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APPENDIX 38

1.0 OBJECTIVES

This standard operating procedure (SOP) describes procedures that the Environmental Standards, Inc. data reviewer will use to validate polychlorinated dibenzodioxin (dioxin) and polychlorinated dibenzofurans (furan) organic data generated by US EPA Method 1613B for General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. Validation will be performed to assess the compliance of the sample data to US EPA Method 1613B and/or other reference documents (*e.g.*, analytical SOPs), as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. In addition, the usability of the dioxin/furan organic data provided by the analytical laboratory(ies) will be determined based on the general guidance provided in the "US EPA Contract Laboratory Program National Functional Guidelines for Chlorinated Dioxin/Furan Data Validation" (Draft 9/2000) (National Functional Guidelines). It should be noted that the National Functional Guidelines applies strictly to data generated by the Contract Laboratory Program (CLP) protocol. As such, it is not directly applicable to validation of data generated by US EPA Method 1613B; therefore, this SOP presents the specific data qualification actions that will be used for validation.

The validation findings will be presented in a quality assurance review (QAR) that will be prepared from one or more sample delivery groups (SDGs). Copies of annotated analytical results summaries (Form I's), including any changes to the analytical results and all data qualifier codes, or a data summary spreadsheet of the qualified analytical results will be included in the analytical results section of the QAR.

2.0 EVALUATION TOOLS

Excel forms available in R:/Templates/Chemistry/XCELforms:

Organic field duplicate comparison Rev 1-01.xls
Organic field quadruplicate comparison Rev 1-01.xls
Organic field triplicate comparison Rev 1-01.xls

Chemistry Apps

FIT
Methods database

3.0 REFERENCE DOCUMENTS

US EPA Method 1613B (10/94)

US EPA Contract Laboratory Program National Functional Guidelines for Chlorinated Dioxin/Furan Data Validation (Draft 9/2000)
Region III – SOP for Dioxin/Furan Data Validation (Draft 3/99)
Region IV – Data Validation SOP for Polychlorinated Dibenzodioxin and Polychlorinated Dibenzofurans Analysis by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (9/96)
Region II – Data Validation SOP for EPA Method 1613, Revision A (Revision 2 9/99)

4.0 PROCEDURE

4.1 EVALUATION OF METHOD COMPLIANCE

The data reviewer will assess the method compliance of the dioxin/furan data based on an evaluation of information presented in the data package deliverables. Compliance to US EPA Method 1613B and/or other reference documents (*e.g.*, analytical SOPs) as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program (as directed by the Project Manager) will be evaluated as part of the assessment. In addition, the deliverables will be evaluated for reporting errors and inconsistencies. The findings of the method compliance assessment will be described in terms of deficiencies and comments about the data/deliverables. The deficiencies/comments will be presented in three subdivisions (*i.e.*, correctable deficiencies, noncorrectable deficiencies, and comments) of the Organic Data Evaluation Section of the QAR. Each deficiency and comment discussed in the QAR will indicate any subsequent impact on the usability of the data or any certain aspect(s) of the data that could not be evaluated due to the deficiency.

The data reviewer should contact the project laboratories to request the correction of deficiencies prior to the submittal of the QAR (if feasible and sanctioned by General Electric Company) at a minimum corrections necessary for a full evaluation of the usability of the data should be requested. Such correctable deficiencies may include sample result errors, missing data deliverables, or calculation errors that would take a significant amount of the data reviewer's time to correct. Any laboratory resubmittals as

a result of such requests will be discussed in the comments subdivision of the QAR and included as an attachment to the QAR.

4.2 DETERMINATION OF DATA USABILITY

The data reviewer will determine the usability of the dioxin/furan organic data based on an evaluation of the information presented in the data package deliverables. The findings of the dioxin/furan organic data usability assessment will be described in terms of certain qualifications of the data that the project team should consider in order to best utilize the data. These qualifications will be presented in the Organic Data Qualifier subsection of the QAR. Each qualification discussed in the QAR will indicate that the affected sample result(s) has been flagged with representative qualifier code(s) in the General Electric Company's database to provide, at a glance, an indication of the quantitative and qualitative reliability of each analytical result. In general, the qualifier statements will be presented in the QAR in the following order: blank qualification, common contaminants that were not qualified, unusable results ("R/UR"), estimated results ("J/UJ"), field duplicate comparison and a general qualifier for all results reported the quantitation limit (if applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program).

The data reviewer's criteria for evaluating the usability of the dioxin/furan organic data and the resulting qualifications will be as stated in the attached Table for the Validation of Polychlorinated Dibenzo-*p*-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B. It should be noted that the Project Manager should be consulted when directed to use "professional judgement" in the attached table.

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Temperature Upon Receipt	0-4°C for aqueous <-10°C for solid and tissue	Due to the stability of PCDDs and PCDFs, there is no direct impact on data usability due to receipt temperatures outside the specified range.
Holding Time (See Note #1 for additional information.)	All matrices should be extracted within 30 days of sample collection and analyzed within 45 days of extraction.	If holding time is exceeded, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If holding time is grossly exceeded (>twice the holding time), qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
Chromatographic Resolution (Isomer Specificity Test Standard, See Note #2 for additional information)	Should be analyzed at the beginning of each 12-hour period of sample and standard analysis. The % valley between unlabeled 2,3,7,8-TCDD and all other unlabeled TCDD should be <25%. The RT of the first and last eluting isomers are used to establish the RT windows for each congener class of PCDD/PCDF compounds.	Use professional judgement if the Isomer Specificity Test Standard was not analyzed at the required frequency. If the % valley between unlabeled 2,3,7,8-TCDD and all other unlabeled TCDD is >25%, qualify positive results for 2,3,7,8-TCDD as estimated ("J").
Window Defining Mix (WDM)	Should be analyzed at the beginning of each 12-hour period of sample and standard analysis.	If frequency is not met, qualify positive results for total homologues as estimated ("J").
Instrument Performance-Mass Spectrometer Performance (PFK)	Should be analyzed at the beginning of each 12-hour period during which samples are to be analyzed and prior to the analysis of the initial and continuing calibration standards. A static resolving power of at least 10,000 (10% valley definition) should be demonstrated at appropriate masses before any analysis is performed and at the end of each 12-hour period.	Use professional judgement if the mass calibration was not performed at the required frequency or if the resolving power was less than 10,000.

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Initial Calibration (See Note #3 for additional information)	Should be established with a minimum of 5 different concentration levels. The %RSD should be ≤20% for the 17 unlabeled standards and ≤35% for the labeled reference compounds. The relative ion abundance ratios should be within the limits specified in Note #3. The retention times of all target compounds, internal standards, and recovery standard should be within the windows established. The two monitored ions for each homologue should be present and should maximize simultaneously within 3 seconds of the corresponding ^{13}C-labeled isomer ions. The signal-to-noise (S/N) ratio for the GC signals present in the selected ion current profiles (SICPs) should be ≥ 10.	If the %RSD >20% but ≤90% (for unlabeled), qualify positive result as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the %RSD >90% (for unlabeled), qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If the relative ion abundance ratios for the two quantitation ions of the target compounds, internal standard, and/or recovery standards were not with the stated range, qualify positive results as unusable ("R"). Qualify positive results associated with the out of criteria ion abundance ratio internal standards and/or recovery standards. If the retention time of any target compound, internal standard, and/or recovery standard is not within the established retention time windows (±10 seconds of retention times in the WDM), qualify all data as unusable ("R/UR"). If the two monitored ions for a native isomer are not present and/or did not maximize simultaneously within 3 seconds of the corresponding ^{13}C-labeled isomer ion, qualify positive results as "not-detected" ("U") (the reported concentration with be reported at the detection limit). If the S/N ratio was <10, qualify "not-detected" results as unusable ("UR").

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Continuing Calibration (See Notes #3 and #4 for additional information)	Should be analyzed at the beginning of each 12-hour shift. The relative ion abundance ratios should be within the limits specified in Note #3. The recoveries (%Rs) should be within the limits specified in Note #4. The retention times for all compounds should be within the windows established. The two monitored ions for each homologue should be present and should maximize simultaneously within 3 seconds of the corresponding ^{13}C-labeled isomer ions. The signal-to-noise (S/N) ratio for the GC signals present in the SICPs should be ≥ 10.	If the unlabeled target compound recovery < lower limit but ≥ 50% of the lower limit, qualify positive result as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the unlabeled target compound recovery is > the upper limit but ≤ 150% of the upper limit, qualify positive result as estimated ("J"). If the unlabeled target compound recovery is < 50% of the lower limit or > 150% of the upper limit, qualify positive and "not-detected" results as unusable ("R/UR"). If the relative ion abundance ratio for the two quantitation ions are not within the specified range, qualify positive results as unusable ("R"). If the retention time of any target compound is not within the specified retention time window, qualify positive results as unusable ("R"). If the two monitored ions for a native isomer are not present and/or did not maximize simultaneously within 3 seconds of the corresponding ^{13}C-labeled isomer ion, qualify positive results as "not-detected" ("U") (the reported concentration with be reported at the detection limit). If the S/N ratio was <10, qualify "not-detected" results as unusable ("UR").
Internal Standards and Recovery Standards	Added to all samples and standards. %Rs should be within the limits specified in Note #4. The relative ion abundance ratios should be within the limits specified in Note #3. The retention times should be within the windows established.	Use professional judgement to determine if qualification is necessary due to relative ion abundance ratio being outside the specified range and if the retention times are not within the windows established. If the %R is > upper limit, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If the %R is < lower limit but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the %R is <10%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Blanks (See Note #5 for additional information)	Summarize all results greater than the estimated detection limit (EDL). The highest positive results associated with a sample should be utilized for evaluation of contamination.	If a target compound is found in blank but not in the associated sample(s), no action is taken. If a sample result is ≤5× the blank result, qualify the results as "not-detected" ("U*"). The value of the positive result should be used as the revised EDL. If a sample result >5× (or 10× for OCDD only) blank result, no qualification is necessary. If gross contamination exists (*i.e.*, saturated peaks by GC/MS), qualify samples as unusable ("R") due to interference.

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	For accuracy, use laboratory acceptance limits. For precision, use RPD limit of 40% for all matrices.	Data should not be qualified due to %Rs (or RPDs calculated on %Rs) that are outside of criteria if original concentration of a compound is > 4× spiking level for that compound. RPDs calculated using MS/MSD results can still be used to evaluate precision. If the recovery is > upper limit, qualify positive results for that compound in the native sample as estimated ("J") and do not qualify "not-detected" results. If the recovery is < lower limit but ≥10%, qualify positive results for that compound in the native sample as estimated ("J") and qualify "not-detected" results for that compound in the native sample as estimated ("UJ"). If the recovery is <10%, qualify positive results in the native sample as estimated ("J") and qualify "not-detected" results for that compound in the native samples as unusable ("UR"). If the precision is >20%, qualify positive results for that compound in the native sample as estimated ("J") and do not qualify "not-detected" results. If the precision criteria (See field duplicate usability criteria) for non-spiked compounds are not met, qualify positive results in the native sample as estimated ("J") and qualify "not-detected" results in the native sample as estimated ("UJ"). If a field duplicate of the native (unspiked) sample was collected and analyzed, the field duplicate should also be qualified if the MS/MSD %Rs or RPD are outside of criteria as stated above for the native sample.
Ongoing Precision and Recovery (OPR) Standard	%Rs should be within the limits specified in Note #4. The relative ion abundance ratios should be within the limits specified in Note #3. The retention times should be within the windows established.	If the recovery for a target compound is outside of the acceptance criteria, qualify all positive results and "not-detected" results as unusable ("UR").

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Field Duplicate/Laboratory Duplicate (See Note #6 for additional information)	Use precision limits of 20% RPD (%RSD for triplicate and quadruplicate analyses) for aqueous samples and 40% RPD (%RSD for triplicate and quadruplicate analyses) for solid samples when sample results are ≥5× RL. Use limit of ± RL (±2× RL for solids) when at least one sample value is <5× RL. (Use one-half the RL as a numerical value for any "not-detected" results in the RPD calculations).	If the criteria are not met, qualify positive results in original sample, and its duplicate as estimated ("J") and qualify "not-detected" results as estimated ("UJ").
Percent Solids	Qualification is for solid samples with less than 50% solid content.	If a solid sample has a percent solid content <50% but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Use professional judgement if a solid sample has a percent solid content <10%.

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Target compound Identification (See Note #7 for additional information)	For 2,3,7,8-substitued isomers for which an isotopically labeled internal standard is present, the absolute RT at the maximum peak height should be within –1 to +3 seconds of the RT of the corresponding labeled standard. For non-2,3,7,8-substitued isomers, the RT should be within the established window. The two quantitation ions for the compounds, internal standards, and recovery standards should maximize simultaneously (within 2 seconds). The relative ion abundance ratios should be within the limits specified in Note #3. All integrated ion current for each characteristic ion of the target compound should have an S/N ratio ≥2.5. The identification of a peak as a PCDF can only be made if no signal having a S/N ≥2.5 is detected at the same time in the corresponding polychlorinated diphenyl ether (PCDPE) channel. Any results reported for 2,3,7,8-TCDF should be confirmed on a DB-225 column.	Use professional judgement to determine if the result should be changed to "not-detected" or flagged "EMPC" if one or more of the identification criteria specified was not met. Use professional judgement if a PCDPE peak was detected at the same retention time as a reported PCDF result.
Compound Quantitation and Detection Limits	The laboratory should reextract samples (utilizing a smaller sample aliquot) with compound concentrations above the instrument calibration range.	If a target compound result exceeds the instrument calibration range, qualify the positive result as estimated ("J"). If a target compound result is below the low calibration standard concentration, qualify the positive result as estimated ("J"). If the laboratory performed a dilution of a sample that had a target compound result that exceeded the instrument calibration range instead of reextracting a smaller sample aliquot, qualify positive results for the dilution analysis as estimated ("J").
System Performance (See Note #8 for additional information)	Professional judgement should be used when assessing the degradation of the system performance during analyses.	Professional judgement should be used to qualify the data if it is determined that the system performance has degraded during sample analysis.

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Overall Assessment of Data	Assess overall quality of the data. Review available materials to assess the quality, keeping in mind the additive nature of the analytical problems.	Use professional judgement to determine the need to qualify data that were not qualified based on the QC previously addressed. Write a brief narrative to give the user an indication of the analytical limitation of the data. If sufficient information on the intended use and required quality of the data is available, the reviewer should include the assessment of the usability of the data within the given context.

Notes for the Validation of PCDD and PCDF Data Generated by the US EPA Method 1613B

1. The holding time of extraction within 30 days of sample collection is a recommendation; however, since PCDDs and PCDFs are very stable in many matrices, the holding time may be as high as one year. Use professional judgement when evaluating samples that were extracted beyond the 30 day holding time.

2.

DB-5 Column GC Retention Time WDM

Congener	First Eluted	Last Eluted
TCDF	1,3,6,8-	1,2,8,9-
TCDD	1,3,6,8-	1,2,8,9-
PeCDF	1,3,4,6,8-	1,2,3,8,9-
PeCDD	1,2,4,7,9-	1,2,3,8,9-
HxCDF	1,2,3,4,6,8-	1,2,3,4,8,9-
HxCDD	1,2,4,6,7,9-	1,2,3,4,6,7-
HpCDF	1,2,3,4,6,7,8-	1,2,3,4,7,8,9-
HpCDD	1,2,3,4,6,7,9-	1,2,3,4,6,7,8-

DB-5 Column TCDD Isomer Specificity Test Standard

1,2,3,4-TCDD	1,2,3,7-TCDD
1,2,3,9-TCDD	2,3,7,8-TCDD

DB-225 Column TCDF Isomer Specificity Test Standard

2,3,4,7-TCDF
2,3,7,8-TCDF
1,2,3,9-TCDF

3. If the initial calibration %RSD is >50% but ≤90%, the linearity of the first three initial calibration standards for the compound should be evaluated. If the first three initial calibration standards for the compound are linear then do not qualify "not-detected"

results. If the first three initial calibration standards for the compound are not linear, then qualify "not-detected" results as estimated ("UJ").

Use professional judgement when evaluating the concentration intercept of a calibration curve. If the concentration intercept is positive then the samples should be evaluated for false positives. If the concentration intercept is negative then the samples should be evaluated for false negatives.

Relative Ion Abundance Criteria for PCDDs and PCDFs

PCDDs	Relative Intensity
Tetra	0.65-0.89
Penta	1.32-1.78
Hexa	1.05-1.43
Hepta	0.88-1.20
Octa	0.76-1.02
PCDFs	Relative Intensity
Tetra	0.65-0.89
Penta	1.32-1.78
Hexa	1.05-1.43
Hexa[1]	0.43-0.59
Hepta	0.88-1.20
Hepta[2]	0.37-0.51
Octa	0.76-1.02

1 - used only for ^{13}C-HxCDF (internal standard)
2 - used only for ^{13}C-HpCDF (internal standard)

4.

Acceptance Criteria for Performance Tests When All PCDD/PCDF Are Tested

PCDD/PCDF	Concentration (ng/ml)	OPR (ng/ml)	Verification (ng/ml)
2,3,7,8-TCDD	10	6.7-15.8	7.8-12.9
2,3,7,8-TCDF	10	7.5-15.8	8.4-12.0
1,2,3,7,8-PeCDD	50	35-71	39-65
1,2,3,7,8-PeCDF	50	40-67	41-60
2,3,4,7,8-PeCDF	50	34-80	41.61
1,2,3,4,7,8-HxCDD	50	35-82	39-64
1,2,3,6,7,8-HxCDD	50	38-67	39-64
1,2,3,7,8,9-HxCDD	50	32-80	41-61
1,2,3,4,7,8-HxCDF	50	36-67	45-56
1,2,3,6,7,8-HxCDF	50	42-65	44-57
1,2,3,7,8,9-HxCDF	50	39-65	45-56
2,3,4,6,7,8-HxCDF	50	35-78	44-57
1,2,3,4,6,7,8-HpCDD	50	35-70	43-58
1,2,3,4,6,7,8-HpCDF	50	41-61	45-55
1,2,3,4,7,8,9-HpCDF	50	39-69	43-58
OCDD	100	78-144	79-126
OCDF	100	63-170	63-159
$^{13}C_{12}$-2,3,7,8-TCDD	100	20-175	82-121
$^{13}C_{12}$-2,3,7,8-TCDF	100	22-152	71-140
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	21-227	62-160
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	21-192	76-130
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	13-328	77-130
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	21-193	85-117
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	25-163	85-118
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	19-202	76-131
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	21-159	70-143
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	17-205	74-135
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	22-176	73-137
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	26-166	72-138
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	21-158	78-129
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	20-186	77-129
$^{13}C_{12}$-OCDD	200	26-397	96-415

Acceptance Criteria for Performance Tests When All PCDD/PCDF Are Tested

PCDD/PCDF	Concentration (ng/ml)	OPR (ng/ml)	Verification (ng/ml)
$^{37}Cl_4$-2,3,7,8-TCDD	10	3.1-19.1	7.9-12.7

Acceptance Criteria for Performance Tests When Only Tetra Compounds Are Tested

PCDD/PCDF	Concentration (ng/ml)	OPR (ng/ml)	Verification (ng/ml)
2,3,7,8-TCDD	10	7.3-14.6	8.2-12.3
2,3,7,8-TCDF	10	8.0-14.7	8.6-11.6
$^{13}C_{12}$-2,3,7,8-TCDD	100	25-141	85-117
$^{13}C_{12}$-2,3,7,8-TCDF	100	26-126	76-131
$^{37}Cl_4$-2,3,7,8-TCDD	10	3.7-15.8	8.3-12.1

Acceptance Criteria for Labeled Compound Recovery in Samples When All PCDD/PCDFs Are Tested

PCDD/PCDF	Concentration (ng/ml)	Recovery (ng/ml)	Recovery (%)
$^{13}C_{12}$-2,3,7,8-TCDD	100	25-164	25-164
$^{13}C_{12}$-2,3,7,8-TCDF	100	24-169	24-169
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	25-181	25-181
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	24-185	24-185
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	21-178	21-178
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	32-141	32-141
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	28-130	28-130
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	26-152	26-152
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	26-123	26-123
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	29-147	29-147
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	28-136	28-136
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	23-140	23-140
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	28-143	28-143
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	26-138	26-138
$^{13}C_{12}$-OCDD	200	34-313	17-157
$^{37}Cl_4$-2,3,7,8-TCDD	10	3.5-19.7	35-197

Acceptance Criteria for Labeled Compound Recovery in Samples When Only Tetra Compounds Are Tested

PCDD/PCDF	Concentration (ng/ml)	Recovery (ng/ml)	Recovery (%)
$^{13}C_{12}$-2,3,7,8-TCDD	100	31-137	31.137
$^{13}C_{12}$-2,3,7,8-TCDF	100	29-140	29-140
$^{37}Cl_4$-2,3,7,8-TCDD	10	4.2-16.4	42-164

5. The frequency of equipment blanks is determined during the sampling event. The results of a equipment/rinse blank should be applied to all samples collected using the same equipment (equipment/rinse blanks only) on the same day (unless only one was collected for a several-day sampling event; results would be applied to all samples in the SDG). In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration for a contaminant.

6. Duplicate samples may be taken and analyzed as an indication of overall precision. Field duplicate analyses measure both field and laboratory precision; therefore, the results may have more variability than laboratory duplicates which measure only laboratory performance. It is also expected that soil duplicate results will have a greater variance than aqueous duplicate results.

7. US EPA Method 1613B (Section 16.6) requires that a result meet all identification criteria or the result should not be reported. The sample should undergo reextraction with additional cleanup to remove any interference. Therefore, the laboratory should not be reporting the estimated maximum possible contamination (EMPC) results. If the presence of a reported positive is questioned (mostly due to chlorinated ether interference or if ratio/retention times are out), quality the result as "EMPC".

8. Poor chromatographic performance affects both qualitative and quantitative results. Indications of substandard performance include:

   a. high background levels or shifts in absolute retention times of internal standards
   b. excessive baseline rise at elevated temperatures
   c. extraneous peaks
   d. loss of resolution
   e. peak tailing or peak splitting that may result in inaccurate quantitation