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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling	
Laboratory:	SGS-AXYS, Sydney, British Columbia, Canada	
Laboratory Group:	WG66477-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-XD-181129	Surface Water
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 or negated 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.

Compounds detected in the laboratory method blank are summarized in Attachment A in Table A-1. There were no compounds detected in the equipment blank PDI-RB-XD-181129 after method blank actions (as described below) were applied.

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.

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 clean-up procedure of sample PDI-WS-T02-1811. Additional sample was not available for the re-extraction of this sample. 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 compounds met criteria in sample PDI-WS-T02-1811.

Nonconformances for samples other than PDI-S-T02-1811 are summarized in Attachment A in Table A-2.

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.

The laboratory spikes the XAD resin with 13C6-1,2,3,4-TCDD prior to deployment. Specific QC acceptance limits have not been established for this compound. However, the recoveries of this labeled compound in all samples were found to range between 88 and 106%. Consequently, it was determined that the XAD resin performance was acceptable for this sample event and data were not qualified on this basis.

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.

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-XD-181129	WS	OCDD		2.56	pg/sample	U	bl
PDI-WS-T01-1811	WS	1,2,3,4,6,7,8-HpCDD	4.87	0.851	pg/sample	J+	bl
PDI-WS-T01-1811	WS	OCDF		1.48	pg/sample	U	bl
PDI-WS-T02-1811	WS	1,2,3,4,6,7,8-HpCDD	2.51	0.926	pg/sample	JN	bl,k
PDI-WS-T02-1811	WS	OCDD	36.2	1.52	pg/sample	JN	bl,k
PDI-WS-T02-1811	WS	OCDF		1.47	pg/sample	U	bl
PDI-WS-T03-1811	WS	1,2,3,4,6,7,8-HpCDD	8.21	0.849	pg/sample	J+	bl
PDI-WS-T03-1811	WS	1,2,3,6,7,8-HxCDD		0.849	pg/sample	UJ	lc
PDI-WS-T03-1811	WS	1,2,3,7,8,9-HxCDD		0.849	pg/sample	UJ	lc
PDI-WS-T03-1811	WS	OCDF		2.26	pg/sample	U	bl
PDI-WS-T04-1812	WS	1,2,3,4,6,7,8-HpCDD	2.46	1.11	pg/sample	J+	bl
PDI-WS-T04-1812	WS	1,2,3,6,7,8-HxCDD		0.847	pg/sample	UJ	lc
PDI-WS-T04-1812	WS	1,2,3,7,8,9-HxCDD		0.847	pg/sample	UJ	lc
PDI-WS-T04-1812	WS	OCDD	28.7	0.847	pg/sample	J+	bl
PDI-WS-T04-1812	WS	OCDF		1.77	pg/sample	U	bl
PDI-WS-T05-1811	WS	1,2,3,4,6,7,8-HpCDD	6.38	0.936	pg/sample	J+	bl
PDI-WS-T05-1811	WS	1,2,3,4,6,7,8-HpCDF	0.856	0.848	pg/sample	JN	k
PDI-WS-T05-1811	WS	1,2,3,6,7,8-HxCDD		0.848	pg/sample	UJ	lc
PDI-WS-T05-1811	WS	1,2,3,7,8,9-HxCDD		0.848	pg/sample	UJ	lc
PDI-WS-T06-1811	WS	1,2,3,4,6,7,8-HpCDD	2.37	1.07	pg/sample	J+	bl
PDI-WS-T06-1811	WS	OCDD	30.3	2.70	pg/sample	J+	bl
PDI-WS-T06-1811	WS	OCDF		1.40	pg/sample	U	bl
PDI-WS-T07-1811	WS	1,2,3,4,6,7,8-HpCDD	3.18	1.00	pg/sample	JN	bl,k
PDI-WS-T07-1811	WS	1,2,3,6,7,8-HxCDD		0.851	pg/sample	UJ	lc
PDI-WS-T07-1811	WS	1,2,3,7,8,9-HxCDD		0.851	pg/sample	UJ	lc

Attachment A

Nonconformance Summary Tables

Table A-1 - Lab Blanks

Blank ID	Compound	Result	QL	BAL	Units	Associated Samples
WG66477-101	OCDD	7.58	33.9	37.9	pg/sample	PDI-RB-XD-181129 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
	1,2,3,4,6,7,8-HpCDD	1.81	16.9	9.05	pg/sample	
	OCDF	5.08	33.9	25.2	pg/sample	
	1,2,3,4,7,8-HxCDD	1.17	16.9	5.85	pg/sample	
	1,2,3,4,7,8,9-HpCDF	0.964	16.9	4.82	pg/sample	

Table A-2 - Labeled Compound Recoveries

Sample ID	Labeled Compound	% Recovery	Lower Limit	Upper Limit
PDI-WS-T07-1811	13C-1,2,3,6,7,8-HxCDD	138	28	130
PDI-WS-T05-1811	13C-1,2,3,6,7,8-HxCDD	156	28	130
PDI-WS-T03-1811	13C-1,2,3,6,7,8-HxCDD	133	28	130
PDI-WS-T04-1812	13C-1,2,3,6,7,8-HxCDD	136	28	130

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