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Waste & Environmental Services

Standard Operating Procedure

for

ROUTINE VALIDATION OF DIOXIN FURAN ANALYTICAL DATA (EPA METHOD 1618 AND SW-846 EPA METHOD 8290)

APPROVAL SIGNATURES:

Subject Matter Expert:	**Organization**	**Signature**	**Date**
Bill Hardesty	WES-EDA	Signature on file	4/21/08
Quality Assurance Specialist:	**Organization**	**Signature**	**Date**
Laura Ortega	QA-IQ	Signature on file	4/30/08
Responsible Line Manager:	**Organization**	**Signature**	**Date**
Craig Eberhart	WES-EDA	Signature on file	4/21/08

1.0 PURPOSE AND SCOPE

This procedure represents the minimum standards for evaluating routine dioxin furan analytical data. This procedure is a mandatory document and shall be implemented by all Los Alamos National Laboratory (LANL or Laboratory) personnel and contractors who evaluate routine dioxin furan analytical data for the specific LANL projects.

2.0 BACKGROUND AND PRECAUTIONS

2.1 Background

This procedure conforms to the requirements of Environmental Protection Agency (EPA) Method 1613B and SW-846 EPA Method 8290). LANL data validation is performed according to procedures based upon the NNSA Model Data Validation Procedure. Data qualifiers and reason codes are assigned according to the specifications in this method specific procedure.

2.2 Precautions

Nothing in this procedure precludes the data validator from going beyond the minimum requirements specified within this procedure. If additional directions are required, the data validator shall reference NNSA Model Data Validation Procedure, EPA method specific guidelines and/or National Functional Guidelines for Organic Data Review. Implementation of this procedure may be followed by a more focused and data use-specific evaluation of the data by the project chemist, especially if the implementation of this procedure indicates the data may contain technical deficiencies.

3.0 EQUIPMENT AND TOOLS

None.

4.0 STEP-BY-STEP PROCESS DESCRIPTION

4.1 Qualifications for Data Validators

Data Validator	1.	Possess a minimum of a bachelor's degree in chemistry, or one of the physical sciences AND either two (2) years of experience in generating analytical data in an environmental analytical laboratory AND two (2) years of data validation experience.
	2.	Complete Attachment 1, Data Validation Cover Sheet, and Attachment 2, Dioxin/Furan Analytical Data Validation Checklist, during data validation.
	3.	Refer to the following attachments for additional guidance: • Attachment 3, Guidance for the Qualifier and Reason Code Application; and • Attachment 4, Theoretical Ion Abundance Ratios and Acceptance Limits for PCDDs and PCDFs for Method 8290.

4.2 Records

Data Validator	1.	Submit the following records generated by this procedure to the Records Processing Facility: • Completed Data Validation Cover Sheets; and • Completed Dioxin/Furan Analytical Data Validation Checklists.

5.0 PROCESS FLOW CHART

For specific validation criteria follow the NNSA Model for Data Validation.

6.0 ATTACHMENTS

Attachment 1	5169-1 Data Validation Cover Sheet (1 page)
Attachment 2	5169-2 Dioxin/Furan Analytical Data Validation Checklist (3 pages)
Attachment 3	5169-3 Guidance for the Qualifier and Reason Code Application (11 pages)
Attachment 4	5169-4 Theoretical Ion Abundance Ratios and Acceptance Limits for PCDDs and PCDFs for Method 8290 (1 page)

7.0 REVISION HISTORY

Author: Bill Hardesty

Revision No. *[Enter current revision number, beginning with Rev.0]*	Effective Date *[DCC inserts effective date for revision]*	Description of Changes *[List specific changes made since the previous revision]*	Type of Change *[Technical (T) or Editorial (E)]*
0	6/3/08	New Document	T

ATTACHMENT 1: EXAMPLE OF A DATA VALIDATION COVER SHEET

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Example of a Data Validation Cover Sheet

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Los Alamos NATIONAL LABORATORY EST.1943

Section I.

REQUEST NUMBER:________________ VALIDATION DATE:____________ LAB CODE:________________

CONTRACT LABORATORY NAME:____

VALIDATOR:____________________ ORGANIZATION:______________________________

ANALYTICAL SUITE (CHECK ALL THAT APPLY):

☐ **TPH-GRO**
☐ **TPH-DRO**
☐ **GENERAL CHEMISTRY**
☐ **HIGH EXPLOSIVES**
☐ **METALS**
☐ **RADIOCHEMISTRY**
☐ **DIOXIN FURANS**
☐ **PCB CONGENERS**
☐ **LCMSMS HIGH EXPLOSIVES**
☐ **LCMSMS PERCHLORATES**
☐ **ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS**

☐ **OTHER (DESCRIBE):**

____

Section II. Completeness Check

YES	**NO**	**N/A**	(**CHECK ONE**)	**YES**	**NO**	**N/A**	(**CHECK ONE**)
☐	☐	☐	1. CHAIN-OF-CUSTODY FORM(S)	☐	☐	☐	6. RAW/BSS DATA
☐	☐	☐	2. CASE NARRATIVE	☐	☐	☐	7. QUALITY CONTROL FORMS
☐	☐	☐	3. SAMPLE RESULT FORMS	☐	☐	☐	8. QUANTITATION REPORTS
☐	☐	☐	4. SAMPLE CHROMATOGRAMS	☐	☐	☐	9. TICS FORMS
☐	☐	☐	5. STANDARD CHROMATOGRAMS	☐	☐	☐	10. TICS MASS SPECTRA

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed-upon date of resolution and contract laboratory point of contact):

VALIDATOR'S SIGNATURE:____________________________________ DATE: ________________

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LOS ALAMOS
Environmental Restoration Project

ATTACHMENT 2: DIOXIN/FURAN ANALYTICAL DATA VALIDATION CHECKLIST

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Dioxin/Furan Analytical Data Validation Checklist

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	1. The IS retention time and qualitative criteria for target compound identification were not met.	R, DF0	R, DF0
☐	☐	☐	2. Required retention time documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, DF0b	R, DF0b
☐	☐	☐	3. Required IS information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, DF1d	R, DF1d
☐	☐	☐	4. The sample result is ≤5 times the concentration of the related analyte in the method blank.	N/A	U, DF4
☐	☐	☐	5. The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was >5x.	N/A	J, DF4a
☐	☐	☐	6. The sample result is ≤5 times the concentration of the related analyte in the trip blank, rinsate blank, or equipment blank.	N/A	U, DF4d
☐	☐	☐	7. Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, DF4e	R, DF4e
☐	☐	☐	8. The affected results were not analyzed with a valid 5-point calibration curve and/or a standard at the reporting limit.	UJ, R, DF7	J, DF7
☐	☐	☐	9. The affected analytes were analyzed with an initial calibration curve that exceeded the %RSD criteria.	UJ, R, DF7a	J, DF7a
☐	☐	☐	10. The affected analytes were analyzed with an out of range ion abundance in the initial calibration and/or CCV.	R, DF7b	R, DF7b
☐	☐	☐	11. The ICV and/or CCV were recovered outside the method specific limits.	UJ, R, DF7c	J, R, DF7c

Yes	**No**	**N/A**		**Assign Qualifier Listed Below If Criterion = Yes**	
(Check One)				**Non-detected Analyte**	**Detected Analyte**
☐	☐	☐	**12. The ICV and/or CCV were not analyzed at the appropriate method frequency.**	**UJ, DF7d**	**J, DF7d**
☐	☐	☐	**13. Required calibration information is missing or samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information.**	**R, DF7f**	**R, DF7f**
☐	☐	☐	**14. The affected analyte is considered rejected because the ion abundances did not meet specifications.**	**N/A**	**R, DF8**
☐	☐	☐	**15. The ion abundance documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.**	**R, DF8a**	**R, DF8a**
☐	☐	☐	**16. If GC column performance was not evaluated at the required frequency or if method criteria were not met, qualify all associated detects as J and all associated non-detects as UJ.**	**UJ, DF8b**	**J, DF8b**
☐	☐	☐	**17. If 2, 3, 7, 8-TCDF was detected in a sample and the result was not confirmed on a second column with successful analysis of the GC column performance mix, qualify all associated detects as U.**	**N/A**	**U, DF8c**
☐	☐	☐	**18. The holding time was >1 and ≤2 times the applicable holding time requirement.**	**UJ, DF9**	**J-, DF9**
☐	☐	☐	**19. The holding time was >2 times the applicable holding time requirement.**	**R, DF9a**	**J-, DF9a**
☐	☐	☐	**20. The LCS percent recovery was <10%.**	**R, DF12**	**J-, DF12**
☐	☐	☐	**21. The LCS percent recovery was < the Lower Acceptance Limit but >10%. Follow the external laboratory limits.**	**UJ, DF12a**	**J-, DF12a**
☐	☐	☐	**22. The LCS percent recovery was > the Upper Acceptance Limit. Follow the external laboratory limits.**	**N/A**	**J+, DF12b**
☐	☐	☐	**23. The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.**	**R, DF12c**	**R, DF12c**
☐	☐	☐	**24. The MS/MSD percent recovery was <10%.**	**R, DF12d**	**R, DF12d**

Yes (Check One)	No	N/A		Assign Qualifier Listed Below If Criterion = Yes Non-detected Analyte	Detected Analyte
☐	☐	☐	25. The MS/MSD percent recovery was >10% but <70%.	UJ, DF12e	J, DF12e
☐	☐	☐	26. The MS/MSD percent recovery was >130%	N/A	J+, DF12f
☐	☐	☐	27. The MS/MSD relative percent difference was >30%.	UJ, DF12g	J, DF12g
☐	☐	☐	28. The fortification sample percent recovery was <10%.	R, DF12h	J-, DF12h
☐	☐	☐	29. The fortification sample percent recovery was <40% but >10%	UJ, DF12i	J-, DF12i
☐	☐	☐	30. The fortification sample percent recovery was >135%.	N/A	J+, DF12j
☐	☐	☐	31. The fortification sample documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, DF12k	R, DF12k
☐	☐	☐	32. The affected analytes have elevated detection limits and may not meet project DQOs because the sample was diluted without any target analytes identified due to matrix interference.	R, DF15	J, DF15
☐	☐	☐	33. Sample clean-up was not performed. If run log notations, spectral data and/or internal standard or labeled compound recoveries indicate interferences and extract cleanup was not performed, qualify all associated detects as J and all non-detects as UJ.	UJ, DF15a	J, DF15a
☐	☐	☐	34. The instrument performance sample did not pass method acceptance criteria.	R, DF16	R, DF16
☐	☐	☐	35. The required instrument performance sample information is missing. Contact the SMO or external laboratory for information.	R, DF16c	R, DF16c
☐	☐	☐	36. The LANL project chemist identified quality deficiencies in the reported data that requires further qualification. This code can only be used and/or under advisement by the LANL project chemist.	UJ, R, DF19	J, R, DF19
☐	☐	☐	37. Duplicate, dilution, or reanalysis.	UJ, DF88	J, DF88

ATTACHMENT 3: GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION

5169-3

Records Use only

Guidelines for the Qualifier and Reason Code Application

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
1	R	R	DF0	The IS retention time and qualitative criteria for target compound identification were not met. For 2,3,7,8-substituted compounds that have an isotopically-labeled internal standard or recovery standard present in the sample extract, the Retention Time (RT) must be -1 to +3 seconds of the isotopically-labeled standard. For 2,3,7,8-substituted compounds that do not have an isotopically-labeled internal standard or recovery standard present in the sample extract, the RT must fall within 0.005 RRT units of the Required Retention Time (RRT) measured in the continuing calibration. For non-2,3,7,8-substituted compounds, the RT must be within the corresponding homologous RT windows established by analyzing the column performance check solution. If the RT of any compound is outside of the RT window, qualify all associated results as R.
2	R	R	DF0b	RRT documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
3	R	R	DF1d	Required IS information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
4	**N/A**	**U**	**DF4**	**The sample result is ≤5 times the concentration of the related analyte in the method blank.** **The criteria for the frequency of extraction and analysis of method blanks as stated in Section 9.5 of Method 1613B shall be followed and demonstrated in the documented data. The maximum amount of PCDD and PCDF isomer contamination in method blanks is stated in Table 2 of Method 1613B. The method blank must be measured on each GC/MS system which is used to measure a group of samples. This requirement includes measuring method blanks on a second GC column if confirmatory analysis of sample extracts on a second column is required by the method or by the laboratory statement of work.** **Any PCDD or PCDF measurement in a sample that is also measured in any associated blank, is qualified with a U flag if the sample concentration is <5 times the blank concentration.**
5	**N/A**	**J**	**DF4a**	**The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was >5x.** **The criteria for the frequency of extraction and analysis of method blanks as stated in Section 9.5 of Method 1613B shall be followed and demonstrated in the documented data. The maximum amount of PCDD and PCDF isomer contamination in method blanks is stated in Table 2 of Method 1613B. The method blank must be measured on each GC/MS system which is used to measure a group of samples. This requirement includes measuring method blanks on a second GC column if confirmatory analysis of sample extracts on a second column is required by the method or by the laboratory statement of work.** **If the maximum contamination requirements of specific TCDD and TCDF isomers stated in Table 2 of Method 1613B are not met, then all isomers in all samples associated with a method blank shall be qualified with a J flag.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
6	**N/A**	**U**	**DF4d**	**The sample result is ≤5 times the concentration of the related analyte in the trip blank, rinsate blank, or equipment blank.** **Any PCDD or PCDF measurement in a sample that is also measured in any associated blank is qualified with a U flag if the sample concentration is less than 5 times the blank concentration.**
7	**R**	**R**	**DF4e**	**Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.** **If the frequency of measuring method blanks is not met by the laboratory in the data submitted, then the results of all samples which do not meet the frequency of extraction and measurement of method blanks shall be qualified with an R flag.**
8	**UJ, R**	**J**	**DF7**	**The affected results were not analyzed with a valid 5-point calibration curve and/or a standard at the reporting limit.** **There shall be an initial calibration curve consisting of five points for each analyte. The initial calibration curve shall be determined < 30 days from the time the first samples of a Sample Delivery Group (SDG) are measured by the laboratory. The laboratory shall use the same calibration standards with the same lot number, for all internal standards, and labeled standards used in measuring the initial calibration curve, verification standards, field samples, and method blanks on both the primary GC column and on the secondary confirmation GC column.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
9	**UJ, R**	**J**	**DF7a**	**The affected analytes were analyzed with an initial calibration curve that exceeded the %RSD criteria.** **A 5-point calibration is prepared for each labeled and unlabeled compound. The RRF %RSD for the unlabeled standards must be ≤30%. Ion abundance ratios must meet the criteria listed in Attachment 4.** **If the %RSD is >20% for any unlabeled calibration standard, or >30% for any labeled calibration standard, but ≤40%, qualify all associated detects as J and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as UJ.** **If the %RSD is >40% but ≤60% for either a labeled or unlabeled calibration standard, qualify all associated detects as J and all associated non-detects as UJ.** **If the %RSD is >60% for either a labeled or unlabeled calibration standard, qualify all associated detects as J and all associated non-detects as R.** **If the ion abundance criteria were not met for any calibration compound, qualify all associated detects as J and all associated non-detects as UJ.** **If the affected results were not analyzed with a valid 5-point calibration curve and/or a standard at the reporting limit qualify the results as not detected.** **Ion abundance must meet the criteria in Attachment 4.**
10	**R**	**R**	**DF7b**	**The affected analytes were analyzed with an out-of-range ion abundance in the initial calibration and/or CCV.** **Ion abundance must meet the criteria in Attachment 4.** **If the ion abundance criteria are not met, qualify results for that analyte R.**
11	**UJ, R**	**J, R**	**DF7c**	**The ICV and/or CCV were recovered outside the method specific limits.** **See DF7a for ICAL specifications.** **CCV calibration must be verified for both labeled and unlabeled compounds at the beginning and end of each 12-hour period during which analysis is performed. The measured RFs must be ≤20% of the mean values established during initial calibration for unlabeled compounds and ≤30% of the mean values established during initial**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
				calibration for labeled compounds. The ion abundance must be within the limits in Attachment 4. **For the calibration verification analyzed at the beginning of a 12-hour period, the effect on data quality of a standard that does not meet criteria must be assessed using professional judgment. Guidance is provided in Section 7.7.4.4 of the EPA method 8290.** **For the calibration verification analyzed at the end of a 12-hour period, a %D of 25% for unlabeled compounds and 35% for labeled compounds is acceptable; however, in this instance, the mean RFs obtained from the beginning and ending daily calibration runs are used to calculate analyte concentrations instead of the RFs obtained from the initial calibration.** **If the %D of the ending calibration is >25% for any unlabeled compound and/or >35% for any labeled compound, then successful performance of another initial calibration must be analyzed within two hours of sample analysis for the data to be acceptable. In this case, the mean RFs from the beginning and ending daily calibration runs are still used to calculate analyte concentrations.** **1. If the ion abundance ratio for any compound is outside of the method limits, qualify all associated detects as J and all associated non-detects as UJ.** **2. If the %D criteria were not met for any CCV compound at the beginning of a 12-hour period and the %D is positive, qualify all associated detects as J+.** **3. If the %D criteria were not met for any CCV compound at the beginning of a 12-hour period and the %D is negative, qualify all associated detects as J- and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as UJ.** **4. If the %D criteria were not met for any compound at the end of a 12-hour period, a new initial calibration was analyzed within two hours of sample analysis, and the %D is positive, qualify all associated detects as J+.** **5. If the %D criteria were not met for any compound at the end of a 12-hour period, a new initial calibration was analyzed within two hours of sample analysis, and the %D is negative, qualify all associated detects as J- and, if any other calibration**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
				criteria have been exceeded for that compound, qualify all associated non-detects as UJ. **6. If the %D criteria were not met for any compound at the end of a 12-hour period and a new initial calibration was not analyzed within two hours of sample analysis, qualify all sample data analyzed during that 12-hour period as R.**
12	**UJ**	**J**	**DF7d**	**The ICV and/or CCV were not analyzed at the appropriate method frequency.** **It should be noted that CLP protocol DFLM01.1 requires that the GC/MS system must be calibrated based upon a daily Calibration Check Standard, whereas, EPA Methods 1613B and 8290 require that the GC/MS system criteria of a daily calibration verification standard must be met with each 12-hour batch of samples measured, and that response factors for native target compounds are derived from the 5-point initial calibration.**
13	**R**	**R**	**DF7f**	**Required calibration information is missing or samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information.**
14	**N/A**	**R**	**DF8**	**The affected analyte is considered rejected because the ion abundances did not meet specifications.** **For identification of any compound, the ion abundance ratios must be within the limits specified in Attachment 4. If ion abundance ratio criteria were not met for any compound, qualify all associated results as R.** **If the RT of any compound is outside of the RT window, qualify all associated results as R.**
15	**R**	**R**	**DF8a**	**The ion abundance documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
16	**UJ**	**J**	**DF8b**	**The GC column performance solution is used for defining the homologous GC RT windows and to document the chromatographic resolution. Column performance must be evaluated at the beginning of each analytical period and must meet method acceptance criteria (see Section 8.2 of the 8290) before sample analysis may begin.** **If GC column performance was not evaluated at the required frequency or if method criteria were not met, qualify all associated detects as J and all associated non-detects as UJ.**
17	**N/A**	**U**	**DF8c**	**The DB-5 GC column generally used for PCDD and PCDF analyses does not adequately separate 2,3,7,8-TCDF from its closest eluting isomer. If 2,3,7,8-TCDF is detected in a sample, the result must be confirmed on a second column capable of separating 2,3,7,8-TCDF from all other TCDF homologues (as proven by successful analysis of the GC column performance column mix with <25% valley between 2,3,7,8-TCDF and its closest eluting isomer).** **If 2,3,7,8-TCDF was detected in a sample and the result was not confirmed on a second column with successful analysis of the GC column performance mix, qualify all associated detects as U.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
18	**UJ**	**J-**	**DF9**	**The extraction/analytical holding time are exceeded by <2 times the published method for holding times.** **Regulations require water samples be preserved by neutralizing any chlorine residual with 0.008% sodium thiosulfate, and cooling to 4ºC using a holding time of 7 days from day of collection to day of extraction of the sample. In addition, the maximum holding time of extracts is 40 days from day of extraction to day of injection of the extract.** **The holdinig time and preservation requirements of 2,3,7,8-TCDD and of other measured PCDD and PCDF isomers in non-water matrixes have not been promulgated by EPA.** **Therefore, the data validator should use the holding time specified in EPA Method 8290, which specifies that all samples, except fish and adipose tissue samples, must be stored at 4ºC in the dark, extracted within 30 days, and completely analyzed within 45 days of extraction. Fish and adipose samples must be stored at -20ºC in the dark, extracted within 30 days, and completely analyzed within 45 days of collection (see Section 6.4 of EPA Method 8290). EPA Method 1613B does not set holding times for PCDD or PCDF isomers. The EPA method does state that water samples which contain a chlorine residual should be treated with 80-mg of sodium thiosulfate per liter of water, samples should be maintained at 4ºC in the dark, and extracts should be analyzed within 40 days of extraction.**
19	**R**	**J-**	**DF9a**	**The extraction/analytical holding time was exceeded by >2 times the published method for holding times.**
20	**R**	**J-**	**DF12**	**The LCS percent recovery was <10%.**
21	**UJ**	**J-**	**DF12a**	**The LCS percent recovery was < the Lower Acceptance Limit but >10%. Follow the external laboratory limits.**
22	**N/A**	**J+**	**DF12b**	**The LCS percent recovery was > the Upper Acceptance Limit. Follow the external laboratory limits.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
23	**R**	**R**	**DF12c**	**The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.**
24	**R**	**R**	**DF12d**	**The MS/MSD percent recovery was <10%.**
25	**UJ**	**J**	**DF12e**	**The MS/MSD percent recovery was >10% but <70%.**
26	**N/A**	**J+**	**DF12f**	**The MS/MSD percent recovery was >130%.**
27	**UJ**	**J**	**DF12g**	**The MS/MSD relative percent difference was >30%.**
28	**R**	**J-**	**DF12h**	**The laboratory must spike all samples with the sample fortification solution and all sample extracts with recovery standard solution. The recovery acceptance criteria for each compound is 40% to 135%. The fortification sample percent recovery was <10%.**
29	**UJ**	**J-**	**DF12i**	**The laboratory must spike all samples with the sample fortification solution and all sample extracts with recovery standard solution. The recovery acceptance criteria for each compound is 40% to 135%. The fortification sample percent recovery was <40% but >10%.**
30.	**N/A**	**J+**	**DF12j**	**The laboratory must spike all samples with the sample fortification solution and all sample extracts with recovery standard solution. The recovery acceptance criteria for each compound is 40% to 135%. The fortification sample percent recovery was >135%.**
31.	**R**	**R**	**DF12k**	**The fortification sample documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.**
32.	**R**	**J**	**DF15**	**The affected analytes have elevated detection limits and may not meet project DQOs because the sample was diluted without any target analytes identified due to matrix interference. (Qualify non-detected results as rejected if the analytical laboratory cannot provide proof for matrix interference.)**
33.	**UJ**	**J**	**DF15a**	**Sample clean-up was not performed. If run log notations, spectral data and/or internal standard or labeled compound recoveries indicate interferences and extract clean-up was not performed, qualify all associated detects as J and all non-detects as UJ.**
34.	**R**	**R**	**DF16**	**The instrument performance sample did not pass method acceptance criteria.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
35	**R**	**R**	**DF16c**	**The required instrument performance sample information is missing. Contact the SMO or external laboratory for information.**
36	**UJ, R**	**J, R**	**DF19**	**The project chemist identified quality deficiencies in the reported data that require further qualification. This code can only be used under advisement by the project chemist.**
37	**UJ**	**J**	**DF88**	**Duplicate, dilution, or reanalysis.**

ATTACHMENT 4: THEORETICAL ION ABUNDANCE RATIOS AND ACCEPTANCE LIMITS FOR PCDDs AND PCDFs FOR EPA METHOD 8290

5169-4

Theoretical Ion Abundance Ratios and Acceptance Limits for PCDDs and PCDFs for EPA Method 8290

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Los Alamos
NATIONAL LABORATORY
EST.1943

Number of Chlorine Atoms	Ion Type	Theoretical Ratio	Acceptance Limits	
			Lower	Upper
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
6[1]	M/M+2	0.51	0.43	0.59
7[2]	M/M+2	0.44	0.37	0.51
7	M+2/M+4	1.04	0.88	1.20
8	M+2/M+4	0.89	0.76	1.02

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[1] Used only for ^{13}C-HXCDF (internal standard).

[2] Used only for ^{13}C-HPCDF (internal standard).