Data Validation Report

Project:	Portland Harbor
Laboratory:	SGS
Sample Delivery Group (SDG):	A8329
Analyses/Method:	Polychlorinated dibenzo-p-dioxins and furans (PCDD/Fs)

Summary

Fourteen sediment samples were collected in Portland Harbor, Oregon on October 19, 2015 and October 20, 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 A8329.

Sample ID	Lab ID	Matrix
PH15-14-A	A8329_001	Sediment
PH15-14-FD	A8329_002	Sediment
PH15-21-A	A8329_003	Sediment
PH15-21-C	A8329_004	Sediment
PH15-21-D	A8329_005	Sediment
PH15-12-A	A8329_006	Sediment
PH15-03-A	A8329_007	Sediment
PH15-03-B	A8329_008	Sediment
PH15-03-C	A8329_009	Sediment
PH15-03-D	A8329_010	Sediment
PH15-06-A	A8329_011	Sediment
PH15-06-B	A8329_012	Sediment
PH15-06-C	A8329_013	Sediment
PH15-07-D	A8329_014	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),*[1] dated October 14, 2015, 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 ± 2°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. Samples containing total HxCDD at concentrations below the reporting limit and less than five time the blank result were qualified as estimated and flagged "J" based on method blank results.

[1] NewFields. (2015). Portland Harbor Supplemental Sediment Study, Portland Oregon Quality Assurance Project Plan (QAPP). October 14, 2015.
[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,

Rinsate Blank: Three rinsate blanks were collected on October 21, 2015, October 22, 2015, and October 23, 2015 (PH15-01-RB, PH15-02-RB, PH15-03-RB, respectively [SDG A8328]) and are associated with the samples in this ETR.

- PH15-01-RB is associated with: PH15-03-A, PH15-03-B, PH15-03-C, PH15-03-D, PH15-06-A, PH15-06-B, PH15-06-C, and PH15-07-D
- PH15-02-RB is associated with: PH15-14-A, PH15-14-FD, PH15-21-A, PH15-21-C, and PH15-21-D
- PH15-03-RB is associated with: PH15-12-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 (%)
PH15-06-B	ES 123678-HxCDF	132	28 – 130
PH15-06-B	CS 123469-HxCDF	150	40 – 130
PH15-06-B	CS 1234689-HpCDF	138	25 – 130

The surrogate recovery for surrogate compounds listed above were above the acceptance criteria. The results of the compounds associated with ES 123678-HxCDF were qualified as not detected, and were "U" qualified. The results of the compounds associated with CS 123469-HxCDF and CS 1234689-HpCDF 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 PH15-14-A and was identified as PH15-14-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	1234678-HpCDD	147	50
		OCDD	172	50
		1234678-HpCDF	125	50
		OCDF	141	50
		Total HxCDD	92.0	50
		Total HpCDD	138.0	50
		Total TCDF	85.5	50
		Total PeCDF	71.8	50
		Total HxCDF	58.4	50

Sample ID	Field Duplicate ID	Analyte	RPD (%)	QC Limit (%)
		Total HpCDF	127.0	50

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

Laboratory Duplicate– 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 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
A8329	PH15-03-C	A8329_13707_DF_009-D5	EPA 1613B	Total HpCDD	1.43	EMPC	pg/g		J	bl
A8329	PH15-06-B	A8329_13707_DF_012_CU1-D5	EPA 1613B	123678-HxCDF	0	U	pg/g		UJ	S
A8329	PH15-06-B	A8329_13707_DF_012_CU1-D5	EPA 1613B	Total HpCDF	17.4	EMPC	pg/g		J	S
A8329	PH15-06-B	A8329_13707_DF_012_CU1-D5	EPA 1613B	Total HxCDF	3.55	EMPC	pg/g		J	S
A8329	PH15-06-C	A8329_13707_DF_013-D5	EPA 1613B	Total HxCDD	3.57	EMPC	pg/g		J	bl
A8329	PH15-07-D	A8329_13707_DF_014-D5	EPA 1613B	Total HxCDD	0.215		pg/g		J	bl
A8329	PH15-14-A	A8329_13707_DF_001-D5	EPA 1613B	Total HxCDF	18.8		pg/g		J	fd
A8329	PH15-14-A	A8329_13707_DF_001-D5	EPA 1613B	1234678-HpCDD	167		pg/g		J	fd
A8329	PH15-14-A	A8329_13707_DF_001-D5	EPA 1613B	1234678-HpCDF	22.7		pg/g		J	fd
A8329	PH15-14-A	A8329_13707_DF_001-D5	EPA 1613B	OCDD	2720		pg/g		J	fd
A8329	PH15-14-A	A8329_13707_DF_001-D5	EPA 1613B	OCDF	80.5		pg/g		J	fd
A8329	PH15-14-A	A8329_13707_DF_001-D5	EPA 1613B	Total HpCDD	292		pg/g		J	fd
A8329	PH15-14-A	A8329_13707_DF_001-D5	EPA 1613B	Total HxCDD	23.8	EMPC	pg/g		J	fd
A8329	PH15-14-A	A8329_13707_DF_001-D5	EPA 1613B	Total PeCDF	6.22	EMPC	pg/g		J	fd
A8329	PH15-14-A	A8329_13707_DF_001-D5	EPA 1613B	Total TCDF	5.06	EMPC	pg/g		J	fd
A8329	PH15-14-A	A8329_13707_DF_001-D5	EPA 1613B	Total HpCDF	61.8		pg/g		J	fd
A8329	PH15-14-A-FD	A8329_13707_DF_002-D5	EPA 1613B	Total TCDF	1.84		pg/g		J	fd
A8329	PH15-14-A-FD	A8329_13707_DF_002-D5	EPA 1613B	1234678-HpCDD	25.7		pg/g		J	fd
A8329	PH15-14-A-FD	A8329_13707_DF_002-D5	EPA 1613B	1234678-HpCDF	5.25		pg/g		J	fd
A8329	PH15-14-A-FD	A8329_13707_DF_002-D5	EPA 1613B	OCDD	208		pg/g		J	fd
A8329	PH15-14-A-FD	A8329_13707_DF_002-D5	EPA 1613B	OCDF	14		pg/g		J	fd
A8329	PH15-14-A-FD	A8329_13707_DF_002-D5	EPA 1613B	Total HpCDD	53.6		pg/g		J	fd
A8329	PH15-14-A-FD	A8329_13707_DF_002-D5	EPA 1613B	Total HpCDF	14.6	EMPC	pg/g		J	fd
A8329	PH15-14-A-FD	A8329_13707_DF_002-D5	EPA 1613B	Total HxCDD	10.4	EMPC	pg/g		J	fd
A8329	PH15-14-A-FD	A8329_13707_DF_002-D5	EPA 1613B	Total HxCDF	10.5	EMPC	pg/g		J	fd
A8329	PH15-14-A-FD	A8329_13707_DF_002-D5	EPA 1613B	Total PeCDF	5.29	EMPC	pg/g		J	fd

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