

Data Validation Report

Project:	Portland Harbor
Laboratory:	Alpha Analytical Laboratory
Environmental Test Record (ETR):	1408040
Analyses/Method:	Polycyclic Aromatic Hydrocarbons (PAH), petroleum biomarkers , and n-Alkanes and Total Petroleum Hydrocarbons (TPH)

Summary

Three sea water samples and four rinsate blanks were collected in Portland Harbor, Oregon on August 19, 2014, August 20, 2014, and August 21, 2014. The sea water samples were analyzed for polycyclic aromatic hydrocarbons (PAH) by EPA Method 8270D modified by selected ion monitoring mode (SIM). The rinsate blanks were analyzed for PAH and petroleum biomarkers by EPA Method 8270D modified by selected ion monitoring mode (SIM), and n-alkanes and total petroleum hydrocarbons (TPH) by EPA Method 8015D. The samples were analyzed by Alpha Analytical Laboratory located in Mansfield, Massachusetts. The laboratory provided Level 4 data packages containing sample results and associated quality assurance (QA) and quality control (QC) data, preparation logs, and raw instrument output. The following samples are associated with the laboratory ETR 1408040.

Sample ID	Lab ID	Matrix
PH14-RB2	1408040-01	Water
PH14-RB3	1408040-02	Water
PH14-RB4	1408040-03	Water
PH14-T09-W	1408040-04	Water
PH14-T05-W	1408040-05	Water
PH14-T05-W-D	1408040-06	Water
PH14-RB-W	1408040-07	Water

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

General: The samples were extracted twice: the original sample was extracted within 7-days of sampling, the second extraction was outside the 14-day holding time. The original analysis was reviewed for data quality purposes.

Holding Time and Sample Preservation

The 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. With the exception of naphthalene, PAH were detected in the method blank below the reporting limit. Samples containing the below listed analytes were qualified as not detected, and were flagged "U" at the reporting limit based on the method blank result.

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,



PAH Compounds	Result	Unit	Lab Qualifier
Naphthalene	362	ng/L	
C1-Naphthalenes	2.25	ng/L	J
Biphenyl	1.25	ng/L	J
Dibenzofuran	0.41	ng/L	J
Fluorene	0.751	ng/L	J
Anthracene	0.175	ng/L	J
Phenanthrene	1.76	ng/L	J
Dibenzothiophene	1.23	ng/L	J
Fluoranthene	0.579	ng/L	J
Pyrene	0.396	ng/L	J
Chrysene/Triphenylene	0.467	ng/L	J
Cyclopenta(cd)pyrene	1.4	ng/L	J
2-Methylnaphthalene	1.58	ng/L	J
1-Methylnaphthalene	2.18	ng/L	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. With the exception of the analytes below, the associated sample results were either non-detect or were greater than 10X the blank concentration. Samples containing the below listed analytes at concentrations below the reporting limit were qualified as not detected, and were flagged "U" at the reporting limit based on the method blank result.

n-Alkanes and TPH Compounds	Result	Unit	Lab Qualifier
n-Nonane (C9)	0.00319	µg/L	J
n-Decane (C10)	0.00957	µg/L	J
2,6,10 Trimethyldodecane (1380)	0.0149	µg/L	J
n-Hexadecane (C16)	0.0213	µg/L	J
n-Eicosane (C20)	0.00957	µg/L	J
n-Heneicosane (C21)	0.0128	µg/L	J
n-Docosane (C22)	0.0117	µg/L	J
n-Tricosane (C23)	0.0351	µg/L	J
n-Tetracosane (C24)	0.0191	µg/L	J
n-Hexacosane (C26)	0.0149	µg/L	J
n-Heptacosane (C27)	0.0298	µg/L	J
n-Nonacosane (C29)	0.682	µg/L	CJ
n-Triacontane (C30)	0.017	µg/L	J
n-Hentriacontane (C31)	0.0181	µg/L	J
n-Dotriacontane (C32)	0.0213	µg/L	J
Total Saturated Hydrocarbons	0.92	µg/L	J
Total Petroleum Hydrocarbons (C9-C44)	11.4	µg/L	J

Surrogate Spikes – Acceptable.

Internal Standard Areas – Acceptable.

Laboratory Control Samples – Acceptable.



Target Compound Identifications– Acceptable.

Compound Quantitation and CRQLs – Acceptable.

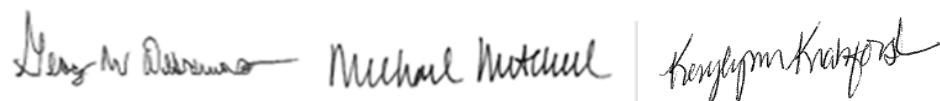
OVERALL ASSESSMENT OF DATA

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 Desreusseau, Mike Mitchel and Kerylynn Krahforst, January 2019.



Staff Scientists - NewFields

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