

## Data Validation Report

Project:	Portland Harbor
Laboratory:	SGS
Sample Delivery Group (SDG):	A8345
Analyses/Method:	Polychlorinated dibenzo-p-dioxins and furans (PCDD/Fs)

### Summary

Fifteen sediment samples were collected in Portland Harbor, Oregon on October 21, 2015, October 22, 2015, and October 23, 2015. Samples were analyzed for polychlorinated dibenzo-p-dioxins and furans (PCDD/Fs) by EPA Method 1613B by SGS Laboratory located in Wilmington, North Carolina. 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 SDG A8345.

Sample ID	Lab ID	Matrix
PH15-34-A	A8345_001	Sediment
PH15-10-A	A8345_002	Sediment
PH15-11-A	A8345_003	Sediment
PH15-11-D	A8345_004	Sediment
PH15-11-D-FD	A8345_005	Sediment
PH15-28-A	A8345_006	Sediment
PH15-31-A	A8345_007	Sediment
PH15-40-A	A8345_008	Sediment
PH15-40-B	A8345_009	Sediment
PH15-40-C	A8345_010	Sediment
PH15-40-D	A8345_011	Sediment
PH15-43-A	A8345_012	Sediment
PH15-50-A	A8345_013	Sediment
PH15-50-C	A8345_014	Sediment
PH15-50-D	A8345_015	Sediment

The data have been independently validated using *USEPA Contact Laboratory Program National Functional Guidelines for High Resolution Superfund Methods Data Review* EPA-542-B-16-001, dated April 2016. 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
- High Resolution Mass Spectrometer (HRMS) check
- 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)
- 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 Supplemental Sediment Study, Portland Oregon Quality Assurance Project Plan (QAPP)*,<sup>1</sup> dated October 14, 2015, and the *Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Quality Assurance Project Plan (QAPP)*,<sup>2</sup> 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.

**HRMS Resolution 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 PCDD/F. PCDD/F were detected in the method blank below the reporting limit. However, with the exception of Total HxCDD, the associated sample results were either non-detect or greater than five times the blank concentration. Sample PH15-40-D contained Total HxCDD a concentration below the reporting limit and less than five time the blank result. For sample PH15-40-D, Total HxCDD was qualified as estimated and flagged “J” based on method blank results.

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<sup>1</sup> NewFields. (2015). Portland Harbor Supplemental Sediment Study, Portland Oregon Quality Assurance Project Plan (QAPP). October 14, 2015.

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



**Rinsate Blank:** Three rinsate blanks were collected on October 23, 2015, October 26, 2015, and October 27, 2015 (PH15-03-RB, PH15-04-RB, PH15-05-RB, respectively [SDG A8328 and SDG A8344]) and are associated with the samples in this ETR.

- PH15-03-RB is associated with: PH15-40-A, PH15-40-B, PH15-40-C, PH15-40-D, PH15-50-A, PH15-50-C, PH15-50-D, PH15-28-A, and PH15-10-A
- PH15-04-RB is associated with: PH15-31-A, PH15-43-A, PH15-11-A, PH15-11-D, and PH15- 11- D- FD
- PH15-05-RB is associated with: PH15-34-A.

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 except as noted below:

Sample ID	Surrogate Compound	Recovery (%)	QC Limit (%)
PH15-11-D	ES OCDD	15.5	17 – 157
PH15-11-D	ES OCDF	15.2	17 – 157

The surrogate recovery for surrogate compounds listed above were above the acceptance criteria. For sample PH15-11-D, OCDD and OCDF were qualified as estimated and flagged “J”.

**Internal Standard Areas** – Acceptable.

**Laboratory Control Samples** – Acceptable.

**Matrix Spike/Spike Duplicate** – Acceptable.

**Field Duplicate**– Acceptable except as noted below:

A field duplicate was submitted for PH15-11-D and was identified as PH15-11-D-FD. The results for the field duplicates were comparable except as noted below

Sample ID	Field Duplicate ID	Analyte	RPD (%)	QC Limit (%)
PH15-14-A	PH15-14-FD	2378-TCDF	57	50
		Total HxCDD	108	50
		Total TCDF	98	50
		Total HxCDF	64	50

The results for the analytes listed above were qualified as estimated and flagged “J” based on elevated field duplicates.

**Laboratory Duplicate**– Acceptable except as noted below:

Sample ID	Analyte	RPD (%)	QC Limit (%)
PH15-34-A	2,3,7,8-TCDF	47	30



Sample ID	Analyte	RPD (%)	QC Limit (%)
	1,2,3,7,8-PeCDF	51	30
	2,3,4,7,8-PeCDF	52	30
	1,2,3,4,7,8-HxCDF	50	30
	2,3,4,6,7,8-HxCDF	32	30
	1,2,3,7,8,9-HxCDF	30	30
	1,2,3,4,7,8,9-HpCDF	68	30
	Total TCDD	55	30
	Total PeCDD	74	30
	Total TCDF	30	30
	Total PeCDF	49	30

The results for the analytes listed above were qualified as estimated and flagged “J” based on elevated laboratory duplicates.

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

George Desreuisseau | Michael Mitchel | Kerylynn Krahforst

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
A8345	PH15-40-D	A8345_13717_DF_011-D5	EPA 1613B	Total HxCDD	0.388		pg/g	0.388	U	bl
A8345	PH15-11-D	A8345_13717_DF_004-D5	EPA 1613B	Total HxCDD	18.7	EMPC	pg/g		J	fd
A8345	PH15-11-D	A8345_13717_DF_004-D5	EPA 1613B	Total HxCDF	5.25	EMPC	pg/g		J	fd
A8345	PH15-11-D	A8345_13717_DF_004-D5	EPA 1613B	Total TCDF	3.23	EMPC	pg/g		J	fd
A8345	PH15-11-D	A8345_13717_DF_004-D5	EPA 1613B	2378-TCDF	0.685		pg/g		J	fd
A8345	PH15-11-D-FD	A8345_13717_DF_005-D5	EPA 1613B	Total HxCDD	6.84	EMPC	pg/g		J	fd
A8345	PH15-11-D-FD	A8345_13717_DF_005-D5	EPA 1613B	Total HxCDF	3.32	EMPC	pg/g		J	fd
A8345	PH15-11-D-FD	A8345_13717_DF_005-D5	EPA 1613B	2378-TCDF	0.381	EMPC J	pg/g		J	fd
A8345	PH15-11-D-FD	A8345_13717_DF_005-D5	EPA 1613B	Total TCDF	1.47	EMPC	pg/g		J	fd
A8345	PH15-34-A	A8345_13717_DF_001-D5	EPA 1613B	2378-TCDF	0.603	EMPC	pg/g		J	ld
A8345	PH15-34-A	A8345_13717_DF_001-D5	EPA 1613B	23478-PeCDF	0.501	EMPC J	pg/g		J	ld
A8345	PH15-34-A	A8345_13717_DF_001-D5	EPA 1613B	1234789-HpCDF	0.304	J	pg/g		J	ld
A8345	PH15-34-A	A8345_13717_DF_001-D5	EPA 1613B	123478-HxCDF	1.34	J	pg/g		J	ld
A8345	PH15-34-A	A8345_13717_DF_001-D5	EPA 1613B	123789-HxCDF	0	U	pg/g		J	ld
A8345	PH15-34-A	A8345_13717_DF_001-D5	EPA 1613B	12378-PeCDF	0.6	J	pg/g		J	ld
A8345	PH15-34-A	A8345_13717_DF_001-D5	EPA 1613B	Total PeCDD	0	U	pg/g		J	ld
A8345	PH15-34-A	A8345_13717_DF_001-D5	EPA 1613B	Total PeCDF	3.18	EMPC	pg/g		J	ld
A8345	PH15-34-A	A8345_13717_DF_001-D5	EPA 1613B	Total TCDD	0.972		pg/g		J	ld
A8345	PH15-34-A	A8345_13717_DF_001-D5	EPA 1613B	Total TCDF	2.05	EMPC	pg/g		J	ld
A8345	PH15-34-A	A8345_13717_DF_001-D5	EPA 1613B	234678-HxCDF	0.378	EMPC J	pg/g		J	ld
A8345	PH15-34-A-DUP	A8345_13717_DF_001DUP-D5	EPA 1613B	12378-PeCDF	1.01	J	pg/g		J	ld
A8345	PH15-34-A-DUP	A8345_13717_DF_001DUP-D5	EPA 1613B	Total TCDF	2.77	EMPC	pg/g		J	ld
A8345	PH15-34-A-DUP	A8345_13717_DF_001DUP-D5	EPA 1613B	Total TCDD	1.72		pg/g		J	ld
A8345	PH15-34-A-DUP	A8345_13717_DF_001DUP-D5	EPA 1613B	Total PeCDF	5.23	EMPC	pg/g		J	ld
A8345	PH15-34-A-DUP	A8345_13717_DF_001DUP-D5	EPA 1613B	Total PeCDD	0.789		pg/g		J	ld
A8345	PH15-34-A-DUP	A8345_13717_DF_001DUP-D5	EPA 1613B	2378-TCDF	0.969		pg/g		J	ld
A8345	PH15-34-A-DUP	A8345_13717_DF_001DUP-D5	EPA 1613B	234678-HxCDF	0.273	J	pg/g		J	ld
A8345	PH15-34-A-DUP	A8345_13717_DF_001DUP-D5	EPA 1613B	123789-HxCDF	0	U	pg/g		J	ld
A8345	PH15-34-A-DUP	A8345_13717_DF_001DUP-D5	EPA 1613B	123478-HxCDF	2.23	J	pg/g		J	ld
A8345	PH15-34-A-DUP	A8345_13717_DF_001DUP-D5	EPA 1613B	1234789-HpCDF	0.616	J	pg/g		J	ld
A8345	PH15-34-A-DUP	A8345_13717_DF_001DUP-D5	EPA 1613B	23478-PeCDF	0.851	J	pg/g		J	ld
A8345	PH15-11-D	A8345_13717_DF_004-D5	EPA 1613B	OCDD	223		pg/g		J	S
A8345	PH15-11-D	A8345_13717_DF_004-D5	EPA 1613B	OCDF	6.08	EMPC	pg/g		J	S

Table 2. Reason Codes and Explanations

Reason Code	Explanation
be	Equipment blank contamination
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