

Data Validation Report

Project:	Portland Harbor
Laboratory:	Alpha Analytical Laboratory
Environmental Test Record (ETR):	1408047
Analyses/Method:	Polycyclic Aromatic Hydrocarbons (PAH), Petroleum Biomarkers, n-Alkanes and Total Petroleum Hydrocarbons (TPH), and Total Organic Carbon (TOC)

Summary

Six sediment samples were collected in Portland Harbor, Oregon on August 22, 2014. Samples were 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 sediment samples are associated with the laboratory ETR 1408047.

Note: Samples Port Wing Tank #4 and Diesel-A5606 are two reference materials (heavy fuel and diesel) were logged as samples 1408047-10 and -11, analyzed and reported. These reference materials are not included in the chain of custody section of this report.

Sample ID	Lab ID	Matrix
PH14-S30-S	1408047-01	Sediment
PH14-S30-Z	1408047-02	Sediment
PH14-S29-S	1408047-03	Sediment
PH14-S29-Z	1408047-04	Sediment
PH14-S28-S	1408047-05	Sediment
PH14-S28-Z	1408047-06	Sediment
Port Wing Tank #4	1408047-10	Oil
Diesel-A5606	1408047-11	Product

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. Data qualifiers assigned to results reported in this sample set are included in Table 1. Reason codes and explanations for qualified data are provided in Table 2.

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. No discrepancies noted.

All coolers were 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 (sediment): The method blank met the QC acceptance criteria for PAH and biomarkers. PAH were detected in the method blank below the reporting limit. However, with the exception cis/trans-decalin, the associated sediment sample results were either non-detect or greater than ten times the blank concentration. Samples containing cis/trans-decalin at concentrations below the

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,



reporting limit and less than ten times the blank result were qualified as not detected, and were flagged “U” at the reporting limit based on the method blank result.

The method blank met the QC acceptance criteria for n-alkanes and TPH. n-Alkanes were detected in the method blank below the reporting limit. However, with the exception of n-decane, the associated sample results were either non-detect or greater than ten times the blank concentration. Samples containing n-decane at concentrations below the reporting limit and less than ten times the blank result were qualified as not detected, and were flagged “U” at the reporting limit based on the method blank result.

Method Blank (oil/product): The method blank met the QC acceptance criteria for PAH and biomarkers. PAH were detected in the method blank below the reporting limit. However, with the exception of the analytes below, the associated oil/product sample results were either non-detect or greater than ten times the blank concentration. Samples containing the below listed analytes at concentrations below the reporting limit and less than ten times the blank result were qualified as not detected, and were flagged “U” at the reporting limit based on the method blank result.

PAH and Biomarker Compounds	Result	Unit	Lab Qualifier
Naphthobenzothiophenes	0.286	mg/Kg	J
Benz[a]anthracene	0.537	mg/Kg	J
Benzo[b]fluoranthene	0.212	mg/Kg	J
Benzo[e]pyrene	0.156	mg/Kg	J
Benzo[g,h,i]perylene	0.163	mg/Kg	J

The method blank met the QC acceptance criteria for n-alkanes and TPH. 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. No sample results were qualified based on the method blank result.

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.

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 of the method was demonstrated by the results of the LCS/LCSD. In addition, a PAH standard reference material (SRM 1941b), was reported with this ETR and met the QC acceptance criteria. The results of the SRM demonstrate the precision and accuracy has been achieved for this ETR.

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 LCS/LCSD.

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

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.

Standard Reference Material – Acceptable.

Field Duplicate— Acceptable.

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:

George Desreuisseau, Mike Mitchel and Kerylynn Krahforst, December 2018.



Staff Scientists - NewFields

Table 1. QA/QC Summary Review

Sdg	SoilSampID	Lab_ID	AnalMeth	Analyte	Result	Lab_Flag	Units	NFG Result	NFG Qualifier	validator_ reason_code
1408047	PH14-S28-S	1408047-05X	EPA 8015M	n-Decane (C10)	0.00562	JB	mg/Kg	0.122	U	bl
1408047	PH14-S28-Z	1408047-06X	EPA 8015M	n-Decane (C10)	0.00487	JB	mg/Kg	0.119	U	bl
1408047	PH14-S29-S	1408047-03X	EPA 8015M	n-Decane (C10)	0.0044	JB	mg/Kg	0.122	U	bl
1408047	PH14-S29-Z	1408047-04X	EPA 8015M	n-Decane (C10)	0.00504	JB	mg/Kg	0.117	U	bl
1408047	PH14-S30-S	1408047-01X	EPA 8015M	n-Decane (C10)	0.00399	JB	mg/Kg	0.0973	U	bl
1408047	PH14-S30-Z	1408047-02X	EPA 8015M	n-Decane (C10)	0.00345	JB	mg/Kg	0.0986	U	bl
1408047	DIESEL-A5606	1408047-11	EPA 8270D	Benzo[b]fluoranthene	277	JB	ug/Kg	1930	U	bl
1408047	DIESEL-A5606	1408047-11	EPA 8270D	Benzo[e]pyrene	393	JB	ug/Kg	1930	U	bl
1408047	DIESEL-A5606	1408047-11	EPA 8270D	Benzo[g,h,i]perylene	247	JB	ug/Kg	1930	U	bl
1408047	DIESEL-A5606	1408047-11	EPA 8270D	Naphthobenzothiophenes	1680	JB	ug/Kg	1930	U	bl
1408047	DIESEL-A5606	1408047-11	EPA 8270D	Benz[a]anthracene	997	JB	ug/Kg	1930	U	bl
1408047	PH14-S28-S	1408047-05X	EPA 8270D	cis/trans-Decalin	0.947	JB	µg/Kg	1.22	U	bl
1408047	PH14-S28-Z	1408047-06X	EPA 8270D	cis/trans-Decalin	0.921	JB	µg/Kg	1.19	U	bl
1408047	PH14-S29-S	1408047-03X	EPA 8270D	cis/trans-Decalin	0.707	JB	µg/Kg	1.22	U	bl
1408047	PH14-S29-Z	1408047-04X	EPA 8270D	cis/trans-Decalin	0.944	JB	µg/Kg	1.17	U	bl
1408047	PH14-S30-S	1408047-01X	EPA 8270D	cis/trans-Decalin	0.684	JB	µg/Kg	0.973	U	bl
1408047	PH14-S30-Z	1408047-02X	EPA 8270D	cis/trans-Decalin	0.42	JB	µg/Kg	0.986	U	bl

Table 2. Reason Codes and Explanations

Reason Code	Explanation
bf	Field blank contamination
bl	Laboratory blank contamination
C	Calibration issue
el	Clean-up standard recovery
d	Reporting limit raised due to chromatographic interference
fd	Field duplicate RPDs
h	Holding Times
i	Internal standard areas
k	Estimated Maximum Possible Concentration (EMPC)
l	LCS or OPR recoveries
le	Labeled compound recovery
ld	Laboratory duplicate RPDs
lp	Laboratory control sample laboratory control sample duplicate RPDs
m	Matrix spike recovery
md	Matrix spike/matrix spike duplicate RPDs
nb	Negative laboratory blank contamination
p	Chemical preservation issue
r	Dual column RPD
q	Quantitation issue
s	Surrogate recovery
su	Ion suppression
t	Temperature preservation issue
x	Percent solids
y	Serial dilution results
z	ICS results