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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling
Laboratory:	SGS AXYS Analytical Services Ltd, Sidney, BC, Canada
Laboratory Group:	WG65124-DX
Analyses/Method:	Clean Water Act - Dioxins and Furans / CWA1613B
Validation Level:	Stage 4
AECOM Project Number:	60566335.2.12
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Completed on:	01/14/2019
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File Name:	WG65124-DX DVR

SUMMARY

The samples listed below were collected by AECOM in Portland Harbor in Portland, OR on August 13, 14, and 15, 2018.

Sample ID	Matrix/Sample Type
PDI-TF-SMB063	Fish Tissue
PDI-TF-SMB073	Fish Tissue
PDI-TF-SMB114	Fish Tissue
PDI-TF-SMB115	Fish Tissue
PDI-TF-SMB116	Fish Tissue
PDI-TF-SMB118	Fish Tissue
PDI-TF-SMB121	Fish Tissue
PDI-TF-SMB122	Fish Tissue
PDI-TF-SMB123	Fish Tissue
PDI-TF-SMB124	Fish Tissue
PDI-TF-SMB125	Fish Tissue
PDI-TF-SMB126	Fish Tissue
PDI-TF-SMB127	Fish Tissue
PDI-TF-SMB131	Fish Tissue
PDI-TF-SMB134	Fish Tissue
PDI-TF-SMB135	Fish Tissue

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
NA	Matrix spike (MS) and/or matrix spike duplicate (MSD) results
✓	Ongoing precision and recovery (OPR) results
✓	Matrix duplicate (MD) 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.

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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;
- 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;
- The calibration verification standard (CCV) method acceptance criteria were met.

It should be noted that according to the laboratory's SOP, the following method modification was noted:

> *"M/Z channels 354/356 and 366/368 are used to confirm and quantify the native and surrogate penta-substituted dioxins, respectively; this change from the method's specification is made in the instrument method in order to avoid a persistent interference in the 356/358 and 368/370 M/Z channels. The theoretical ratio for the P5CDD M/M+2 ions is 0.61; therefore, the acceptance range is 0.52 - 0.70."*

No data validation actions were taken on this basis.

Laboratory Blanks

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

Target compounds were detected in the method blanks associated with the samples in this data set. Note, the laboratory does not qualify sample results "B" associated with method blank contamination. NFG guidance stipulates that a conservative approach should be taken with regards to qualification of PCDD/PCDFs 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. As allowed in the NFG, a blank action limit (BAL) was determined as 5 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 ≥ the blank result and ≤ the BAL, the sample result was qualified as estimated and potentially biased high (J+).
- When the sample result was > the BAL, 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 and/or RPDs were reviewed for conformance with the method QC acceptance criteria. All method QC acceptance criteria were met.

MD Results

MD RPDs were reviewed for conformance with the laboratory QC acceptance criteria of ≤ 40% [if one or both results were greater than five times the quantitation limit (QL)] for solid matrices. All method QC acceptance criteria were met.

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 or sample detection limit (SDL) are qualified by the laboratory as estimated (J). This "J" qualifier is retained during data validation.

As stated in the laboratory's case narrative, the following reporting issue was noted:

> *To account for instrument variability and lab background levels, it is SGS AXYS policy to report detection limits no lower than 0.5 pg absolute (0.000072 ug/Kg on a 10g sample ….The reported detection limit is the greater of the SDL and the 0.5 pg absolute reporting limit.*

This is also noted in the laboratory's SOP.

PFK Lock Mass

The laboratory confirmed that a lock mass is monitored for each instrument descriptor. All QC acceptance criteria were met with the following exception.

Sample results associated with lock mass interference, qualified by the laboratory with a "G", were reanalyzed by the laboratory on an isomer specific column (i.e., DB-225) with compliant results. Results reported from non-compliant column (i.e., DB-5) are not reportable. No data validation actions were taken on this basis.

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.

All QC acceptance criteria were met with the following exceptions. Sample results which don't meet all of the method stipulated qualitative identification criteria are considered to be Estimated Maximum Possible Concentrations (EMPCs). Details concerning sample results in this data set which did not meet these identification criteria are noted below along with any data qualifications, as applicable.

The laboratory qualified all EMPC sample results with a "K" laboratory qualifier to indicate that the ion ratio criterion was not met. All ion ratios were verified and affected sample results which did not meet the ion ratio criteria were qualified as estimated and tentatively identified (JN). Furthermore, sample results which do not meet relative retention time criteria were also qualified JN. Qualified sample results are shown in Table 1.

Furthermore, sample results which do not meet relative retention time method acceptance criteria were qualified as estimated and tentatively identified (JN).

It should be noted that the "JN" qualifier was retained rather than replacement with the conventional overall "J" qualifier in instances where sample results were qualified for multiple quality control nonconformances.

Second Column Confirmation (2,3,7,8-TCDF and 1,2,3,7,8,9-HxCDD)

The sample data were reviewed to ensure that results for 2,3,7,8-TCDF and 1,2,3,7,8,9-HxCDD 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 sample results requiring confirmation were confirmed and results were reported from the confirmation column.

Note, affected results associated with the primary column (DB-5) are set to “not reportable” in the database.

Percent Solids Content

Since the sample matrix was fish tissue, all sample results have been reported on a “wet weight” basis.

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	RDL	EDL	Units	Validation Qualifiers	Validation Reason
PDI-TF-SMB063	TF	1,2,3,4,7,8-HxCDD	0.000142	0.0000735	0.000235	ug/kg	JN	k
PDI-TF-SMB063	TF	1,2,3,6,7,8-HxCDD	0.000694	0.0000735	0.000191	ug/kg	JN	k
PDI-TF-SMB063	TF	1,2,3,7,8-PeCDF	0.000237	0.0000735	0.000191	ug/kg	JN	k
PDI-TF-SMB063	TF	OCDF	0.0000873	0.0000735	0.000368	ug/kg	JN	bl,k
PDI-TF-SMB073	TF	1,2,3,4,6,7,8-HpCDF	0.0000891	0.0000723	0.000174	ug/kg	JN	k
PDI-TF-SMB073	TF	1,2,3,7,8,9-HxCDD	0.000177	0.000149		ug/kg	JN	k
PDI-TF-SMB073	TF	2,3,4,6,7,8-HxCDF	0.000101	0.0000723	0.000145	ug/kg	JN	k
PDI-TF-SMB114	TF	1,2,3,7,8-PeCDD	0.000257	0.0000756	0.000378	ug/kg	JN	k
PDI-TF-SMB114	TF	OCDD	0.000196	0.0000756	0.00147	ug/kg	JN	bl,k
PDI-TF-SMB115	TF	1,2,3,6,7,8-HxCDD	0.000155	0.0000820	0.000213	ug/kg	JN	k
PDI-TF-SMB115	TF	1,2,3,7,8-PeCDD	0.000221	0.0000820	0.000410	ug/kg	JN	k
PDI-TF-SMB115	TF	2,3,4,7,8-PeCDF	0.000228	0.0000820	0.000230	ug/kg	JN	k
PDI-TF-SMB115	TF	2,3,7,8-TCDD	0.000150	0.0000820	0.000161	ug/kg	JN	k
PDI-TF-SMB115	TF	OCDD	0.000338	0.0000820	0.00159	ug/kg	J+	bl
PDI-TF-SMB116	TF	1,2,3,4,6,7,8-HpCDD	0.000141	0.0000745	0.000179	ug/kg	JN	k
PDI-TF-SMB116	TF	1,2,3,7,8-PeCDF	0.000127	0.0000745	0.000194	ug/kg	JN	k
PDI-TF-SMB116	TF	OCDD	0.000261	0.0000745	0.00145	ug/kg	J+	bl
PDI-TF-SMB118	TF	1,2,3,6,7,8-HxCDD	0.000144	0.0000779	0.000203	ug/kg	JN	k
PDI-TF-SMB118	TF	1,2,3,7,8-PeCDF	0.000103	0.0000779	0.000203	ug/kg	JN	k
PDI-TF-SMB118	TF	2,3,7,8-TCDD	0.000233	0.0000779	0.000153	ug/kg	JN	k
PDI-TF-SMB118	TF	OCDD	0.000357	0.0000779	0.00151	ug/kg	J+	bl
PDI-TF-SMB121	TF	1,2,3,6,7,8-HxCDD	0.000138	0.0000699	0.000182	ug/kg	JN	k
PDI-TF-SMB121	TF	1,2,3,7,8-PeCDD	0.000277	0.0000699	0.000349	ug/kg	JN	k
PDI-TF-SMB121	TF	1,2,3,7,8-PeCDF	0.0000907	0.0000699	0.000182	ug/kg	JN	k
PDI-TF-SMB121	TF	2,3,4,7,8-PeCDF	0.000196	0.0000699	0.000196	ug/kg	JN	k
PDI-TF-SMB121	TF	2,3,7,8-TCDD	0.000312	0.0000699	0.000137	ug/kg	JN	rt
PDI-TF-SMB122	TF	1,2,3,4,6,7,8-HpCDD	0.0000845	0.0000699	0.000168	ug/kg	JN	k
PDI-TF-SMB122	TF	1,2,3,6,7,8-HxCDD	0.0000961	0.0000699	0.000182	ug/kg	JN	k
PDI-TF-SMB122	TF	2,3,7,8-TCDF	0.000357	0.0000699		ug/kg	JN	k
PDI-TF-SMB122	TF	OCDD	0.000166	0.0000699	0.00136	ug/kg	J+	bl
PDI-TF-SMB123	TF	1,2,3,4,6,7,8-HpCDF	0.0000877	0.0000730	0.000175	ug/kg	JN	k
PDI-TF-SMB123	TF	1,2,3,4,7,8-HxCDD	0.000137	0.0000730	0.000234	ug/kg	JN	k
PDI-TF-SMB123	TF	2,3,7,8-TCDD	0.000185	0.0000730	0.000143	ug/kg	JN	k
PDI-TF-SMB124	TF	1,2,3,4,6,7,8-HpCDF	0.0000924	0.0000719	0.000173	ug/kg	JN	k
PDI-TF-SMB124	TF	1,2,3,4,7,8-HxCDF	0.0000898	0.0000719	0.000187	ug/kg	JN	k
PDI-TF-SMB124	TF	1,2,3,6,7,8-HxCDD	0.000235	0.0000719	0.000187	ug/kg	JN	k
PDI-TF-SMB124	TF	2,3,4,7,8-PeCDF	0.000180	0.0000719	0.000201	ug/kg	JN	k
PDI-TF-SMB124	TF	2,3,7,8-TCDF	0.000315	0.0000719		ug/kg	JN	k

Sample ID	Matrix	Compound	Result	RDL	EDL	Units	Validation Qualifiers	Validation Reason
PDI-TF-SMB125	TF	1,2,3,4,6,7,8-HpCDF	0.0000945	0.0000719	0.000173	ug/kg	JN	k
PDI-TF-SMB125	TF	1,2,3,4,7,8-HxCDD	0.000110	0.0000719	0.000230	ug/kg	JN	k
PDI-TF-SMB125	TF	1,2,3,7,8,9-HxCDF	0.0000763	0.0000719	0.000259	ug/kg	JN	k
PDI-TF-SMB125	TF	1,2,3,7,8-PeCDD	0.000227	0.0000719	0.000359	ug/kg	JN	k
PDI-TF-SMB125	TF	1,2,3,7,8-PeCDF	0.0000874	0.0000719	0.000187	ug/kg	JN	k
PDI-TF-SMB125	TF	2,3,4,7,8-PeCDF	0.000169	0.0000719	0.000201	ug/kg	JN	k
PDI-TF-SMB125	TF	OCDF	0.000125	0.0000719	0.000359	ug/kg	JN	bl,k
PDI-TF-SMB126	TF	1,2,3,6,7,8-HxCDD	0.000149	0.0000731	0.000190	ug/kg	JN	k
PDI-TF-SMB126	TF	1,2,3,7,8-PeCDD	0.000383	0.0000731	0.000366	ug/kg	JN	k
PDI-TF-SMB126	TF	1,2,3,7,8-PeCDF	0.0000847	0.0000731	0.000190	ug/kg	JN	k
PDI-TF-SMB126	TF	OCDD	0.000116	0.0000731	0.00142	ug/kg	JN	bl,k
PDI-TF-SMB127	TF	1,2,3,4,6,7,8-HpCDD	0.0000912	0.0000769	0.000185	ug/kg	JN	k
PDI-TF-SMB127	TF	2,3,4,7,8-PeCDF	0.0000957	0.0000769	0.000215	ug/kg	JN	k
PDI-TF-SMB127	TF	2,3,7,8-TCDD	0.000180	0.0000769	0.000151	ug/kg	JN	k
PDI-TF-SMB127	TF	2,3,7,8-TCDF	0.000303	0.0000769		ug/kg	JN	k
PDI-TF-SMB127	TF	OCDD	0.000241	0.0000769	0.00149	ug/kg	JN	bl,k
PDI-TF-SMB131	TF	1,2,3,4,6,7,8-HpCDD	0.000189	0.0000794	0.000191	ug/kg	JN	k
PDI-TF-SMB131	TF	1,2,3,4,7,8-HxCDD	0.0000940	0.0000794	0.000254	ug/kg	JN	k
PDI-TF-SMB131	TF	1,2,3,6,7,8-HxCDD	0.000316	0.0000794	0.000206	ug/kg	JN	k
PDI-TF-SMB131	TF	OCDD	0.000216	0.0000794	0.00154	ug/kg	JN	bl,k
PDI-TF-SMB134	TF	1,2,3,4,6,7,8-HpCDD	0.000150	0.0000811	0.000195	ug/kg	JN	k
PDI-TF-SMB134	TF	1,2,3,4,7,8-HxCDD	0.0000850	0.0000811	0.000259	ug/kg	JN	k
PDI-TF-SMB134	TF	1,2,3,6,7,8-HxCDD	0.000334	0.0000811	0.000211	ug/kg	JN	k
PDI-TF-SMB134	TF	1,2,3,6,7,8-HxCDF	0.000106	0.0000811	0.0000892	ug/kg	JN	k
PDI-TF-SMB134	TF	2,3,4,7,8-PeCDF	0.000651	0.0000811	0.000227	ug/kg	JN	k
PDI-TF-SMB134	TF	2,3,7,8-TCDF	0.000410	0.0000811		ug/kg	JN	k
PDI-TF-SMB135	TF	1,2,3,4,6,7,8-HpCDD	0.000142	0.0000702	0.000169	ug/kg	JN	k
PDI-TF-SMB135	TF	1,2,3,4,7,8-HxCDF	0.000106	0.0000702	0.000183	ug/kg	JN	k
PDI-TF-SMB135	TF	1,2,3,6,7,8-HxCDD	0.000283	0.0000702	0.000183	ug/kg	JN	k
PDI-TF-SMB135	TF	1,2,3,7,8-PeCDD	0.000346	0.0000702	0.000351	ug/kg	JN	k
PDI-TF-SMB135	TF	2,3,7,8-TCDD	0.000185	0.0000702	0.000138	ug/kg	JN	k
PDI-TF-SMB135	TF	OCDD	0.000250	0.0000702	0.00136	ug/kg	J+	bl

Attachment A

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 B

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
ma	Multiple analyses, sample analyzed more than once, a value from another analysis should be used
nb	Negative laboratory blank contamination
p	Chemical preservation issue
r	Dual column RPD
rt	SIM ions not within + 2 seconds or not within relative retention time (RRT) window
q	Quantitation issue
s	Surrogate recovery
su	Ion suppression
t	Temperature preservation issue
x	Percent solids
y	Serial dilution results
z	ICS results