

Data Validation Report

Project: Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling

Laboratory: SGS-AXYS, Sydney, British Columbia, Canada

Laboratory Group: WG66481-DX

Analyses/Method: Dioxins and Furans by HRGC/HRMS / E1613

Validation Level: Stage 2A

AECOM Project Number: 60566335.2.12

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SUMMARY

The samples listed below were collected by AECOM in Portland Harbor in Portland, OR on November 27-30, 2018 and December 1, 2018.

Sample ID	Matrix/Sample Type
PDI-RB-XF-181129	Equipment Blank
PDI-WS-T01-1811	Surface Water
PDI-WS-T02-1811	Surface Water
PDI-WS-T03-1811	Surface Water
PDI-WS-T04-1812	Surface Water
PDI-WS-T05-1811	Surface Water
PDI-WS-T06-1811	Surface Water
PDI-WS-T07-1811	Surface Water

Data validation activities were conducted with reference to:

- EPA Method 1613B: *Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS (October 1994)*,
- USEPA Contract Laboratory Program National Functional Guidelines for High Resolution Superfund Methods Data Review (April 2016),
- Quality Assurance Project Plan, Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling, Portland Harbor Superfund Site (March 2018), and the
- laboratory quality control (QC) limits.

The National Functional Guidelines were modified to accommodate the non-CLP methodologies. In the absence of method-specific information, laboratory QC limits, project-specific requirements and/or AECOM professional judgment were used as appropriate.

REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

- ✓ Data completeness (chain-of-custody (COC)/sample integrity)
- ✓ Holding times and sample preservation
- ✗ Laboratory blanks/equipment blanks
- NA Matrix spike (MS) and/or matrix spike duplicate (MSD) results
- ✓ Ongoing precision and recovery (OPR) results
- NA Field duplicate results
- ✗ Labeled compound and clean-up standard recoveries
- ✗ Sample results/reporting issues

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. An NA indicates that the parameter was not included as part of this data set or was not applicable to this validation and therefore not reviewed. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

The data appear valid as qualified and may be used for decision making purposes. Select data points were qualified as estimated due to nonconformances of certain QC criteria (see discussion below). Qualified sample results are presented in Table 1.

RESULTS

Data Completeness (COC)/Sample Integrity

The data package was reviewed and found to meet acceptance criteria for completeness:

- The COCs were reviewed for completeness of information relevant to the samples and requested analyses, and for signatures indicating transfer of sample custody.
- The laboratory sample login sheet(s) were reviewed for issues potentially affecting sample integrity, including the condition of sample containers upon receipt at the laboratory.
- Completeness of analyses was verified by comparing the reported results to the COC requests. :

Holding Times and Sample Preservation

Sample preservation and preparation/analysis holding times were reviewed for conformance with method criteria. All method QC acceptance criteria were met.

Laboratory Blanks/Equipment Blanks

Laboratory method blanks and equipment blank results are evaluated as to whether there are contaminants detected above the estimated detection limit (EDL).

Target compounds were detected in the laboratory method blank and equipment blank associated with the samples in this data set.

Detected compounds are summarized in Attachment A in Table A-1 and Table A-2. The results for the equipment blank PDI-RB-XF-181129 are provided for informational purposes only.

The NFG guidance stipulates that a conservative approach should be taken with regards to qualification of dioxins due to the toxicity of these compounds and the reporting of false negative results should be avoided.

Therefore, in order to avoid the reporting of false negative results, professional judgment was used to qualify the data in the following manner on the basis of laboratory method blank contamination. As allowed in the NFG, a blank action limit (BAL) was determined as five times the blank result:

- When the sample results were < the blank result, the sample result was qualified as nondetect (U) at the sample result.
- When the sample result was \geq the blank result and \leq the BAL, the sample result was qualified as estimated and potentially biased high (J+).
- When the sample result was > the BAL, the sample result was not qualified.

Qualified sample results are summarized in Table 1.

MS/MSD Results

MS/MSD analyses were not performed on a sample in this data set. No data validation actions were taken on this basis.

OPR Results

The OPR %Rs were reviewed for conformance with the method QC acceptance criteria. All method QC acceptance criteria were met.

The laboratory noted in the case narrative that the solvent went to dryness for the OPR and samples PDI-WS-T06-1811 and PDI-RB-XF-181129 during sample extraction. A new OPR was created and extracted, replacing the original OPR. The OPR was re-united with the batch and the clean-up of the samples continued. Additional sample was not available for the re-extraction of these samples. No data validation actions were taken on this basis.

Field Duplicate Results

A field duplicate pair was not submitted with this data set. No data validation actions were taken on this basis.

Labeled Compound and Clean-up Standard Recoveries

The labeled compounds and labeled clean-up standard %Rs were reviewed for conformance with the QC acceptance criteria. The laboratory noted in the case narrative that the solvent went dry during the extraction procedure of samples PDI-WS-T06-1811 and PDI-RB-XF-181129 along with the associated OPR. Additional sample was not available for the re-extraction of these samples. However, given that the extract went dry after the addition of the labeled extraction standards and that isotope dilution quantification corrects for such losses, data are not considered significantly impacted. All labeled extraction standards met criteria in sample PDI-WS-T06-1811.

Nonconformances noted for sample PDI-RB-XF-181129 are summarized in Attachment A in Table A-3. Samples were qualified as follows:

Actions: (Based on NFG 2016)

Criteria ¹		Actions ²	
		Detected	Nondetected
%R > Upper Acceptance Limit		J	UJ
%R >10% but < Lower Acceptance Limit		J	UJ
%R <10%		See below	
<10% and S/N >10:1		J	R
<10% and S/N <10:1		R	R
Ion abundance ratio criteria not met	Calibration compliant	J	UJ
	Calibration non-compliant	J	R
Clean-up Standard Recovery < Lower Acceptance Limit		J	UJ
¹ See Table 7 in method 1613B for acceptance criteria ² The dioxin method is performed using isotope dilution technique; therefore, professional judgment was applied and bias codes were not included in data qualification.			

Qualified sample results are summarized in Table 1.

Sample Results/Reporting Issues

All sample results detected at concentrations less than the lowest calibration standard but greater than the EDL are qualified by the laboratory as estimated (J). This "J" qualifier is retained during data validation.

It should be noted that the sample reported detection limit is the sample specific estimated detection limit (EDL) with the following exceptions. In cases where the EDL is less than the nominal concentration of 0.5 pg/sample, the EDL is raised to the nominal concentration of 0.5 pg/sample and is adjusted to include the appropriate preparation factors.

Second Column Confirmation (2,3,7,8-TCDF)

The sample data were reviewed to ensure that results for 2,3,7,8-TCDF when analyzed on a DB-5 (or equivalent) column were confirmed on a second column (i.e., DB-225 or equivalent) when isomer specificity is not achieved. All 2,3,7,8-TCDF results were reported from the confirmation column. Qualification of the data was not required.

Additionally, the laboratory confirmed the results for 1,2,3,7,8,9-HxCDD on a second column (i.e., DB-225 or equivalent) in cases where this compound is detected on the DB-5 (or equivalent) column. All 1,2,3,7,8,9-HxCDD results were reported from the confirmation column.

Estimated Maximum Possible Concentrations (EMPCs)

The data were reviewed to identify sample results that were indicated by the laboratory to be estimated maximum possible concentrations (EMPCs) because of identification criteria not being met.

The laboratory identified the presence of EMPCs for the samples in this data set by qualifying affected results with a K laboratory qualifier. Samples were qualified as follows:

Actions: (Based on AECOM professional judgment)

Criteria	Actions
A native target compound was reported by the laboratory as an EMPC.	Report result as an EMPC and qualify as estimated and presumptively present (JN).
A labeled compound was flagged by the laboratory indicating all identification criteria were not met.	Qualify associated positive and nondetect results as estimated (J/UJ).

It should be noted that in instances of multiple nonconformances, the bias is considered indeterminate where there is a conflicting low and high bias or when a result does not exhibit a consistent bias. These results have an overall qualification of estimated (J) with the exception noted below.

When applicable, the "JN" qualifier was retained rather than replacement with the conventional overall "J" qualifier in instances where EMPC results were qualified for multiple quality control nonconformances.

Qualified sample results are summarized in Table 1.

QUALIFICATION ACTIONS

Sample results qualified as a result of validation actions are summarized in Table 1. All actions are described above.

ATTACHMENTS

Attachment A: Nonconformance Summary Tables

Attachment B: Qualifier Codes and Explanations

Attachment C: Reason Codes and Explanations

Table 1 - Data Validation Summary of Qualified Data

Sample ID	Matrix	Compound	Result	EDL	Units	Validation Qualifiers	Validation Reason
PDI-RB-XF-181129	WS	1,2,3,4,6,7,8-HpCDD	2.17	0.841	pg/sample	JN	k
PDI-RB-XF-181129	WS	1,2,3,4,7,8,9-HpCDF	1.77	0.841	pg/sample	JN	lc,k
PDI-RB-XF-181129	WS	OCDF	3.44	1.26	pg/sample	J+	bl
PDI-WS-T01-1811	WS	1,2,3,4,7,8,9-HpCDF	2.63	0.862	pg/sample	JN	k
PDI-WS-T01-1811	WS	1,2,3,4,7,8-HxCDD	3.72	1.09	pg/sample	JN	k
PDI-WS-T01-1811	WS	1,2,3,6,7,8-HxCDF	1.73	0.862	pg/sample	JN	k
PDI-WS-T01-1811	WS	1,2,3,7,8,9-HxCDD	8.47	1.12	pg/sample	JN	k
PDI-WS-T01-1811	WS	1,2,3,7,8-PeCDD	1.06	0.862	pg/sample	JN	k
PDI-WS-T01-1811	WS	2,3,4,6,7,8-HxCDF	1.39	0.862	pg/sample	JN	k
PDI-WS-T01-1811	WS	2,3,7,8-TCDD	1.10	0.862	pg/sample	JN	k
PDI-WS-T01-1811	WS	2,3,7,8-TCDF	2.34	0.862	pg/sample	JN	k
PDI-WS-T02-1811	WS	1,2,3,4,7,8,9-HpCDF	3.21	1.20	pg/sample	JN	k
PDI-WS-T02-1811	WS	1,2,3,7,8,9-HxCDD	13.4	1.36	pg/sample	JN	k
PDI-WS-T02-1811	WS	2,3,4,6,7,8-HxCDF	2.08	0.884	pg/sample	JN	k
PDI-WS-T02-1811	WS	2,3,4,7,8-PeCDF	1.82	0.884	pg/sample	JN	k
PDI-WS-T02-1811	WS	2,3,7,8-TCDD	1.10	0.884	pg/sample	JN	k
PDI-WS-T02-1811	WS	2,3,7,8-TCDF	3.79	0.884	pg/sample	JN	k
PDI-WS-T03-1811	WS	1,2,3,4,7,8-HxCDD	4.21	1.11	pg/sample	JN	k
PDI-WS-T03-1811	WS	1,2,3,7,8,9-HxCDD	10.2	1.52	pg/sample	JN	k
PDI-WS-T03-1811	WS	2,3,4,7,8-PeCDF	1.21	0.842	pg/sample	JN	k
PDI-WS-T04-1812	WS	1,2,3,4,7,8,9-HpCDF	1.35	0.980	pg/sample	JN	k
PDI-WS-T04-1812	WS	1,2,3,4,7,8-HxCDD	1.50	0.872	pg/sample	JN	k
PDI-WS-T04-1812	WS	1,2,3,6,7,8-HxCDF	1.28	0.867	pg/sample	JN	k
PDI-WS-T04-1812	WS	1,2,3,7,8,9-HxCDD	5.92	1.79	pg/sample	JN	k
PDI-WS-T04-1812	WS	1,2,3,7,8-PeCDF	1.22	0.867	pg/sample	JN	k
PDI-WS-T04-1812	WS	2,3,4,6,7,8-HxCDF	0.953	0.867	pg/sample	JN	k
PDI-WS-T04-1812	WS	2,3,7,8-TCDD	1.01	0.867	pg/sample	JN	k
PDI-WS-T05-1811	WS	1,2,3,4,7,8-HxCDF	3.61	0.868	pg/sample	JN	k
PDI-WS-T05-1811	WS	1,2,3,6,7,8-HxCDF	2.69	0.868	pg/sample	JN	k
PDI-WS-T05-1811	WS	1,2,3,7,8,9-HxCDD	13.4	1.55	pg/sample	JN	k
PDI-WS-T05-1811	WS	1,2,3,7,8-PeCDD	1.66	1.18	pg/sample	JN	k
PDI-WS-T05-1811	WS	2,3,4,6,7,8-HxCDF	2.23	0.868	pg/sample	JN	k
PDI-WS-T06-1811	WS	1,2,3,4,7,8,9-HpCDF	2.07	1.61	pg/sample	JN	k
PDI-WS-T06-1811	WS	1,2,3,4,7,8-HxCDD	2.41	1.03	pg/sample	JN	k
PDI-WS-T06-1811	WS	1,2,3,4,7,8-HxCDF	1.94	0.846	pg/sample	JN	k
PDI-WS-T06-1811	WS	1,2,3,7,8,9-HxCDD	7.69	1.16	pg/sample	JN	k

Sample ID	Matrix	Compound	Result	EDL	Units	Validation Qualifiers	Validation Reason
PDI-WS-T06-1811	WS	2,3,7,8-TCDD	1.97	0.846	pg/sample	JN	k
PDI-WS-T07-1811	WS	1,2,3,4,7,8,9-HpCDF	2.83	0.831	pg/sample	JN	k
PDI-WS-T07-1811	WS	1,2,3,4,7,8-HxCDF	2.01	0.831	pg/sample	JN	k
PDI-WS-T07-1811	WS	1,2,3,7,8,9-HxCDD	7.51	1.23	pg/sample	JN	k
PDI-WS-T07-1811	WS	1,2,3,7,8-PeCDD	0.944	0.831	pg/sample	JN	k
PDI-WS-T07-1811	WS	1,2,3,7,8-PeCDF	0.921	0.831	pg/sample	JN	k
PDI-WS-T07-1811	WS	2,3,4,7,8-PeCDF	0.937	0.831	pg/sample	JN	k
PDI-WS-T07-1811	WS	2,3,7,8-TCDD	1.05	0.831	pg/sample	JN	k
PDI-WS-T07-1811	WS	2,3,7,8-TCDF	0.864	0.831	pg/sample	JN	k

Attachment A

Nonconformance Summary Tables

Table A-1 - Lab Blanks

Blank ID	Compound	Result	QL	BAL	Units	Associated Samples
WG66481-101	OCDD	3.34	34.5	16.7	pg/sample	PDI-RB-XF-181129
WG66481-101	OCDF	1.51	34.5	7.55	pg/sample	PDI-WS-T01-1811 PDI-WS-T02-1811 PDI-WS-T03-1811 PDI-WS-T04-1812 PDI-WS-T05-1811 PDI-WS-T06-1811 PDI-WS-T07-1811

Table A-2 - Field Blanks

Blank ID	Compound	Result	QL	Units	Associated Samples
PDI-RB-XF-181129	OCDD	21.7	33.6	pg/sample	PDI-WS-T01-1811
	1,2,3,4,6,7,8-HpCDD	2.17	16.8	pg/sample	PDI-WS-T02-1811
	OCDF	3.44	33.6	pg/sample	PDI-WS-T03-1811 PDI-WS-T04-1812
	1,2,3,4,7,8,9-HpCDF	1.77	16.8	pg/sample	PDI-WS-T05-1811 PDI-WS-T06-1811 PDI-WS-T07-1811

Table A-3 - Labeled Compound Recoveries

Sample ID	Labeled Compound	% Recovery	Lower Limit	Upper Limit
PDI-RB-XF-181129	13C-1,2,3,4,7,8,9-HpCDF	22.3	26	138

Attachment B
Qualifier Codes and Explanations

Qualifier	Explanation
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
J-	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample with a potential low bias.
J+	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample with a potential high bias.
JN	The analyte was tentatively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Attachment C

Reason Codes and Explanations

Reason Code	Explanation
be	Equipment blank contamination
bf	Field blank contamination
bl	Laboratory blank contamination
c	Calibration issue
cl	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
lc	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