AECOM
250 Apollo Drive
Chelmsford, MA 01824

978.905.2100 tel
978.905.2101 fax

Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling
Laboratory:	SGS-AXYS, Sydney, British Columbia, Canada
Laboratory Group:	WG65583-DX
Analyses/Method:	Dioxins and Furans by HRGC/HRMS / E1613
Validation Level:	Stage 4
AECOM Project Number:	60566335.2.12
Prepared by:	Paula DiMattei/AECOM Completed on: 12/18/2018
Reviewed by:	Elissa McDonagh/AECOM File Name: WG65583-DX DVR

SUMMARY

The samples listed below were collected by AECOM in Portland Harbor in Portland, OR on August 20-25, 2018.

Sample ID	Matrix/Sample Type
PDI-RB-XF-180820	Equipment Blank
PDI-WS-T01-1808	Surface Water
PDI-WS-T02-1808	Surface Water
PDI-WS-T03-1808	Surface Water
PDI-WS-T04-1808	Surface Water
PDI-WS-T05-1808	Surface Water
PDI-WS-T06-1808	Surface Water
PDI-WS-T07-1808	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
✓	Mass resolution/ window defining mix (WDM)/isomer specificity check (ISC) results
✓	Initial calibration/continuing calibration verification
✗	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.

Mass Resolution/ WDM/ISC Results

The data were reviewed to ensure that

- the perfluorokerosene (PFK) molecular leak was performed at the correct frequency (at the beginning and end of a 12-hour shift) and the mass resolution was at a resolving power of > 10,000;
- the window defining mix (WDM) containing the first and last eluting isomers in each homologous series was analyzed at the correct frequency; and
- the isomer specificity check (ISC) standard criteria were met for the chromatographic resolution of 2,3,7,8-TCDD on the DB-5 column and of 2,3,7,8-TCDF on the DB-225 column.

All method QC acceptance criteria were met.

Initial Calibration/Continuing Calibration Verification

The data were reviewed to ensure that

- the absolute and relative retention time, signal/noise (S/N), and ion abundance ratio method acceptance criteria were met (as summarized by the laboratory);
- the initial calibration percent relative standard deviation (%RSD) method acceptance criteria were met for all native and labeled compounds; and
- the calibration verification standard (VER) method acceptance criteria were met.

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-180820 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 percent recoveries (%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. All method QC acceptance criteria were met.

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.

Laboratory Duplicate Analysis

The laboratory was unable to extract the entire number of filters received for each sample due to limitations of their Dean Stark apparatus. Approximately $1/5^{th}$ of each homogenized original filter sample was spiked with labeled standards and extracted rather than the entire amount that was collected. Consequently, a laboratory duplicate analysis was performed to ensure that the results achieved were representative of the entire sample.

Professional judgement was applied to use a relative percent difference criterion of <20% for results greater than five times the quantitation limit. All QC acceptance criteria were met.

Compound Identification

The data were reviewed to ensure that

- the retention time, relative retention time, ion abundance ratios, SIM ion co-maximization, and S/N method acceptance criteria were met for compound identification; and
- the quantitative determination of PCDFs were not affected by the presence of polychlorinated diphenyl ether (PCDPE) interferences detected above the 2.5:1 S/N ratio limit.

Samples were qualified as follows:

Actions: (Based on NFG 2016 and 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).
PCDPE interferences exist at the RT or a target compound furan	Consider the magnitude of the PCDPE and the target analyte. If the raw abundance of the PCDPE interference is significant (i.e., >10% of that for the associated target CDF analytes), use professional judgment to qualify the affected target CDF either as ND (U) at the EDL or unusable (R). If interference is minor (i.e., ≤10% of the associated target CDF), qualify detects as estimated (J) and nondetects as (UJ).

Qualified sample results are shown in Table 1.

It should be noted that in instances of multiple nonconformances, the bias is considered indeterminate in cases where a conflicting low and high bias exists 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. In these cases, bias is indeterminate.

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.

The laboratory indicated on the quantitation report that the peak for 1,2,3,7,8,9-HxCDD in samples PDI-WS-T01-1808 and PDI-WS-T06-1808 were on the tail or shoulder of a larger peak and there was no obvious valley between the two peaks. Consequently, these results were considered EMPCs. It should be noted that in these cases the ion ratio did meet criteria, but due to the noted interferences, these instances were considered to be EMPCs. These results were qualified as estimated and tentatively identified (JN).

Lock Mass Interferences

The positive result for 1,2,3,7,8,9-HxCDD in sample PDI-WS-T05-1808 was qualified as estimated and potentially biased low (J-) as a result of ion suppression as indicated by the monitored lock mass. However, this result was also identified as an EMPC; therefore, as noted above, the overall qualification of this result was reported as estimated and tentatively identified (JN).

Verification of calculations was performed on a subset of the data as deemed appropriate. No discrepancies were noted.

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-180820	WQ	OCDD	18.9	4.49	pg/sample	J+	bl
PDI-WS-T01-1808	WS	1,2,3,7,8,9-HxCDD	9.86	4.31	pg/sample	JN	k
PDI-WS-T01-1808	WS	2,3,4,7,8-PeCDF	6.53	4.31	pg/sample	JN	k
PDI-WS-T01-1808	WS	2,3,7,8-TCDD	4.49	4.31	pg/sample	JN	k
PDI-WS-T02-1808	WS	1,2,3,7,8,9-HxCDD	13.1	4.34	pg/sample	JN	k
PDI-WS-T02-1808	WS	1,2,3,7,8-PeCDD	5.96	4.34	pg/sample	JN	k
PDI-WS-T03-1808	WS	1,2,3,7,8,9-HxCDD	17.5	4.35	pg/sample	JN	k
PDI-WS-T03-1808	WS	1,2,3,7,8-PeCDF	16.3	4.35	pg/sample	JN	k
PDI-WS-T03-1808	WS	2,3,7,8-TCDF	19.2	4.35	pg/sample	JN	k
PDI-WS-T04-1808	WS	1,2,3,4,7,8-HxCDD	6.29	4.33	pg/sample	JN	k
PDI-WS-T04-1808	WS	1,2,3,6,7,8-HxCDD	23.5	4.33	pg/sample	JN	k
PDI-WS-T04-1808	WS	1,2,3,7,8,9-HxCDD	16.2	4.33	pg/sample	JN	k
PDI-WS-T05-1808	WS	1,2,3,4,7,8-HxCDD	6.07	4.39	pg/sample	JN	k
PDI-WS-T05-1808	WS	1,2,3,7,8,9-HxCDD	8.14	4.39	pg/sample	JN	k,su
PDI-WS-T06-1808	WS	1,2,3,4,7,8-HxCDD	6.09	4.30	pg/sample	JN	k
PDI-WS-T06-1808	WS	1,2,3,7,8,9-HxCDD	11.8	4.30	pg/sample	JN	k
PDI-WS-T06-1808	WS	1,2,3,7,8-PeCDD	4.30	4.30	pg/sample	JN	k
PDI-WS-T06-1808	WS	2,3,7,8-TCDD	4.75	4.30	pg/sample	JN	k
PDI-WS-T07-1808	WS	1,2,3,7,8,9-HxCDD	13.0	4.25	pg/sample	JN	k

Attachment A

Nonconformance Summary Tables

Table A-1 - Lab Blanks

Blank ID	Compound	Result	QL	Units	Associated Samples
WG65583-101	OCDD	7.93	171	pg/sample	PDI-RB-XF-180820 PDI-WS-T01-1808 PDI-WS-T02-1808 PDI-WS-T03-1808 PDI-WS-T04-1808 PDI-WS-T05-1808 PDI-WS-T06-1808 PDI-WS-T07-1808

Table A-2 - Field Blanks

Blank ID	Compound	Result	QL	Units
PDI-RB-XF-180820	OCDD	18.9	180	pg/sample

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