the laboratory ETR 1408048.

Sample ID	Laboratory ID	Matrix
PH14-T05-TB	1408048-01	Solid

The data have been independently validated using *USEPA Contact Laboratory Program National Functional Guidelines for Organic Superfund Methods Data Review* EPA-540-R-2017-002, dated January 2017. Validation includes reconstruction of the analytical data to verify that data are traceable and sufficiently complete in order for a qualified individual other than the originator to perform reconstruction of the data. The validation included the following checks:

- Sample Receipt/Transcription error check
- Sample preservation
- Sample holding times
- Tune Summary
- Initial calibration
- Continuing calibration verification (CCV)
- Laboratory blank contamination
- Equipment blank contamination
- Surrogate spike recoveries
- Internal Standard recoveries
- Matrix spike/Matrix spike duplicate (MS/MSD) recoveries, relative percent difference (RPD)
- Standard Reference Material Sediment accuracy check
- Laboratory control sample (LCS), LCS Duplicate (LCSD) recoveries, RPD values
- Calculation checks
- Contract Required Quantitation Limit (CRQL)
- Field duplicate results
- Laboratory duplicate results
- Overall assessment of the data



Data validation is based on the QC criteria documented in *Portland Harbor Sediment Forensic Chemistry Study, Portland Harbor Oregon Quality Assurance Project Plan (QAPP)*,¹ dated July 29, 2014, and the *Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Quality Assurance Project Plan (QAPP)*,² dated March 23, 2018. There were no data qualifiers assigned to results reported in this sample set.

Sample Receipt

Chain of custody documentation were reviewed for completeness of information relevant to the samples and requested analysis. Sample IDs and sample collection dates from the chain of custody records were matched to the reported data. The discrepancies noted below.

The chain of custody documentation for PH14-T05-TB lists the matrix as sediment, however field notes and photographs document the matrix as a tar ball. The sample was logged in as a solid.

The cooler was received within $4 \pm 2^\circ\text{C}$.

ORGANIC ANALYSES

Holding Time and Sample Preservation

All samples were extracted and analyzed within holding times.

GC/MS Instrument Performance Check – Acceptable.

Initial Calibration and Continuing Calibration Verifications – Acceptable.

Blanks– Acceptable except as noted below:

Method Blank: The method blank met the QC acceptance criteria for PAH and biomarkers (EPA Method 8270D). PAH were detected in the method blank below the reporting limit. However the associated sample results were either non-detect or were greater than ten times the blank concentration. Data were not qualified based on method blank results.

The method blank met the QC acceptance criteria for n-alkanes and TPH (EPA Method 8015D). n-Alkanes were detected in the method blank below the reporting limit. However the associated sample results were either non-detect or greater than ten times the blank concentration. Data were not qualified base on method blank results.

Rinsate Blank: One rinsate blank (PH14-RB4) was collected on August 21, 2014 (ETR 1408040) and is associated with the sediment samples in this ETR. Detections of target compounds in rinsate blanks were evaluated relative to sediment method detection limits (MDL). No target analytes were found in rinsate blanks at relative concentrations at, or above, the sediment MDL. No data were qualified based on the rinsate blank results.

¹ NewFields. (2014). Portland Harbor Sediment Forensic Chemistry Study, Portland Harbor Oregon Quality Assurance Project Plan (QAPP). July 29, 2014.

² AECOM and Geosyntec. 2018. Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Portland Harbor Superfund Site, Quality Assurance Project Plan. March 23, 2018,



Surrogate Spikes – Acceptable.

Internal Standard Areas – Acceptable.

Laboratory Control Samples – Acceptable.

Matrix Spike/Spike Duplicate – A matrix spike/spike duplicate was not performed for this ETR. The precision and accuracy of the method was demonstrated by the results of the LCS/LCSD.

Field Duplicate– There were no field duplicates associated with this ETR. The precision of the method was demonstrated by the results of the LCS/LCSD.

Laboratory Duplicate– Acceptable.

Target Compound Identifications– Acceptable

Compound Quantitation and CRQLs – Acceptable

CONVENTIONAL ANALYSES

Holding Time and Sample Preservation – Acceptable

Initial Calibration and Continuing Calibration Verifications – Acceptable

Blanks– Acceptable

Matrix Spike/Spike Duplicate – Acceptable.

Note: There were no MSDs analyzed for TOC in this ETR. The precision of the method was demonstrated by the results of the laboratory duplicate. The accuracy of the method was demonstrated by the results of the TOC standard reference materials (SRM 1941b and SRM1944) that was reported with this ETR and met the QC acceptance criteria.

Standard Reference Material – Acceptable.

Field Duplicate– There were no field duplicates associated with this ETR. The precision of the method was demonstrated by the results of the laboratory duplicate.

Laboratory Duplicate– Acceptable.

Compound Quantitation and CRQLs – Acceptable

OVERALL ASSESSMENT OF DATA

The data reported in this laboratory ETR is considered usable for meeting the project objectives.

The completeness is calculated by the number of usable data points divided by the total number of data points generated, multiplied by 100. The completeness for the laboratory ETR is 100%.



Validation performed by and Date:

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Staff Scientists - NewFields