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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling
Laboratory:	Test America, West Sacramento, California
Laboratory Group:	580-76388-2
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:	06/24/2018
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File Name:	580-76388-2 DVR

SUMMARY

The samples listed below were collected by AECOM in Portland Harbor in Portland, OR on April 4 and 5, 2018.

Sample ID	Matrix/Sample Type
PDI-SG-B060-BL1-D	Field Duplicate of PDI-SG-B060-BL1
PDI-SG-B047-BL1	Sediment
PDI-SG-B048-BL1	Sediment
PDI-SG-B049-BL1	Sediment
PDI-SG-B050-BL1	Sediment
PDI-SG-B051-BL1	Sediment
PDI-SG-B052-BL1	Sediment
PDI-SG-B053-BL1	Sediment
PDI-SG-B055-BL1	Sediment
PDI-SG-B056-BL1	Sediment
PDI-SG-B057-BL1	Sediment
PDI-SG-B058-BL1	Sediment
PDI-SG-B060-BL1	Sediment
PDI-SG-B061-BL1	Sediment

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
✗	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 and/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.

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

It should be noted that according to section 17.1 of the laboratory's SOP, the following method modification was noted:

> Section 10.2.4 of Method 1613 reads "The absolute retention time of 13C-1,2,3,4-TCDD shall exceed 25.0 minutes on the DB-5 column, and the retention time of 13C-1,2,3,4-TCDD shall exceed 15.0 minutes on the DB-225 column; otherwise the GC temperature program shall be adjusted and this test repeated until the above-stated minimum retention time criteria are met."
>
> Our retention time on both columns deviate from the above method, but using section 1.5 of Method 1613 "the analyst is permitted to modify the method to lower the cost of measurements, provided that all performance criteria in this method are met," we have modified the GC program to provide a shorter runtime while still meeting method performance criteria thus lowering the cost of analysis.

No data validation actions were taken on this basis.

Nonconformances for the CCV relative retention time (RRT) criteria and CCV recovery standard RTs were noted and are summarized in Attachment A in Table A-1 and Table A-2, respectively.

Actions: (Based on NFG 2016)

Criteria	Actions	
Initial calibration	**Detected**	**Nondetected**
Initial calibration not performed	R	R
Initial calibration not performed at required frequency (but all other factors acceptable)	J	UJ
Ion abundance ratio exceeds QC limits	R*	R*

%RSD >20% for native compounds determined by isotope dilution %RSD >35% for 123789-HxCDD, OCDF, non-2378 substituted compounds and labeled compounds (EPA Method 1613B criteria)	J	UJ
Sensitivity <10:1 S/N ratio for all SICPs	J	R
RTs not within appropriate windows and absolute RT of internal standard $^{13}C_{12}$-1234-TCDD >25 minutes on DB-5 (or equivalent) or >15 minutes on DB-225 (or equivalent).	R	R
Calibration verification	**Detected**	**Nondetected**
CCV analysis not performed at the specified frequency or sequence	Use professional judgment	Use professional judgment
Ion abundance ratio exceeds QC limits	J	R
Absolute RT of internal standard $^{13}C_{12}$-1234-TCDD >25 minutes on DB-5 (or equivalent) or >15 minutes on DB-225 (or equivalent).	Use professional judgment	
Internal standards in the CCV not within 15 seconds of the RT in the initial calibration	Use professional judgment for qualification of target compounds; qualify homologues as estimated (J, UJ)	
RRT not within the specified QC limits	Use professional judgment	Use professional judgment
Sensitivity <10:1 S/N ratio for all SICPs	J	R
Acceptance criteria in Table 6 or Table 6a in method 1613B are not met	J	UJ
*If the ion abundance ratio is not within the QC limits for a compound in the CS1, qualify the low-end results for that compound (below the CS2 concentration) as unusable (R) or qualify as a nondetect at the level of the next lowest standard (i.e., CS2 standard). Flexibility is allowed since system baseline noise near the lower limit of detection may cause calibration peaks to fail even in an otherwise adequately performing system. However, if the ion abundance ratio is not within the limits for a compound in the CS3-CS5 standards, qualify all results for that compound as unusable (R).		

Professional judgment was used to take no action in instances where the labeled compounds were not within the RRT criterion since the required RRT criteria were met for all native compounds.

Additionally, professional judgment was used to take no action in instances where the recovery standard RT in the CCV was not within $\pm$ 15 seconds of the RT in the mid-level standard of the associated ICAL. These nonconformances result from routine column maintenance. A WDM is analyzed daily prior to sample analysis and retention times are adjusted accordingly; thus, data are not adversely impacted.

Laboratory Blanks/Equipment Blanks

Laboratory method blank and equipment blank results are evaluated as to whether there are contaminants detected above the estimated detection limit (EDL). An equipment blank was not submitted in this data set.

Target compounds were detected in the blanks associated with the samples in this data set.

Detected compounds are summarized in Attachment A in Table A-3.

The 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 $\geq$ 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, 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 RPDs were reviewed for conformance with the method QC acceptance criteria. All method QC acceptance criteria were met.

Field Duplicate Results

Field duplicate RPDs were reviewed for conformance with the AECOM QC acceptance criteria of $\leq$ 50% [if both results were greater than five times the quantitation limit (QL)] for solid matrices and $\leq$ 30% [if one or both results were greater than five times the QL] for aqueous matrices.

Nonconformances are summarized in Attachment A in Table A-4. Samples were qualified as follows:

Actions: (Based on AECOM professional judgment)

Criteria	RPD	Action	
		Detected	Nondetected
Sample and duplicate are nondetect results	Not calculable (NC)	No qualification	No qualification
Sample and duplicate results <QL	Not applicable	No qualification	No qualification
Sample and duplicate results $\geq$5xQL	>30% Aqueous >50% All other sample types	J	Not Applicable
Sample and duplicate results are >QL and <5xRL	>60% Aqueous >100% All other sample types	J	Not Applicable
If sample or duplicate result is >5xQL and the other is not detected	NC	J	UJ

Criteria	RPD	Action	
		Detected	Nondetected
If sample or duplicate result is <RL and the other is not detected	NC	No qualification	No qualification

Qualified sample results are summarized in Table 1.

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 overall bias is considered to be indeterminate in cases where cumulative nonconformances do not show a consistent bias or in cases of the presence of conflicting high and low biases.

PFK Lock Mass

The laboratory confirmed that a lock mass is monitored for each instrument descriptor. It should be noted that instrument 4D5 only displays three of the five monitored lock masses on the instrument raw data print out due to a graphics display issue.

The following compound in the samples listed was qualified as estimated and potentially biased low (J-) due ion suppression reflected in the associated lock mass.

1,2,3,7,8,9-HxCDD: PDI-SG-B052-BL1
1,2,3,7,8,9-HxCDF: PDI-SG-B047-BL1 and PDI-SG-B051-BL1

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 sample results with a "q" 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). Qualified sample results are shown in Table 1.

Nonconformances regarding the relative retention time identification criteria were observed for some sample results in this data set. Nonconformances are summarized in Attachment A in Table A-5.

The native compound results which did not meet the relative retention time (RRT) criteria were qualified as estimated and tentatively identified (JN) in the affected samples. Professional judgment was used to take no actions on the RRT nonconformances of the labeled compounds since all SICPs were reviewed to ensure that detected compounds were within the retention time windows established by the WDM analyses. Additionally, no peaks were observed prior to the expected elution of the first eluting peak or after the expected retention time of the last eluting peak in each window.

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.

The sample data were reviewed for the presence of polychlorinated diphenyl ethers (PCDPEs) which may interfere with the quantitative determination of any PCDFs. PCDPE interference was noted for the following compounds in the listed samples.

1,2,3,4,7,8,9-HpCDF: PDI-SG-B049-BL1, PDI-SG-B051-BL1, PDI-SG-B056-BL1

OCDF: PDI-SG-B050-BL1

These results were qualified as estimated and potentially biased high (J+).

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 sample results requiring confirmation were confirmed and results were reported from the confirmation column.

Percent Solids Content

The percent solids data were reviewed since the amount of moisture in a solid sample may have an impact on data representativeness. Due to the extremely low solubility of dioxins and furans in water, these analytes should be contained in the solid phase. Consequently, the NFG guidance does not stipulate a percent solids criterion. If applicable, EPA Regional guidance is used when assessing percent solids content. In the absence of EPA Regional guidance, AECOM uses 30% solids (from the NFG semivolatile guidance) as a benchmark to evaluate the percent solids content and professional judgment is used to determine the necessity to qualify data. Qualification on this basis was not required.

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-SG-B047-BL1	SE	1,2,3,4,6,7,8-HpCDF	0.012	0.00038	ug/kg	JN	k
PDI-SG-B047-BL1	SE	1,2,3,4,7,8,9-HpCDF	0.0011	0.00049	ug/kg	J+	bl
PDI-SG-B047-BL1	SE	1,2,3,4,7,8-HxCDD	0.00070	0.00017	ug/kg	JN	k,bl
PDI-SG-B047-BL1	SE	1,2,3,6,7,8-HxCDF	0.00092	0.00014	ug/kg	J+	bl
PDI-SG-B047-BL1	SE	1,2,3,7,8,9-HxCDD	0.0020	0.00014	ug/kg	J+	bl
PDI-SG-B047-BL1	SE	1,2,3,7,8,9-HxCDF	0.00075	0.00013	ug/kg	JN	k,bl,su
PDI-SG-B047-BL1	SE	1,2,3,7,8-PeCDD	0.00043	0.00014	ug/kg	J+	bl
PDI-SG-B047-BL1	SE	1,2,3,7,8-PeCDF	0.0011	0.00018	ug/kg	J+	bl
PDI-SG-B047-BL1	SE	2,3,4,6,7,8-HxCDF	0.00053	0.00012	ug/kg	J+	bl
PDI-SG-B047-BL1	SE	2,3,4,7,8-PeCDF	0.00073	0.00019	ug/kg	J+	bl
PDI-SG-B047-BL1	SE	2,3,7,8-TCDF	0.00089	0.000080	ug/kg	J+	bl
PDI-SG-B048-BL1	SE	1,2,3,4,6,7,8-HpCDF	0.00048	0.000061	ug/kg	JN	k,bl
PDI-SG-B048-BL1	SE	1,2,3,4,7,8,9-HpCDF		0.00021	ug/kg	U	bl
PDI-SG-B048-BL1	SE	1,2,3,4,7,8-HxCDD		0.00019	ug/kg	U	bl
PDI-SG-B048-BL1	SE	1,2,3,4,7,8-HxCDF		0.00013	ug/kg	U	bl
PDI-SG-B048-BL1	SE	1,2,3,6,7,8-HxCDD		0.00024	ug/kg	U	bl
PDI-SG-B048-BL1	SE	1,2,3,6,7,8-HxCDF		0.000066	ug/kg	U	bl
PDI-SG-B048-BL1	SE	1,2,3,7,8,9-HxCDD		0.00015	ug/kg	U	bl
PDI-SG-B048-BL1	SE	1,2,3,7,8,9-HxCDF		0.00025	ug/kg	U	bl
PDI-SG-B048-BL1	SE	2,3,7,8-TCDF	0.00025	0.00025	ug/kg	U	bl
PDI-SG-B048-BL1	SE	OCDF	0.0010	0.000070	ug/kg	J+	bl
PDI-SG-B049-BL1	SE	1,2,3,4,7,8,9-HpCDF	0.0012	0.00045	ug/kg	JN	k,bl,q
PDI-SG-B049-BL1	SE	1,2,3,4,7,8-HxCDD	0.0011	0.00028	ug/kg	J+	bl
PDI-SG-B049-BL1	SE	1,2,3,6,7,8-HxCDF	0.0012	0.00013	ug/kg	JN	k,bl
PDI-SG-B049-BL1	SE	1,2,3,7,8,9-HxCDF		0.00044	ug/kg	U	bl
PDI-SG-B049-BL1	SE	1,2,3,7,8-PeCDD	0.00049	0.00014	ug/kg	J+	bl
PDI-SG-B049-BL1	SE	2,3,4,6,7,8-HxCDF	0.00092	0.00011	ug/kg	J+	bl
PDI-SG-B050-BL1	SE	1,2,3,4,7,8-HxCDD		0.00015	ug/kg	U	bl
PDI-SG-B050-BL1	SE	1,2,3,4,7,8-HxCDF		0.00023	ug/kg	U	bl
PDI-SG-B050-BL1	SE	1,2,3,6,7,8-HxCDD	0.00043	0.000054	ug/kg	J+	bl
PDI-SG-B050-BL1	SE	1,2,3,6,7,8-HxCDF		0.00018	ug/kg	U	bl
PDI-SG-B050-BL1	SE	1,2,3,7,8,9-HxCDD		0.00032	ug/kg	U	bl
PDI-SG-B050-BL1	SE	1,2,3,7,8,9-HxCDF		0.00027	ug/kg	U	bl
PDI-SG-B050-BL1	SE	1,2,3,4,6,7,8-HpCDF	0.0012	0.00012	ug/kg	J+	bl
PDI-SG-B050-BL1	SE	1,2,3,7,8-PeCDF		0.00016	ug/kg	U	bl
PDI-SG-B050-BL1	SE	2,3,7,8-TCDF	0.00034	0.000047	ug/kg	J+	bl
PDI-SG-B050-BL1	SE	OCDF	0.0034	0.000079	ug/kg	J+	q

Sample ID	Matrix	Compound	Result	EDL	Units	Validation Qualifiers	Validation Reason
PDI-SG-B051-BL1	SE	1,2,3,4,7,8,9-HpCDF	0.0011	0.00044	ug/kg	J+	bl,q
PDI-SG-B051-BL1	SE	1,2,3,4,7,8-HxCDD	0.00070	0.00025	ug/kg	JN	k,bl
PDI-SG-B051-BL1	SE	1,2,3,4,7,8-HxCDF	0.0019	0.00022	ug/kg	J+	bl
PDI-SG-B051-BL1	SE	1,2,3,6,7,8-HxCDF	0.00079	0.00022	ug/kg	JN	k,bl
PDI-SG-B051-BL1	SE	1,2,3,7,8,9-HxCDD	0.0020	0.00021	ug/kg	J+	bl
PDI-SG-B051-BL1	SE	1,2,3,7,8,9-HxCDF	0.00073	0.00020	ug/kg	J	bl,su
PDI-SG-B051-BL1	SE	1,2,3,7,8-PeCDD	0.00030	0.00016	ug/kg	JN	k,bl
PDI-SG-B051-BL1	SE	1,2,3,7,8-PeCDF	0.00084	0.00016	ug/kg	J+	bl
PDI-SG-B051-BL1	SE	2,3,4,7,8-PeCDF	0.00078	0.00018	ug/kg	J+	bl
PDI-SG-B051-BL1	SE	2,3,7,8-TCDF	0.00096	0.000091	ug/kg	J+	bl
PDI-SG-B052-BL1	SE	1,2,3,4,7,8-HxCDD	0.0011	0.00021	ug/kg	JN	k,bl
PDI-SG-B052-BL1	SE	1,2,3,6,7,8-HxCDD	0.0037	0.00019	ug/kg	JN	k
PDI-SG-B052-BL1	SE	1,2,3,7,8,9-HxCDD	0.0018	0.00017	ug/kg	JN	k,bl,su
PDI-SG-B052-BL1	SE	1,2,3,7,8,9-HxCDF		0.00043	ug/kg	U	bl
PDI-SG-B052-BL1	SE	1,2,3,7,8-PeCDD	0.00039	0.000094	ug/kg	J+	bl
PDI-SG-B052-BL1	SE	2,3,4,6,7,8-HxCDF	0.00057	0.00016	ug/kg	JN	k,bl
PDI-SG-B052-BL1	SE	2,3,7,8-TCDD	0.00042	0.000021	ug/kg	J+	bl
PDI-SG-B053-BL1	SE	1,2,3,4,7,8,9-HpCDF		0.00030	ug/kg	U	bl
PDI-SG-B053-BL1	SE	1,2,3,4,7,8-HxCDD		0.00027	ug/kg	U	bl
PDI-SG-B053-BL1	SE	1,2,3,4,7,8-HxCDF	0.00065	0.000062	ug/kg	JN	k,bl
PDI-SG-B053-BL1	SE	1,2,3,6,7,8-HxCDD	0.00050	0.00011	ug/kg	JN	bl,k
PDI-SG-B053-BL1	SE	1,2,3,6,7,8-HxCDF		0.00022	ug/kg	U	bl
PDI-SG-B053-BL1	SE	1,2,3,7,8,9-HxCDD		0.00036	ug/kg	U	bl
PDI-SG-B053-BL1	SE	1,2,3,7,8,9-HxCDF		0.00026	ug/kg	U	bl
PDI-SG-B053-BL1	SE	1,2,3,7,8-PeCDF		0.00030	ug/kg	U	bl
PDI-SG-B053-BL1	SE	2,3,4,7,8-PeCDF		0.00018	ug/kg	U	bl
PDI-SG-B053-BL1	SE	2,3,7,8-TCDF	0.00042	0.000047	ug/kg	J+	bl
PDI-SG-B055-BL1	SE	1,2,3,4,6,7,8-HpCDF	0.0012	0.00011	ug/kg	JN	bl,k
PDI-SG-B055-BL1	SE	1,2,3,4,7,8,9-HpCDF		0.00040	ug/kg	U	bl
PDI-SG-B055-BL1	SE	1,2,3,4,7,8-HxCDD		0.00032	ug/kg	U	bl
PDI-SG-B055-BL1	SE	1,2,3,4,7,8-HxCDF		0.00031	ug/kg	U	bl
PDI-SG-B055-BL1	SE	1,2,3,6,7,8-HxCDD		0.00033	ug/kg	U	bl
PDI-SG-B055-BL1	SE	1,2,3,6,7,8-HxCDF		0.000097	ug/kg	U	bl
PDI-SG-B055-BL1	SE	1,2,3,7,8,9-HxCDD		0.00027	ug/kg	U	bl
PDI-SG-B055-BL1	SE	1,2,3,7,8,9-HxCDF		0.00036	ug/kg	U	bl
PDI-SG-B055-BL1	SE	2,3,7,8-TCDF		0.00024	ug/kg	U	bl
PDI-SG-B055-BL1	SE	OCDF	0.0027	0.000082	ug/kg	J+	bl
PDI-SG-B056-BL1	SE	1,2,3,4,6,7,8-HpCDF	0.0099	0.00028	ug/kg	JN	k
PDI-SG-B056-BL1	SE	1,2,3,4,7,8,9-HpCDF	0.00080	0.00039	ug/kg	J+	bl,q
PDI-SG-B056-BL1	SE	1,2,3,4,7,8-HxCDD	0.00084	0.00010	ug/kg	J+	bl

Sample ID	Matrix	Compound	Result	EDL	Units	Validation Qualifiers	Validation Reason
PDI-SG-B056-BL1	SE	1,2,3,4,7,8-HxCDF	0.0011	0.00016	ug/kg	JN	k,bl
PDI-SG-B056-BL1	SE	1,2,3,6,7,8-HxCDF	0.00054	0.00015	ug/kg	J+	bl
PDI-SG-B056-BL1	SE	1,2,3,7,8,9-HxCDD	0.0013	0.000089	ug/kg	J+	bl
PDI-SG-B056-BL1	SE	1,2,3,7,8,9-HxCDF		0.00045	ug/kg	U	bl
PDI-SG-B056-BL1	SE	1,2,3,7,8-PeCDD	0.00041	0.00011	ug/kg	JN	k,bl
PDI-SG-B056-BL1	SE	1,2,3,7,8-PeCDF		0.00053	ug/kg	U	bl
PDI-SG-B056-BL1	SE	2,3,4,6,7,8-HxCDF	0.00030	0.00010	ug/kg	JN	k,bl
PDI-SG-B056-BL1	SE	2,3,4,7,8-PeCDF		0.00032	ug/kg	U	bl
PDI-SG-B056-BL1	SE	2,3,7,8-TCDD	0.00030	0.00011	ug/kg	JN	k,bl
PDI-SG-B056-BL1	SE	2,3,7,8-TCDF	0.00079	0.00010	ug/kg	J+	bl
PDI-SG-B057-BL1	SE	1,2,3,4,7,8,9-HpCDF	0.00086	0.00019	ug/kg	JN	k,bl
PDI-SG-B057-BL1	SE	1,2,3,4,7,8-HxCDD		0.00045	ug/kg	U	bl
PDI-SG-B057-BL1	SE	1,2,3,6,7,8-HxCDD	0.0011	0.000091	ug/kg	JN	k,bl
PDI-SG-B057-BL1	SE	1,2,3,7,8,9-HxCDD	0.00086	0.000079	ug/kg	J+	bl
PDI-SG-B057-BL1	SE	1,2,3,7,8,9-HxCDF		0.00037	ug/kg	U	bl
PDI-SG-B057-BL1	SE	2,3,4,6,7,8-HxCDF	0.00071	0.00014	ug/kg	J+	bl
PDI-SG-B058-BL1	SE	1,2,3,4,6,7,8-HpCDF	0.0050	0.00017	ug/kg	JN	k
PDI-SG-B058-BL1	SE	1,2,3,4,7,8,9-HpCDF	0.00060	0.00021	ug/kg	J+	bl
PDI-SG-B058-BL1	SE	1,2,3,4,7,8-HxCDD		0.00045	ug/kg	U	bl
PDI-SG-B058-BL1	SE	1,2,3,4,7,8-HxCDF	0.00086	0.00013	ug/kg	J+	bl
PDI-SG-B058-BL1	SE	1,2,3,6,7,8-HxCDD	0.0011	0.000064	ug/kg	J+	bl
PDI-SG-B058-BL1	SE	1,2,3,6,7,8-HxCDF		0.00036	ug/kg	U	bl
PDI-SG-B058-BL1	SE	1,2,3,7,8,9-HxCDD	0.00067	0.000059	ug/kg	J+	bl
PDI-SG-B058-BL1	SE	1,2,3,7,8,9-HxCDF		0.00030	ug/kg	U	bl
PDI-SG-B058-BL1	SE	1,2,3,7,8-PeCDD	0.00028	0.000090	ug/kg	J+	bl
PDI-SG-B058-BL1	SE	1,2,3,7,8-PeCDF		0.00039	ug/kg	U	bl
PDI-SG-B058-BL1	SE	2,3,4,6,7,8-HxCDF	0.00021	0.000089	ug/kg	J+	bl
PDI-SG-B058-BL1	SE	2,3,4,7,8-PeCDF		0.00026	ug/kg	U	bl
PDI-SG-B058-BL1	SE	2,3,7,8-TCDF	0.00063	0.000088	ug/kg	J+	bl
PDI-SG-B060-BL1	SE	2,3,7,8-TCDF	0.0011	0.000056	ug/kg	J+	bl
PDI-SG-B060-BL1	SE	1,2,3,4,6,7,8-HpCDD	0.018	0.00023	ug/kg	J	fd
PDI-SG-B060-BL1	SE	1,2,3,4,7,8,9-HpCDF		0.00036	ug/kg	U	bl
PDI-SG-B060-BL1	SE	1,2,3,4,7,8-HxCDD		0.00027	ug/kg	U	bl
PDI-SG-B060-BL1	SE	1,2,3,4,7,8-HxCDF	0.00084	0.000086	ug/kg	JN	k,bl
PDI-SG-B060-BL1	SE	1,2,3,6,7,8-HxCDD	0.00077	0.000039	ug/kg	JN	k,bl
PDI-SG-B060-BL1	SE	1,2,3,6,7,8-HxCDF		0.00029	ug/kg	U	bl
PDI-SG-B060-BL1	SE	1,2,3,7,8,9-HxCDD		0.00038	ug/kg	U	bl
PDI-SG-B060-BL1	SE	1,2,3,7,8,9-HxCDF		0.00022	ug/kg	U	bl
PDI-SG-B060-BL1	SE	1,2,3,7,8-PeCDD		0.00019	ug/kg	U	bl
PDI-SG-B060-BL1	SE	1,2,3,7,8-PeCDF	0.00056	0.000061	ug/kg	J+	bl

Sample ID	Matrix	Compound	Result	EDL	Units	Validation Qualifiers	Validation Reason
PDI-SG-B060-BL1	SE	2,3,4,6,7,8-HxCDF		0.00010	ug/kg	U	bl
PDI-SG-B060-BL1	SE	2,3,4,7,8-PeCDF		0.00035	ug/kg	U	bl
PDI-SG-B060-BL1	SE	OCDD	0.19	0.00019	ug/kg	J	fd
PDI-SG-B060-BL1-D	SE	1,2,3,4,6,7,8-HpCDD	0.031	0.00035	ug/kg	J	fd
PDI-SG-B060-BL1-D	SE	1,2,3,4,7,8,9-HpCDF	0.00080	0.00020	ug/kg	J+	bl
PDI-SG-B060-BL1-D	SE	1,2,3,4,7,8-HxCDD		0.00037	ug/kg	U	bl
PDI-SG-B060-BL1-D	SE	1,2,3,6,7,8-HxCDD	0.0012	0.000051	ug/kg	J+	bl
PDI-SG-B060-BL1-D	SE	1,2,3,6,7,8-HxCDF	0.00084	0.000081	ug/kg	J+	bl
PDI-SG-B060-BL1-D	SE	1,2,3,7,8,9-HxCDD	0.00060	0.000046	ug/kg	J+	bl
PDI-SG-B060-BL1-D	SE	1,2,3,7,8,9-HxCDF		0.00032	ug/kg	U	bl
PDI-SG-B060-BL1-D	SE	1,2,3,7,8-PeCDD		0.00023	ug/kg	U	bl
PDI-SG-B060-BL1-D	SE	1,2,3,7,8-PeCDF	0.0015	0.000080	ug/kg	J+	bl
PDI-SG-B060-BL1-D	SE	2,3,4,6,7,8-HxCDF		0.00020	ug/kg	U	bl
PDI-SG-B060-BL1-D	SE	2,3,4,7,8-PeCDF	0.00084	0.000085	ug/kg	J+	bl
PDI-SG-B060-BL1-D	SE	2,3,7,8-TCDD		0.00013	ug/kg	U	bl
PDI-SG-B060-BL1-D	SE	OCDD	0.33	0.00027	ug/kg	J	fd
PDI-SG-B061-BL1	SE	1,2,3,4,6,7,8-HpCDF	0.0064	0.00025	ug/kg	JN	k
PDI-SG-B061-BL1	SE	1,2,3,4,7,8,9-HpCDF	0.00045	0.00027	ug/kg	J+	bl
PDI-SG-B061-BL1	SE	1,2,3,4,7,8-HxCDD		0.00048	ug/kg	U	bl
PDI-SG-B061-BL1	SE	1,2,3,4,7,8-HxCDF	0.00087	0.00011	ug/kg	J+	bl
PDI-SG-B061-BL1	SE	1,2,3,6,7,8-HxCDF	0.00042	0.00011	ug/kg	J+	bl
PDI-SG-B061-BL1	SE	1,2,3,7,8,9-HxCDD	0.00086	0.000079	ug/kg	J+	bl
PDI-SG-B061-BL1	SE	1,2,3,7,8,9-HxCDF		0.00032	ug/kg	U	bl
PDI-SG-B061-BL1	SE	1,2,3,7,8-PeCDD		0.00023	ug/kg	U	bl
PDI-SG-B061-BL1	SE	1,2,3,7,8-PeCDF	0.00057	0.000072	ug/kg	J+	bl
PDI-SG-B061-BL1	SE	2,3,4,6,7,8-HxCDF		0.00019	ug/kg	U	bl
PDI-SG-B061-BL1	SE	2,3,4,7,8-PeCDF		0.00035	ug/kg	U	bl
PDI-SG-B061-BL1	SE	2,3,7,8-TCDF	0.00090	0.000071	ug/kg	J+	bl

Attachment A

Nonconformance Summary Tables

Table A-1 - Continuing Calibration (CCV) RRTs

CCV ID (instrument ID)	Compound	Relative Retention Time (quant ion/conf ion)[1]	QC Limits	Associated Samples
320-218507/2 (3D5)	13C-1,2,3,4,7,8-HxCDF	0.942/0.941	0.944-0.970	PDI-SG-B056-BL1, PDI-SG-B058-BL1, PDI-SG-B060-BL1, PDI-SG-B060-BL1-D, PDI-SG-B061-BL1
	13C-1,2,3,4,6,7,8-HpCDD	1.079	1.086-1.110	
320-218319/2 (4D5)	13C-1,2,3,4,6,7,8-HpCDD	1.071	1.086-1.110	PDI-SG-B049-BL1, PDI-SG-B048-BL1, PDI-SG-B047-BL1, PDI-SG-B051-BL1, PDI-SG-B050-BL1, PDI-SG-B055-BL1, PDI-SG-B052-BL1, PDI-SG-B053-BL1, PDI-SG-B057-BL1
320-218548/2 (9D2)	37Cl4-2,3,7,8-TCDD	0.984	0.989-1.052	PDI-SG-B049-BL1, PDI-SG-B047-BL1, PDI-SG-B051-BL1, PDI-SG-B051-BL1, PDI-SG-B057-BL1, PDI-SG-B060-BL1, PDI-SG-B060-BL1-D, PDI-SG-B061-BL1
[1]The RRT of the quantitation ion is the same as the confirmation ion unless otherwise noted.				

Table A-2 - Recovery Standards RT

CCV ID (instrument ID)	Recovery Standard	Absolute Retention Time	Retention Time in ICAL Standard[1] (criterion +/- 15 seconds)	Associated Samples
320-218507/2 (3D5)	13C-1,2,3,4-TCDD	17.448	17.702 (17.452-17.952)	PDI-SG-B056-BL1, PDI-SG-B058-BL1, PDI-SG-B060-BL1, PDI-SG-B060-BL1-D, PDI-SG-B061-BL1
320-218319/2 (4D5)	13C-1,2,3,4-TCDD	18.791	18.101 (17.851-18.351)	PDI-SG-B049-BL1, PDI-SG-B048-BL1, PDI-SG-B047-BL1, PDI-SG-B051-BL1, PDI-SG-B050-BL1, PDI-SG-B055-BL1, PDI-SG-B052-BL1,
	13C-1,2,3,7,8,9-HxCDD	32.710	32.301 (32.051-32.551)	

CCV ID (instrument ID)	Recovery Standard	Absolute Retention Time	Retention Time in ICAL Standard[1] (criterion +/- 15 seconds)	Associated Samples
				PDI-SG-B053-BL1, PDI-SG-B057-BL1
Professional judgment was used to use the mid-point ICAL standard to determine the QC limits				

Table A-3 - Lab Blanks

Blank ID	Compound	Result	QL	BAL	Units	Associated Samples
MB 320-217420/1-A	2,3,7,8-TCDD	0.000266	0.0010	0.00133	ug/kg	All samples in 580-76388-2
	2,3,7,8-TCDF	0.000313	0.0010	0.00156	ug/kg	
	1,2,3,7,8-PeCDD	0.000263	0.0050	0.00132	ug/kg	
	1,2,3,7,8-PeCDF	0.000538	0.0050	0.00269	ug/kg	
	2,3,4,7,8-PeCDF	0.000358	0.0050	0.00179	ug/kg	
	1,2,3,4,7,8-HxCDD	0.000616	0.0050	0.00308	ug/kg	
	1,2,3,6,7,8-HxCDD	0.000336	0.0050	0.00168	ug/kg	
	1,2,3,7,8,9-HxCDD	0.000423	0.0050	0.00212	ug/kg	
	1,2,3,4,7,8-HxCDF	0.000414	0.0050	0.00207	ug/kg	
	1,2,3,6,7,8-HxCDF	0.000403	0.0050	0.00202	ug/kg	
	1,2,3,7,8,9-HxCDF	0.000610	0.0050	0.00305	ug/kg	
	2,3,4,6,7,8-HxCDF	0.000232	0.0050	0.00116	ug/kg	
	1,2,3,4,6,7,8-HpCDD	0.000470	0.0050	0.00235	ug/kg	
	1,2,3,4,6,7,8-HpCDF	0.000362	0.0050	0.00181	ug/kg	
	1,2,3,4,7,8,9-HpCDF	0.000430	0.0050	0.00215	ug/kg	
	OCDD	0.00192	0.010	0.00960	ug/kg	
	OCDF	0.000661	0.010	0.00330	ug/kg	

Table A-4 - Field Duplicates

Sample ID	Duplicate ID	Compound	Sample Result	Qual	Duplicate Result	Qual	QL	Units	RPD
PDI-SG-B060-BL1	PDI-SG-B060-BL1-D	OCDD	0.19		0.33		0.0077/0.008	ug/kg	53.8
PDI-SG-B060-BL1	PDI-SG-B060-BL1-D	1,2,3,4,6,7,8-HpCDD	0.018		0.031		0.0038/0.004	ug/kg	53.1

Table A-5 – Sample RRTs

Sample ID	Compound	Relative Retention Time (quant ion/conf ion)[1]	QC Limits
PDI-SG-B049-BL1	37Cl4-2,3,7,8-TCDD	0.984	0.989-1.052
	13C-1,2,3,4,6,7,8-HpCDD	1.071	1.086-1.110

Sample ID	Compound	Relative Retention Time (quant ion/conf ion)[1]	QC Limits
PDI-SG-B048-BL1	13C-1,2,3,4,6,7,8-HpCDD	1.071	1.086-1.110
PDI-SG-B047-BL1	13C-1,2,3,4,6,7,8-HpCDD	1.071	1.086-1.110
	37Cl4-2,3,7,8-TCDD	0.985	0.989-1.052
PDI-SG-B051-BL1	13C-1,2,3,4,6,7,8-HpCDD	1.071	1.086-1.110
	37Cl4-2,3,7,8-TCDD	0.984	0.989-1.052
PDI-SG-B050-BL1	13C-1,2,3,4,6,7,8-HpCDD	1.071	1.086-1.110
PDI-SG-B055-BL1	13C-1,2,3,4,6,7,8-HpCDD	1.071	1.086-1.110
PDI-SG-B052-BL1	13C-1,2,3,4,6,7,8-HpCDD	1.071	1.086-1.110
	37Cl4-2,3,7,8-TCDD	0.985	0.989-1.052
PDI-SG-B053-BL1	13C-1,2,3,4,6,7,8-HpCDD	1.071	1.086-1.110
PDI-SG-B053-BL1	13C-1,2,3,4,6,7,8-HpCDD	1.071	1.086-1.110
	37Cl4-2,3,7,8-TCDD	0.985	0.989-1.052
PDI-SG-B056-BL1	13C-1,2,3,4,7,8-HxCDF	0.942	0.944-0.970
	13C-1,2,3,6,7,8-HxCDF	0.949	0.949-0.975
	13C-1,2,3,4,6,7,8-HpCDD	1.078	1.086-1.110
PDI-SG-B058-BL1	13C-1,2,3,4,7,8-HxCDF	0.942	0.944-0.970
	13C-1,2,3,6,7,8-HxCDF	0.949	0.949-0.975
	13C-1,2,3,4,6,7,8-HpCDD	1.078	1.086-1.110
PDI-SG-B060-BL1	13C-1,2,3,4,7,8-HxCDF	0.941	0.944-0.970
	13C-1,2,3,6,7,8-HxCDF	0.949	0.959-1.021
	13C-1,2,3,4,6,7,8-HpCDD	1.079	1.086-1.110
	37Cl4-2,3,7,8-TCDD	0.985	0.989-1.052
PDI-SG-B060-BL1-D	13C-1,2,3,4,7,8-HxCDF	0.941/0.942	0.944-0.970
	13C-1,2,3,6,7,8-HxCDF	0.949	0.959-1.021
	13C-1,2,3,4,6,7,8-HpCDD	1.078	1.086-1.110
	37Cl4-2,3,7,8-TCDD	0.985	0.989-1.052
	2,3,7,8-TCDD	OK/1.003	0.999-1.002
PDI-SG-B061-BL1	13C-1,2,3,4,7,8-HxCDF	0.941/0.942	0.944-0.970
	13C-1,2,3,6,7,8-HxCDF	0.949	0.959-1.021
	13C-1,2,3,4,6,7,8-HpCDD	1.078	1.086-1.110
	37Cl4-2,3,7,8-TCDD	0.985	0.989-1.052

[1]The RRT of the quantitation ion is the same as the confirmation ion unless otherwise noted.
2,3,7,8-TCDD in sample PDI-SG-B060-BL1-D was negated due to blank contamination, no further action was required.

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