Superfund Records Center
SITE: Ciba-Geigy
BREAK: 19.00
OTHER: 651246

WESTON
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July 26, 1991
Y-0-7-12

REC'D
8-8-91
FB.

Mr. Dennis Gagne
Regional Sample Control Custodian
U.S. Environmental Protection Agency
90 Canal Street
Boston, Massachusetts 02114

Re: TID No. 01-9105-06
SAS No. 6149A, SDG No. 6149A-01
Triangle Laboratory
Ciba Geigy
Cranston, RI
Dioxin: 1/Aqueous/6149A-01
6/Soil/6149A-04, 6149A-05, 6149A-06, 6149A-07, 6149A-08, 6149A-09

Dear Mr. Gagne:

A validation was performed on the dioxin/furan analytical data for one aqueous sample and six soil samples collected by Alliance Technologies Corporation at the Ciba Geigy Site. Included with the field samples were two soil Performance Evaluation Mix (PEM) samples. The samples were analyzed according to SAS 6149A specifications. The data were evaluated based on the following parameters:

- data completeness
- * PEM samples
- * window defining mix
- calibrations
- * column performance
- internal standards
- * recovery standards
- method blanks
- * matrix spike/matrix spike duplicates
- field duplicates
- * concentration/EMPC/EDL
- total congener concentrations
- * toxic equivalency factors

* - All criteria were met for this parameter.

<u>Table I summarizes the validation recommendations which were based on the following information:</u>

SEMS DocID 651246

Data Completeness

The laboratory was contacted on July 11, 1991 and the following information pertaining to SAS 6149A was requested:

- summary of area counts for CC3 and IC standards
- raw data for window defining mix, File 2852
- recalculated TEF-adjusted values
- confirmation of total TCDF value for sample 6149A-09
- confirmation of concentration value for sample 6149A-08DL

Information was received from the laboratory on July 24, 1991.

PEM Samples

No 2,3,7,8-TCDD was reported in the blank PEM sample 6149A-02 (NKG722). In the spike PEM sample 6149A-03 (MJO345), 1.77 ug/Kg of 2,3,7,8-TCDD was reported, which is within the 95% prediction limits of 1.18 to 1.98.

OCDD contamination was observed in both the blank and spike PEM samples. No action is necessary since the PEM samples are not reported on the data summary tables. Qualifications of sample results based on the contamination are discussed in the Method Blank section.

Calibrations

The 3/8/91 initial calibration on the DB-5 column did not meet the %RSD criteria of 15.0% for 12378-PeCDD (16.7%). Positive results and EMPCs for 12378-PeCDD and total PeCDDs will be estimated (J) and non-detected results will be accepted for all samples. Therefore, the 12378-PeCDD EMPC value and the total PeCDD in sample 6149A-08 will be estimated (J) on the data summary table.

The 4/17/91 initial calibration on the SP-2331 column did not meet the %RSD criteria of 15.0% for OCDF (30%) and ^{13}C-OCDD (18%). No action is necessary on the data summary tables since the SP-2331 column was not used for quantitation.

Internal Standards

The recovery of ^{13}C-123678-HXCDD for sample 6149A-08DL on the confirmation column (SP-2331) exceeded the QC limit of 150% (150.4%). No action is necessary on the data summary tables since the SP-2331 column was not used for quantitation.

Due to interferences, the recovery of ^{13}C-OCDD for sample 6149A-06 exceeded the QC limit of 150% (484.2%). Additional clean-up was performed. Since all recoveries were within criteria for the reanalysis, results for sample 6149A-06RE will be reported on the data summary tables rather than the initial analysis results.

Method Blanks

Significant OCDD contamination was observed in DFBLK2. The blank PEM sample 6149A-02, analyzed on the same day as DFBLK2, also showed OCDD contamination. The following samples were analyzed on the same day as DFBLK2 and may be affected by contamination: 6149A-04, 6149A-05, 6149A-07, 6149A-08, and 6149A-09. The positive OCDD value for sample 6149A-08 will not be estimated (J) since a concentration of OCDD significantly greater than could be attributed to blank contamination was detected. The positive OCDD value for sample 6149A-05 will not be estimated since the OCDD concentration was confirmed by duplicate sample 6149A-06RE. The positive values and EMPCs for OCDD in samples 6149A-04, 6149A-07, and 6149A-09 will be estimated (J) on the data summary tables.

Significant total TCDD and total TCDF contamination was observed in all blanks, DFBLK1, DFBLK2, and DFBLK3. In all samples, positive values and EMPCs for total TCDD and total TCDF will be estimated (J) on the data summary tables.

Field Duplicates

The relative percent difference (RPD) for OCDF did not meet criteria in field duplicate samples 6149A-05 and 6149A-06RE. The positive OCDF value in sample 6149A-06RE will be estimated (J) and the non-detected OCDF value in 6149A-05 will be estimated (UJ) on the data summary table.

Total Congener Concentrations

The laboratory analyzed sample 6149A-08 on an SP-2331 column for confirmation since TEF-adjusted concentrations were greater than 0.7 ug/Kg. The laboratory reported in the case narrative that since the 12/90 Dioxin SOW does not provide

information on first and last eluters for each PCDD/PDCF group, SMO was contacted to determine what congeners were needed for the window defining mix. Since SMO informed the laboratory that the primary interest was for determination of specific 2378-congeners, the laboratory analyzed a five point calibration and used the CC3 calibration standard to set descriptor switching times for 2378-congeners. Therefore, total congener concentrations were not confirmed for sample 6149A-08.

Toxicity Equivalence Factor

The toxicity equivalence factors used to calculated the TEF-adjusted concentrations are from the "Interim Procedure for Estimating Risk Associated with Exposures to Mixtures of Chlorinated Dibenzo-p-dioxins and Dibenzofurans (CDDs and CDFs)", EPA625-3-89-016, March 1989.

Data Summary

The overall quality of the data package was good. However, due to significant blank contamination, the positive values and EMPCs for OCDD in samples 6149A-04, 6149A-07, and 6149A-09 will be estimated (J) and the total TCDD and total TCDF results for all samples will be estimated (J) on the data summary tables.

The positive value for OCDF will be estimated (J) in sample 6149A-06RE and the non-detected OCDF value in sample 6149A-05 will be estimated (UJ) due to poor field duplicate precision. The EMPC value for 12378-PeCDD and total PeCDD in sample 6149A-08 will be estimated (J) since the %RSD did not meet criteria.

Very truly yours,
ROY F. WESTON, INC.

Cheryl Blaine
Senior Organic Data Reviewer

John J. Hagopian, P. G.
Team Manager
ESAT Region I

/dag
Enclosure(s)

Ciba Geigy
SAS 6149A

PCDD/PCDF ANALYSES

SAMPLE NUMBER:	6149A-01	6149A-04	6149A-05	6149A-06	6149A-07	6149A-08	6149A-09
DIOXINS							
2,3,7,8-TCDD	A	A	A	A	A	A	A
1,2,3,7,8-PeCDD	A	A	A	A	A	A	J[3]
1,2,3,4,7,8-HxCDD	A	A	A	A	A	A	A
1,2,3,6,7,8-HxCDD	A	A	A	A	A	A	A
1,2,3,7,8,9-HxCDD	A	A	A	A	A	A	A
1,2,3,4,6,7,8-HpCDD	A	A	A	A	A	A	A
OCDD	A	J[1]	A	A	J[1]	A	J[1]
FURANS							
2,3,7,8-TCDF	A	A	A	A	A	A	A
1,2,3,7,8-PeCDF	A	A	A	A	A	A	A
2,3,4,7,8-PeCDF	A	A	A	A	A	A	A
1,2,3,4,7,8-HxCDF	A	A	A	A	A	A	A
1,2,3,6,7,8-HxCDF	A	A	A	A	A	A	A
2,3,4,6,7,8-HxCDF	A	A	A	A	A	A	A
1,2,3,7,8,9-HxCDF	A	A	A	A	A	A	A
1,2,3,4,6,7,8-HpCDF	A	A	A	A	A	A	A
1,2,3,4,7,8,9-HpCDF	A	A	A	A	A	A	A
OCDF	A	A	J[2]	J[2]	A	A	A

A - Accept the data.

J[1] - Estimate the positive value and EMPC (J) for OCDD due to blank contamination. Some or all levels of OCDD found in the sample may be attributed to contamination.

J[2] - Estimate the positive value (J) and estimate the non-detected value (UJ) for OCDD since the relative percent difference criteria was not met. Non-homogeneity of the sample may result in inaccurate quantitation.

J[3] - Estimate the EMPC value for 1,2,3,7,8-PeCDD since calibration criteria was not met. Instrument response to this compound was unstable and quantitation may not be accurate.

Data Summary Key

A	-	Acceptable data.
J	-	The associated numerical value is an estimated quantity.
R	-	Reject data due to quality control criteria. The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
U	-	The compound was analyzed for; but, was not detected. The associated numerical value is the sample quantitation limit.
UJ	-	The compound was analyzed for; but, was not detected. The sample quantitation limit is an estimated quantity.
--	-	The compound was analyzed for; but, was not detected. The sample quantitation limit is the same as the CRQL presented.

SITE: CIBA GEIGY - CRANSTON, RI

CASE/SAS NO: 6149A

Sample No	6149A-01		6149A-04		6149A-05	
Matrix	AQUEOUS		SOIL		SOIL	
TCDD/TCDF Conc	ng/L	DL/EMPC*	ug/kg	DL/EMPC*	ug/kg	DL/EMPC*
2,3,7,8-TCDD	U	0.100	U	0.0063	U	0.0317
1,2,3,7,8-PeCDD	U	0.382	U	0.0216	U	0.0536
1,2,3,4,7,8-HxCDD	U	0.250	U	0.0127	U	0.0634
1,2,3,6,7,8-HxCDD	U	0.216	U	0.0114	U	0.0536
1,2,3,7,8,9-HxCDD	U	0.226	U	0.0127	U	0.0585
1,2,3,4,6,7,8-HpCDD	U	0.412	U	0.0254	0.5682	
OCDD	U	0.375	0.0853 J		10.1707	
2,3,7,8-TCDF	U	0.070	U	0.0050	0.0414	
1,2,3,7,8-PeCDF	U	0.170	U	0.0127	U	0.0390
2,3,4,7,8-PeCDF	U	0.183	U	0.0127	U	0.0390
1,2,3,4,7,8-HxCDF	U	0.174	U	0.0089	U	0.0390
1,2,3,6,7,8-HxCDF	U	0.156	U	0.0076	U	0.0341
2,3,4,6,7,8-HxCDF	U	0.145	U	0.0076	U	0.0317
1,2,3,7,8,9-HxCDF	U	0.211	U	0.0114		0.0512*
1,2,3,4,6,7,8-HpCDF	U	0.184	U	0.0089	0.0878	
1,2,3,4,7,8,9-HpCDF	U	0.271	U	0.0127	U	0.0414
OCDF	U	0.203	U	0.0140	UJ	0.0292
TOTAL TCDD		17.891*J		2.0968*J		5.5024*J
TOTAL PeCDD	U	0.382	U	0.0216	U	0.0536
TOTAL HxCDD	U	0.250	U	0.0127	0.0487	
TOTAL HpCDD	U	0.412	U	0.0054	1.1463	
TOTAL TCDF		0.389*J		0.0458*J	0.5365 J	0.0024 J
TOTAL PeCDF	U	0.183	U	0.0127	0.0243	0.2560
TOTAL HxCDF	U	0.211	U	0.0114	0.0243	0.1219
TOTAL HpCDF	U	0.271	U	0.0127	0.1707	
TOXICITY EQUIVALENCY	0		0.000067		0.011	
DILUTION FACTOR	1		1		1	
% MOISTURE	N/A		21.5		59.0	
DATE OF RECEIPT	03/29/91		03/29/91		03/29/91	
SAMPLE EXTRACTION DATE	03/29/91		03/29/91		03/29/91	
ANALYSIS DATE	04/02/91		04/03/91		04/03/91	
GC/MS I.D.	VG70E		VG70E		VG70E	

Soil results in dry weight.

SITE: CIBA GEIGY - CRANSTON, RI

CASE/SAS NO: 6149A

Sample No	6149A-06RE		6149A-07		6149A-08	
Matrix	SOIL		SOIL		SOIL	
TCDD/TCDF Conc	ug/kg	DL/EMPC*	ug/kg	DL/EMPC*	ug/kg	DL/EMPC*
2,3,7,8-TCDD	U	3.91	U	0.0104	0.0360	
1,2,3,7,8-PeCDD	U	0.0939	U	0.0379		0.1422*J
1,2,3,4,7,8-HxCDD	U	0.0835	U	0.0314	U	0.0573
1,2,3,6,7,8-HxCDD	U	0.0757	U	0.0274	7.4267	
1,2,3,7,8,9-HxCDD	U	0.0783	U	0.0287	1.6709	
1,2,3,4,6,7,8-HpCDD	0.6762		U	0.0340	617.9#	
OCDD	11.8041			0.0471*J	5396.4#	
2,3,7,8-TCDF	0.0522		U	0.0091	0.2484	
1,2,3,7,8-PeCDF	U	0.0469	U	0.0170		0.0403*
2,3,4,7,8-PeCDF	U	0.0496	U	0.0183	0.0870	
1,2,3,4,7,8-HxCDF	U	0.0496	U	0.0196	0.7685	
1,2,3,6,7,8-HxCDF	U	0.0443	U	0.0183	0.2016	
2,3,4,6,7,8-HxCDF	U	0.0417	U	0.0170	0.1231	
1,2,3,7,8,9-HxCDF		0.0704*	U	0.0248		0.0445*
1,2,3,4,6,7,8-HpCDF	0.1227		U	0.0209	25.78	
1,2,3,4,7,8,9-HpCDF	U	0.1357	U	0.0314	1.947	
OCDF	0.5117J		U	0.0261	92.304	
TOTAL TCDD		6.9033*J		1.7225*J	0.0849 J	4.582*J
TOTAL PeCDD	U	0.0939	U	0.0379		88.917*J
TOTAL HxCDD	U	0.0835	U	0.0314	29.172	29.813
TOTAL HpCDD	1.1488		U	0.0340	1122.6	
TOTAL TCDF	0.6266 J	0.8224 J	U	0.0287*J	0.9341 J	1.312 J
TOTAL PeCDF		0.2036*	U	0.0183	2.187	2.435
TOTAL HxCDF	0.1305	0.2010	U	0.0248	28.386	28.531
TOTAL HpCDF	0.2349		U	0.0314	136.454	
TOXICITY EQUIVALENCY	0.010		0.000036		4.414	
DILUTION FACTOR	1		1		1	
% MOISTURE	61.7		23.6		52.9	
DATE OF RECEIPT	03/29/91		03/29/91		03/29/91	
SAMPLE EXTRACTION DATE	03/29/91		03/29/91		03/29/91	
ANALYSIS DATE	04/11/91		04/03/91		04/03/91	
GC/MS I.D.	VG70E		VG70E		VG70E	

- Results reported from diluted analysis

Soil results in dry weight

SITE: CIBA GEIGY - CRANSTON, RI

CASE/SAS NO: 6149A

Sample No	6149A-09					
Matrix	SOIL		SOIL		SOIL	
TCDD/TCDF Conc	ug/kg	DL/EMPC*	ug/kg	DL/EMPC*	ug/kg	DL/EMPC*
2,3,7,8-TCDD	0.014					
1,2,3,7,8-PeCDD	U	0.084				
1,2,3,4,7,8-HxCDD	U	0.056				
1,2,3,6,7,8-HxCDD	U	0.048				
1,2,3,7,8,9-HxCDD	U	0.052				
1,2,3,4,6,7,8-HpCDD	0.128					
OCDD	1.652 J					
2,3,7,8-TCDF	U	0.018				
1,2,3,7,8-PeCDF	U	0.032				
2,3,4,7,8-PeCDF	U	0.034				
1,2,3,4,7,8-HxCDF	U	0.066				
1,2,3,6,7,8-HxCDF	U	0.06				
2,3,4,6,7,8-HxCDF	U	0.054				
1,2,3,7,8,9-HxCDF	U	0.08				
1,2,3,4,6,7,8-HpCDF	0.032					
1,2,3,4,7,8,9-HpCDF	U	0.062				
OCDF	0.09					
TOTAL TCDD		3.926*J				
TOTAL PeCDD	U	0.084				
TOTAL HxCDD		0.054*				
TOTAL HpCDD	0.24					
TOTAL TCDF	0.08 J	0.172 J				
TOTAL PeCDF	0.12					
TOTAL HxCDF		2.262*				
TOTAL HpCDF	0.04	0.212				
TOXICITY EQUIVALENCY	0.002					
DILUTION FACTOR	1					
% MOISTURE	50.0					
DATE OF RECEIPT	03/29/91					
SAMPLE EXTRACTION DATE	03/29/91					
ANALYSIS DATE	04/03/91					
GC/MS I.D.	VG70E					

Soil results in dry weight

ATTACHMENT I

DIOXIN DATA REVIEW WORKSHEET

Regional Review of Dioxin Data Package

The hard copied (laboratory name Triangle Laboratories data package received at Region I has been reviewed and the quality assurance and performance data summarized. The data review included:

Case No. NA
Sampling Date 3.28.91
Matrix 1AQ, 8SOil (6samples + 2PEMs)
Date Received by Laboratory 3.29.91
Field Duplicates: 6149A-05, 6149A-06
Sample Nos.:

SAS No. 6149A
No. of Samples 9
Shipping Date 3.28.91

PEM Nos.	Laboratory ID	EPA Identification No.
Fortified Std	MJO 345	6149A-03
Fortified Blank	NGK 722	6149A-02

The general criteria used to determine the performance were based on an examination of:

- PEM Samples
- Initial and continuing calibrations
- Retention time marker solutions
- Estimated maximum possible concentration
- Internal Standard response
- Recovery standard areas
- Matrix spike analysis
- Duplicate analysis
- Method blanks
- Instrument sensitivity check
- Chromatographic resolution

Definition of Qualifiers

A - Acceptable data
J - Approximate data due to quality control criteria
R - Reject data due to quality control criteria
U - Not detected
S - $[M-COCl]^+$ ion did not meet S/N ratio >2.5 requirement
H - Did not meet the ion abundance criteria

DIOXIN DATA REVIEW WORKSHEET

I PEM SAMPLES

A. Fortified Blank

Compounds Found	Laboratory ID NGK722 Concentration	EPA ID 6149A-02 Expected Concentration	Laboratory No.
Ø	2378-TCDD	Ø	
OCDD	0.067	Ø	
total TCDD/TCDF	EMPCs	Ø	

B. Fortified Std.

Compounds Found	Laboratory ID MJ0345 Concentration	EPA ID 6149A-03 EPA 95% Prediction Level	Laboratory No.
2,3,7,8-TCDD	1.77ppb	1.4 .98-2.18	
total TCDD	3.37		
1234678 HpCDD	~~HP~~ EMPC		
total TCDF	EMPC		

Action: If the 2,3,7,8-TCDD reported in the fortified standard is beyond the 95 % prediction level or a false positive for 2,3,7,8-TCDD in the blank is reported, reject all data.

DB-5

II. INITIAL AND CONTINUING CALIBRATION

Date of Initial Calibration: 3/8/91

Date of Continuing Calibration: 4/2/91 ; 4/3/91 ; 4/11/91

Date	Instrument	% RSD, %D	Compound (Results)	Samples Affected
3/8/91	E	% RSD 17%	1,2,3,7,8-PeCDD	All
3/8/91	E	% RSD 15.1	234678 HxCDF	All
"	"	% RSD 15.2	1234789 HpCDF	All

→ The laboratory analyzed a 5-point calibration for all compounds even though not required by the SOW. Because neither 234678 HxCDF or 1234789 HpCDF require initial 5-point calibrations according to the SOW, no action is necessary for these compounds.

Action: % RSD >15% approximate all the associated data (J or UJ)
% D >30% approximate all the associated data.

Was a CC3 analyzed just prior to sample analysis and every 12 hours ? yes

1. Correct concentrations used to calculate RFs –
2. Correct ions used to calculate RFs –
3. Correct IS used for target analytes –
4. Signal to noise ratios ~~≥25%~~ >10 (per SAS)
5. Chromatographic resolution - all TCDD ≤ 25 ✓
   all HxCDD ≤ 50

3

SP2331

II. INITIAL AND CONTINUING CALIBRATION

Date of Initial Calibration: 4/17/91

Date of Continuing Calibration: 4/17/91

Date	Instrument	% RSD, %D	Compound (Results)	Samples Affected
4/17	E	%RSD 30%	OCDF	
"	"	" 18%	13C-OCDD	

Action: % RSD >15% approximate all the associated data (J or UJ)
% D >30% approximate all the associated data.

Was a CC3 analyzed just prior to sample analysis and every 12 hours ? yes

1. Correct concentrations used to calculate Rfs ✓
2. Correct ions used to calculate Rfs —
3. Correct IS used for target analytes —
4. signal to noise ratio <10 (per SAS)
5. Chromatographic resolution —

4

Initial Calibration Calculations 3/8/91 8:50

- Ion Abundance Ratios :

CC1	TCDF	304/306	$\frac{298}{392} = .760$	(lab .770) ✓
	PeCDD	356/358	$\frac{71}{42} = 1.690$	(lab 1.750) ✓
CC2	PeCDF	340/342	$\frac{420}{265} = 1.585$	(lab 1.576) ✓
CC3	HxCDD	390/392	$\frac{1933}{1556} = 1.242$	(lab 1.247) ✓
CC4	HpCDF	~~6079~~ 408/410	$\frac{6079}{5805} = 1.047$	(lab 1.045) ✓
CC5	OCDF	442/444	$\frac{18497}{21028} = .880$	(lab .888) ✓

- RRF :

CC1 TCDF $\frac{298+392}{1239+1564} \times \frac{.5}{.1} = 1.231$ (lab: 1.144)

CC2 TCDD $\frac{526+\cancel{505}\ 573}{750+954} \times \frac{.5}{.25} = 1.290$ (lab 1.148)

CC3 PeCDD $\frac{494+820}{911+1187} \times \frac{.5}{.5} = .388$ (lab .509)

CC4 HpCDF $\frac{6079+5805}{1661+1591} \times \frac{1.0}{2.5} = 1.462$ (lab 1.362)

CC5 OCDD $\frac{3877+4387}{554+611} \times \frac{1.0}{10} = .709$ (lab 1.327)

- Signal to Noise Ratios all ~~less~~ greater than 10:1. —

- Chromatographic resolution – all TCDD ≤ 25 ✓
  all HxDD ≤ 50 —

CBlum

Continuing Calibration 4/2/91 14:04

- Ion Abundance Ratios

TCDF $\frac{304}{306} \rightarrow \frac{809}{1045} = .774$ (lab = .782) ✓

HxCDF $\frac{374}{376} \rightarrow \frac{1593}{1283} = 1.242$ (lab = 1.254) ✓

- RRF

TCDF $\frac{809+1045}{629+771} \times \frac{.5}{.5} = \frac{1854}{1400} = 1.324$ (lab = 1.293) ✓

HxCDF $\frac{1593+1283}{433+411} \times \cancel{\frac{1.25}{1.0}} = \frac{1.0}{1.25} = 2.73$ (lab = 2.48)

- signal to noise ratios > ~~10%~~ 10:1 ✓
- chromatographic resolution:

TCDD ≤ 25% ✓

HxCDD $\frac{1.5}{10} = 15\%$ (≤ 50%) ✓

6 OB lam

Continuing Calibration 4/3/91

- Ion Abundance Ratios

TCDD $\frac{320}{322} \rightarrow \frac{453}{576} = .786$ (lab .788 ✓)

HpCDF $\frac{408}{410} \rightarrow \frac{833}{790} = 1.054$ (lab 1.054 ✓)

- RRF

TCDD $\frac{453+576}{355+438} \times \frac{.5}{.5} = 1.298$ (lab 1.240 ✓) 5.3% ✓

HpCDF $\frac{833+790}{469+440} \times \frac{1.0}{1.25} = 1.428$ (lab 1.413)

- signal to noise ratios ~~10~~% >10:1 ✓ OK
- chromatographic resolution

TCDD <25% ✓

HxCDD $\frac{1.5}{10} = 15$ ✓ (≤ 50%) ✓

DBlank

Continuing Calibration 4/11/91

- Ion Abundance Ratios

TCDF $\frac{304}{306} \rightarrow \frac{764}{969} = .788$ (lab = .779) ✓

PeCDF $\frac{340}{342} \rightarrow \frac{470}{301} = 1.561$ (lab = 1.568) ✓

- RRf

TCDF $\frac{764+969}{604+761} \times \frac{.5}{.5} = 1.270$ (lab = 1.313) —

PeCDF $\frac{470+301}{604+761} \times \frac{.5}{.5} = .565$ (lab = .640)

- Signal to Noise - <10 >10:1 OB
- Chromatographic resolution -

TCDD ≤ 25 —

HxCDD ≤ 50 —

[illegible]

8

SP-2331

Initial Calibration Calculations 4/17/91 9:22

- Ion Abundance Ratios

CC1 TCDF $\frac{353}{450} = .784$ (lab = .785) ✓

CC2 PeCDF $\frac{586}{376} = 1.559$ (lab = 1.584) ✓

CC3 HxCDD $\frac{2228}{1799} = 1.238$ (lab = 1.243) ✓

CC4 TCDD $\frac{2781}{3537} = .786$ (lab = .782) ✓

CC5 HpCDD $\frac{6021}{5769} = 1.044$ (lab = 1.044) ✓

RRF:

CC1 TCDF $\frac{353+450}{1481+1861} \times \frac{.5}{.1} = 1.201$ (lab = 1.185 ✓)

CC2 PeCDF ~~$\frac{641+641}{03}$~~ $\frac{586+376}{1481+1861} \times \frac{.5}{.25} = .576$ (lab = .757)

CC3 HxCDD $\frac{2228+1799}{812+641} \times \frac{.5}{1.25} = 1.109$ (lab = 1.186)

~~CC4 TCDD $\frac{1140+1452}{920+1160} \times \frac{.5}{1}$~~ = OB

CC4 TCDD $\frac{2781+3537}{1207+1547} \times \frac{.5}{1} = 1.147$ (lab = 1.160)

CC5 HpCDD $\frac{6021+5769}{871+694} \times \frac{.5}{5} = .753$ (lab = .944)

- Signal to Noise Ratios greater than 10:1 (per OAS) OB
- Chromatographic Resolution. TCDD ≤ 25 ✓
  HxCDD ≤ 50

[illegible]

CCI Check

File 2910 DB-5 p.994

1. absolute retention time check of recovery standards

	R.T. 2910	R.T. CC3 ~~2639~~ 2897 OB	
C_{12} 1234-TCDD	~18:30	~~19:00~~ 18:36	~~30sec~~ 65sec ✓
C_{12} 123678-HxCDD	~29:30	~~30:10~~ 30.15	~~40sec~~ 45sec

2. signal to noise >10 for all IS and recovery standards

3. ion abundance criteria

HxCDD $\frac{390}{392}$ $\frac{194}{154}$ = 1.26 ✓ QC Limits = 1.05-1.43

TCDF $\frac{304}{306}$ $\frac{217}{279}$ = 0.78 ✓ QC Limits 0.65-0.89

10

CC1 check

File 2851 DB-5 p.973

1. absolute retention time check of recovery standards

	R.T. 2851	R.T. CC3 ~~2635~~ 2845 OB	
C_{12} 1234-TCDD	18:30	~~19:00~~ 18:36	~~30 sec~~ 6 sec
C_{12} 123678-HxCDD	~29:40	~~30:10~~ 30:15	~45 sec

2. signal to noise > 10 for all IS + recovery standards

3. ion abundance criteria

TCDF $\frac{304}{306}$ $\frac{271}{348}$ = 0.78 QC limits 0.65-0.89

PeCDF $\frac{340}{342}$ $\frac{151}{99}$ = 1.53 QC limits 1.24-1.86

[signature]

12

CCI Check

File 2862 DB-5 p. 984

1. absolute retention time check of recovery standards

	R.T. 2862	R.T. CC3 ~~2639~~ 2853		
C_{12} 1234-TCDD	~18:30	~~~19:00~~	18:33	~~~30 sec~~ 3 sec
C_{12} 123678-HxCDD	~29:40	~~~30:10~~	30:12	~~~40 sec~~ 42

2. signal to noise >10 for all Is + recovery standards

3. ion abundance criteria

HxCDD $\frac{390}{392}$ $\frac{159}{124}$ = 1.28 ✓ QC limits = 1.05 - 1.43

TCDF $\frac{304}{306}$ $\frac{176}{222}$ = 0.79 ✓ QC limits = 0.65 - 0.89

[signature]

CCI check

File 2941 SP 2331 p.789

1. absolute retention time check for recovery standards:

	R.T. 2941	R.T. CC3 2935	
C_{12} 1234 TCDD	~~13:45~~ 13:58	~~13:45~~ 13:58	✓
C_{12} 123678-HxCDD	~~22:40~~ 23:57	~~22:40~~ 23:57	✓

2. signal to noise >10 for all IS and recovery standards

3. ion abundance criteria

TCDF $\frac{304}{306}$ $\frac{270}{344}$ = 0.78 ✓ QC limits 0.65–0.89

PeCDF $\frac{340}{342}$ $\frac{215}{138}$ = 1.56 ✓ QC limits 1.24–1.86

[illegible] 13

3/8/91 8:52

F:6 2634 DB-5

III. WINDOW DEFINING MARKER

Was a window defining marker solution analyzed with the case?

	First Isomer	Last Isomer
✓TCDD	16.54	20.14
✓PeCDD	22.03	25.16
✓HxCDD	27.51	30.43
✓HpCDD	34.56	36.20
✓TCDF	15.55	20.15
✓PeCDF	20.31	25.27
✓HxCDF	26.56	31.18
✓HpCDF	34.19	37.03

Were the PCDD/PCDF isomers reported within the defined windows?

Actions: If any of the PCDD/PCDF congeners were outside the retention window make sure that new descriptors are used.

analyzed w/ IC 3/8/91 8:50

4/2/91 9:06

File 2841 DB-5

III. WINDOW DEFINING MARKER

Was a window defining marker solution analyzed with the case?

	First Isomer	Last Isomer
✓TCDD	16:29	19:44
✓PeCDD	21:32	24:43
✓HxCDD	27:17	30:07
✓HpCDD	34:19	35:43
✓TCDF	15:30	19:45
✓PeCDF	20:01	24:54
✓HxCDF	26:22	30:42
✓HpCDF	33:42	36:26

Were the PCDD/PCDF isomers reported within the defined windows? yes

Actions: If any of the PCDD/PCDF congeners were outside the retention window make sure that new descriptors are used.

analyzed prior to CC 4/2/91 14:04 ✓

4/3/91 8:56

File 3852

DB-5

III. WINDOW DEFINING MARKER

Was a window defining marker solution analyzed with the case?

	First Isomer	Last Isomer
→ TCDD	16:31	19:47
PeCDD	21:38	24:46
HxCDD	27:19	30:10
HpCDD	34:22	35:45
TCDF	15:33	19:48
PeCDF	20:04	24:57
HxCDF	26:24	30:45
HpCDF	33:44	36:28

Were the PCDD/PCDF isomers reported within the defined windows? yes

Actions: If any of the PCDD/PCDF congeners were outside the retention window make sure that new descriptors are used.

analyzed prior to CC 4/3/91 9:46 ✓

9/11/91 7:44

DB-5

III. WINDOW DEFINING MARKER

F162896

Was a window defining marker solution analyzed with the case?

	First Isomer	Last Isomer
✓TCDD	16:33	19:22
✓PeCDD	21:38	24:49
✓HxCDD	27:23	30:15
✓HpCDD	34:26	35:51
✓TCDF	15:35	19:51
✓PeCDF	20:06	25:01
✓HxCDF	26:28	30:50
✓HpCDF	33:49	36:34

Were the PCDD/PCDF isomers reported within the defined windows? yes

Actions: If any of the PCDD/PCDF congeners were outside the retention window make sure that new descriptors are used.

analyzed prior to CC 6/11/91 8:35

File 2934

Apil 17, 1991

III. WINDOW DEFINING MARKER

5P2331

Was a window defining marker solution analyzed with the case?

	First Isomer	Last Isomer
TCDD		
PeCDD		
HxCDD		
HpCDD		
TCDF		
PeCDF		
HxCDF		
HpCDF		

Were the PCDD/PCDF isomers reported within the defined windows? yes

Actions: If any of the PCDD/PCDF congeners were outside the retention window make sure that new descriptors are used.

analyzed prior to IC 4/17/91 9:22

The laboratory reported in the case narrative that since the 12/90 SOW does not provide information on first and last elutes for each PCDD/PCDF group, SMO was contacted to determine what congeners were needed for the window defining mix. SMO informed the laboratory that the primary interest was in determining specific 2378 congeners, therefore the laboratory used the CC3 calibration standard to set descriptor switching times for 2378 congeners.

IV. COLUMN PERFORMANCE RESOLUTION CHECK

Was the chromatographic resolution of ^{13}C-TCDDs and HxCDDs in the CC3 solution for DB-5 columns calculated for each 12 hour period?

% Valley ^{13}C-2378-TCDD/^{13}C-1234-TCDD _____ (QC limit ≤25%)

% Valley between HxCDDs _____ (QC limit ≤50%)

For SP-2331 columns:

% Valley 1478-TCDD/2378-TCDD _____ (QC limit ≤25%)

% Valley 2378-TCDD/(1237/1238)-TCDD _____ (QC limit ≤25%)

Actions: If the peak resolution is >25 %, the reviewer must use his/her professional judgement on the severity of the problem and its effect on the final results.

DB-5

IC 3/8/91 CC3 TCDD 1% ✓
HxCDD 20% ✓

CC 3/2/91 CC3 TCDD 3% ✓
HxCDD 15% ✓

~~CC 4/17/91 CC3 TCDD 4%~~ 95

CC 4/3/91 CC3 TCDD 3% ✓
HxCDD 15% ✓

CC 4/11/91 CC3 TCDD 4% ✓
HxCDD 20% ✓

V. METHOD BLANKS

Was a method blank prepared and analyzed for each matrix prior to analysis of samples?

Yes [✓] No [ ]

Blank ID	Compound	<2% IS Signal	>2% IS Signal
1. DFBLK2	OCDD	0.020 EMPC	

all samples analyzed with DFBLK2 with positive OCDD or EMPC OCDD will be estimated (J) with the exception of 6149A-05 and 6149A-08 (significant concentration of OCDD present).

this blank also had total TCDD contamination and was reanalyzed as DFBLK3.

2. DFBLK3 this blank also had significant total TCDF contamination (>5%).

this is a reanalysis of DFBLK2. Total TCDD contamination >5% (31a% of IS.). All samples should have been reanalyzed but were not. Therefore in all samples, total TCDD will be estimated (J)

there was also still significant total TCDF contamination so all total TCDFs will be estimated (J)

3. DFBLK1 this blank had ~~Total~~ TCDF and TCDD contamination ∴ all totals will be estimated (J)

Action: If a method blank associated with a sample group is contaminated the associated positive samples and any sample containing any peaks that do not meet all identification criteria must be reextracted and reanalyzed.

2896 BLK
2847 -01

2848 BLK
2849 -2
2850 -3
2854 -4
2855 -4S
2856 -4D
2858 -5
2860 -6
2857 -7
2859 -8
2905 -8DL
2861 -9
2940 -8C

2906 BLK
2907 -06RE

Method Blks

DFBLK1 File 2846B

- possible HxCDD contamination

  390 & 392 peaks, but no $M-[COCl]^+$ peak ∴ no confirmation of identification

  $\frac{155.26}{5037.60} = 0.03 \times 100 = 3.08$ → <5% limit for chemical interference ∴ no contamination

- EMPC calculation TCDF $\frac{50ng \times 81.67 \times 1}{1\ell \times 6800 \times 1.293} = .464$ Clab .464

EDI calculation PeCDF $\frac{2.5 \times 50ng \times (.611 + \cancel{2.190}\ 1.827)}{1\ell \times (819 + 1009) \times .650} = \frac{304.8}{1188} = .257$ (lab .28

DFBLK2 F.6 2848

- possible TCDD contamination

  320/322 but no 259 ion ⇒ no confirmation of ID

  $\frac{1560.58}{8248.09} = .189 \times 100 = 18.9\%$ but not w/in R.T. window

  therefore, no contamination

- EMPC Calculation TCDD $\frac{50ng \times (1560.58 + \cancel{8248.09}\ CB) \times 1}{10g\ \cancel{\ell} \times (\cancel{8248.09}\ 3684.13) \times 1.236} = 1.714$ ✓

  TCDF $\frac{50ng \times 61.98 \times 1}{10g \times 5646.43 \times 1.293} = .042$ (lab .042)

- EDL Calculation 1,2,3,7,8-PeCDF $\frac{2.5 \times 50ng \times (.499 + .950) \times 1}{10g \times (652 + 822) \times (\cancel{.597}\ CB)(.642)} = \frac{181}{9463}$ .01

  (lab 6019

DFBLL3 RE FILE 2906

- possible HpCDD contamination
  434/426 - ion ratio off $\frac{1141}{336} = 3.4$ (.88 - 1.20)
  outside of R-T. window 33.41 (35.47)

- EMPC calculation TCDF $\frac{50ng \times 39.60 \times 1}{10g \times 3884.71 \times 1.313} = .039$ Clab = .039

- EDL calculation PeCDF $\frac{2.5 \times 50ng \times (.245 + .330) \times 1}{10g \times (434 + 556) \times (.640)} = \frac{71.88}{6336}$ (.011)
  (lab = .0

6149A-04S

VI. MATRIX SPIKE

List percent recovery which did not meet the limits criteria.

Compound	MS % Recovery	Limits
TCDD	_____	60-140
PeCDD	_____	60-140
HxCDD	_____	60-140
HpCDD	_____	60-140
OCDD	_____	60-140
TCDF	_____	60-140
PeCDF	_____	60-140
HxCDF	_____	60-140
HpCDF	_____	60-140
OCDF	_____	60-140

Actions: Recheck calculations

all % Recoveries within criteria -
calculations on separate sheets ->

24

lab analyzed MSD

VII. DUPLICATE

04-MSD

Was a duplicate run for each matrix?

Yes [✓] No [ ]

The RPD of each analyte detected must be within 50-150% range.

all RPD's within range.

Actions: Recheck all calculations if beyond the specified range. Professional judgement should be used to ascertain effect on final data.

6149A-045 File 2855

2378 TCDD

ion ratio $\frac{320}{322} \rightarrow \frac{1487.27}{288.49} = 5.16$ —

$\frac{1925.65}{2449.08} = .79$ —

Concentration $\frac{50ng \times 4375}{12.21g \times 4070 \times 1.240} = 8.55$ —

% R $\frac{50ng}{12.21g} = 4.095 ng/kg$ $\frac{8.55}{4.095} \times 100 = 86.7\%$ —
(lab = 86.7%)

~~Sample $\frac{(3.550 \times 12.21g) - (3.912 \times 12.21g)}{\frac{(3.550 \times 12.21) + (3.912 \times 12.21g)}{2}} =$~~ (Dup.) (11.58)

Sample 3.550
dup 3.912

$\frac{(43.341) - (45.301)}{44.324} = 4.4\%$ —
(lab = 4.4)

6149A-04D

2378-TCDD $\frac{320}{322} \rightarrow \frac{1233.18}{237.97} = 5.18$

$\frac{1594.78}{2042.22} = .78$

concentration $\frac{50ng \times 3637 \text{ (at } 18:43)}{11.58g \times 3237 \text{ (at } 18:42) \times 1.240} = 3.912$

%R $\frac{50ng}{11.58} = 4.318$ ug/kg $\frac{3.912}{4.318} \times 100 = 90.6\%$
(lab = 90.6)

REGION I
Data Review Worksheets

VIII. FIELD DUPLICATE PRECISION

TR Nos. 6149A-05, 6149A-06RE

Matrix: SOIL

List the concentrations of the compounds which do not meet the follow RPD criteria:

1. An RPD of <30% for water duplicates.
2. An RPD of <50% for soil duplicates.

FRACTION	COMPOUND	SAMPLE CONC	DUP SAMPLE CONC	RP
—	2378 TCDF	.017	.020	16.2
—	1234678 HpCDF	.036	.047	26.5
—	1234678 HpCDD	.233	.259	10.6
—	OCDD	4.710	4.521	4.1
—	OCDF	0.0120	.196 J	

The positive OCDF value in 6149A-06RE will be estimated
The non-detected OCDF value in 6149A-05 will be estimated

ACTIONS:

1. If the results for any componds do not meet the RPD criteria, flag the positive results for that compound as estimated.
2. If one value is non-detected, and one is above the CRQL:
   a. Flag the positive result as estimated (J).
   b. Flag the non-detected result as estimated (UJ).

NOTE: Professional judgement may be utilized to apply duplicate actions to all samples of a similar matrix.

A separate worksheet should be filled out for each field duplicate pair

VIII. RECOVERY STANDARD RESPONSE

CC-3 Standard ID: 2845

Column: DB-5

	^{13}C-1,2,3,4-TCDD	^{13}C-1,2,3,7,8,9-HxCDD
Area Counts		
Upper Limit	85800	45800
Lower Limit	171600	91600
	42900	22900

List samples which did not meet criteria.

(Sample 6149A-01 met ✓
-02 met ✓
-03 met ✓)

all O.K.

CC-3 Standard ID: 2853

Column: DB-5

	^{13}C-1,2,3,4-TCDD	^{13}C-1,2,3,7,8,9-HxCDD
Area Counts		
Upper Limit	753	417
Lower Limit	1507	834
	377	209

List samples which did not meet criteria.

(Sample 6149A-04 ✓
-04MS ✓
-04MSD ✓
-05 ✓
-06 ✓
-07 ✓
-08 ✓
-09 ✓)

all O.K.

CC-3 Standard ID: 2897

Column: DB-5

	^{13}C-1,2,3,4-TCDD	^{13}C-1,2,3,7,8,9-HxCDD
Area Counts		
Upper Limit	878	460
Lower Limit	1756	920
	439	230

List samples which did not meet criteria.

(Sample 6149A-08DL ✓
-06RE ✓)

Sample is a 1:20 DL, internal standards ~~Recovery Standards~~ were diluted out.

all O.K.

CC3 - Standard ID: 2935 SP2331

	BC-1234-TCDD	13C 123789-HxCDD
Area counts		
Upper	2080	1453
Lower	4160	2906
	1040	726.5

(Sample 6149A-08DL ✓)

O.K.

X. TOXIC EQUIVALENT FACTOR (TEF)

Were samples with a TEF > 0.7ug/Kg for soil/sediment or fly ash; 7.0ug/Kg for chemical waste and 0.007ug/L for aqueous samples confirmed on SP-2300, SP-2331 or equivalent column?

Were EMPC values were included in the TEF calculations?

Yes [✓] No [ ] EDLs also -

Check that the TEF values were calculated including EMPCs using the following guidelines:

Compound	Multiplying Factor	Concentration Toxic Equivalent
2,3,7,8-TCDD	1.00	
Other TCDD	0.00	
2,3,7,8-PeCDD	0.50	
Other PeCDD	0.00	
2,3,7,8-HxCDDs	0.10	
Other HxCDD	0.00	
2,3,7,8-HpCDD	0.01	
Other HpCDD	0.00	
OCDD	0.001	
2,3,7,8-TCDF	0.100	
Other TCDF	0.000	
1,2,3,7,8-PeCDF	0.050	
2,3,4,7,8-PeCDF	0.500	← [illegible] 0.05 [illegible] for all samples -
Other PeCDF	0.000	
2,3,7,8-HxCDF	0.100	
Other HxCDF	0.000	
2,3,7,8-HpCDF	0.010	
Other HpCDF	0.000	
OCDF	0.001	
Total Toxic Equivalent		

Reference: "Interim Procedure for Estimating Risk Associated with Exposures to Mixtures of Chlorinated Dibenzo-p-dioxins and Dibenzofurans (CDDs and CDFs)" EPA/625/3-89/016.

Sample 6NGA-03 had total TEF > 0.7 ug/kg - however, second column confirmation not necessary since it was the PE sample.

Sample 6NGA-08DL had total TEF > 0.7 ug/kg and was confirmed on the SP2331 column.

XI SAMPLE CALCULATION:

See [illegible] sample calculation of concentrations [illegible]

$$\text{Concentration (ng/g)} = \frac{Qis \times (Ax^1+Ax^2)}{(W \text{ or } V) \times (Ais^1+ais^2) \times RRFx \times D}$$

$$EDL = \frac{2.5 \times Qis \times (Hx^1+Hx^2) \times D}{(W \text{ or } V) \times (His^1+His^2) \times RRFx}$$

$$EMPC = \frac{Qis \times (Ax^1+Ax^2) \times D}{W \text{ or } V \times (Ais^1+Ais^2) \times RRFx}$$

where:

Qis = quantity (ng) of appropriate internal standard added to sample before extraction

Ax^1 and Ax^2 =integrated areas of the two quantitation ions

W and V = weight (g) or volume (L) of sample extracted

RRFx = calculated relative response factor from the continuing calibration

Hx^1+Hx^2 = peak heights of the noise for the quantitation ions

His^1+His^2 = peak heights of the internal standard quantitation ions

D = dilution

Sample 6149A-01
File 2847

CC3 = 2845

TCDD: ① ion ratio $\frac{304}{306} \rightarrow \frac{53.56}{26.14} = 2.05$ range = .65 - .89

② rt of TCDD in CC3 = 18:06 → rt ≠ meet

rt of TCDD in sample = 18:33

③ 3rd ion present, signal to noise < 2.5

no TCDD present

recovery of IS -

13C 2,3,7,8 TCDD ion ratio $\frac{332}{334} \rightarrow \frac{600}{746} = .80$ (lab = .79) ✓

$\frac{5027.12 \times 50ng}{5396.12 \times 1.104} = 84.4\%$ ✓ (lab = 84%) ✓

13C 123678 HxCDD ion ratio $\frac{402/404}{1555.49 / 1192.53} = 1.30$ (lab = 1.30) ✓

$\frac{2748.02 \times 50ng}{3272.74 \times .900} = \frac{46.65}{50.00} = 93\%$ (lab = 93%) ✓

R.T. & recovery stds		CC3
13C12 1234-TCDD	18:32 ✓	18:36
13C12 123789-HxCDD	30:13 ✓	30:15

EDL PeCDF $\frac{2.5 \times 50ng \times (.61 + 1.41)}{1\ell \times (886 + 1103) \times .642} = \frac{252.5}{1276.94} = 0.2$ ✓ (lab = .184)

Total TCDD $\frac{50ng \times 2223.27}{1\ell \times 5027.12 \times 1.236} = 17.891$ (lab = 17.891) ✓

Sample 6149A-02
File 2849

CC3 = 2845

2378-TCDD ion ratio $\frac{370}{372} \rightarrow \frac{1098.75}{215.69} = 5.1$ ↑

3rd ion 259 does not match ∴ no 2378TCDD present

OCDD can't check ion ratio

↳ $\frac{100ng \times 11.84}{9.48g \times 1304.61 \times 1.438} = 0.067$ (lab = 0.067 ✓)

recovery of IS

13C 2378TCDD ion ratio $\frac{332}{334} \rightarrow \frac{1195.62}{1479.89} = .81$ (lab .81) ✓

$\frac{2675.51 \times 50ng}{3430.40 \times 1.104} = \frac{35.32}{50} = 71\%$ (lab 70.6%) ✓

13C 123678HxCDD ion ratio $\frac{402}{404} \rightarrow \frac{780.1}{602.76} = 1.29$ (lab 1.29 ✓) ✓

$\frac{1382.94 \times 50ng}{1659.85 \times .900} = \frac{46.29}{50} = 93\%$ (lab 92.6%) ✓

R.T. of recovery stds

		CC3
$13C_{12}$ 1234 TCDD	18:32 ✓	18:36
$13C_{12}$ 123789 HxCDD	30:11 ✓	30:15

EDL PeCDD $\frac{2.5 \times 50ng \times (.702 + .428)}{(.81)\ 9.48g \times (402 + 489) \times .515} = \frac{141.25}{3523.53} = .040$ (lab .037)

Total TCDF $\frac{50ng \times (50.50 + 0.08)}{9.48g \times 4156.01 \times 1.293} = 0.059$ (lab = 0.059) ✓

total TCDD $\frac{50ng \times 2391}{8.18g \times 2087.57 \times 1.236} = 5.664$
(lab = 5.667)

Sample 61-GA-03
File 2850

CC3 = 2845

• recovery of IS.

13C 2378 TCDD ion ratio $\frac{332}{334} \rightarrow \frac{1273.04}{1592.25} = .80$ (lab .81)

$\frac{2087.57 \times 50ng}{2865.29 \times 1.104} = \frac{33}{50} = 66\%$ (lab = 65%)

13C 123678 HxCDD ion ratio $\frac{402}{404} \rightarrow \frac{700.33}{529.79} = 1.32$ (lab = 1.32)

$\frac{1230.12 \times 50ng}{1483.59 \times .900} = \frac{46.06}{50} = 92\%$ (lab = 92.1%)

• R.T. of recovery stds

		CC3
$13C_{12}$ 1234 TCDD	18:33	18:36
$13C_{12}$ 123789-HxCDD	30:12	30:15

• 2378 TCDD - $\frac{320}{322} \rightarrow \frac{297.77}{379.26} = 0.79$ $\frac{325.79}{419.87} = 0.78$

$\frac{50ng \times 745.66}{8.18g \times 2087.57 \times 1.236} = 1.77$ (lab = 1.766)

• OCDD EMPC $\frac{100ng \times 8.66}{8.18g \times 1172.23 \times 1.438} = .062$

• 12378 PeCDF EDL $\frac{2.5 \times 50ng \times (.562 + .867)}{8.18g \times (350 + 441) \times .642} = .043$ (lab = .041)

Sample 6149A-04
File 2854

CC3 = 2853

1. Retention Time of Recovery Stds

		CC3
$^{13}C_{12}$1234-TCDD	18:32	18:33 ✓
$^{13}C_{12}$123789 HxCDD	30:12	30:12 ✓

2. Recovery of I.S.

- 13C 2378 TCDF ion ratio $\frac{316}{318} \rightarrow \frac{2131.34}{2656.56} = .80$ (lab = .80) ✓

$$\frac{50 \times 4787.90}{1.813 \times 4118.19} = \frac{32}{50} \times 100 = 64\%$$ (lab = 64.1) ✓

- 13C 1234678 HpCDF ion ratio $\frac{420}{422} \rightarrow \frac{3203.91}{3020.93} = 1.061$ (lab = 1.06)

$$\frac{50 \times 6624.84}{1.118 \times 2859.41} = \frac{103.616}{100} \times 100 = 104$$ (lab 97.4)

3. OCDD ion ratio $\frac{458}{460} \rightarrow \frac{16.11}{15.40} = 1.046$ (lab 0.96) ?

$$\frac{100ng \times 29.26}{11.32g \times 2705.83 \times 1.426} = 0.067$$ (lab = 0.067) ✓

4. EDL calculation

2378 TCDD $\frac{2.5 \times 50ng \times (.237 + .253)}{11.32 \times (479 + 593) \times 1.240} = \frac{61.24}{15047.5} = .004$ (lab = .00

5. total TCDF $\frac{50ng \times 51.04}{11.32g \times 4787.90 \times 1.293} = 0.036$ (lab = 0.036) ✓

Sample 6149A-05
File 2858

CC3 = 2853

1. Retention Time of Recovery Std

		CC3
$^{13}C_{12}$ 1234-TCDD	18:33	18:33 ✓
$^{13}C_{12}$ 123789 HxCDD	30:13	30:12 ✓

2. Recovery of I.S.

- $^{13}C_{12}$ 2378 TCDF ion ratio $\frac{316}{318} \rightarrow \frac{2627.01}{3285.66} = 0.80$ (lab = 0.80) ✓

$$\frac{50 \times 5912.67}{1.813 \times 4106.56} = \frac{39.71}{50} \times 100 = 79.4\% \quad (\text{lab} = 79.4\%)$$ ✓

- $^{13}C_{12}$ 34678 HpCDF ion ratio $\frac{420}{422} \rightarrow \frac{3772.03}{3538.45} = 1.07$ (lab = 1.07)

$$\frac{50 \times 7310.48}{1.118 \times 3025.58} = \frac{108.05}{100} \times 100 = 108.1\% \quad (\text{lab} = 108.1)$$ ✓

3. EDL 2378 TCDD

$$\frac{2.5 \times 50ng \times (.556 + .441)}{8.24g \times (465 + 577) \times 1.240} = \frac{124.6}{10646.7} = .012$$ ✓ (lab = .01)

4. EMPC 123789 HxCDF

$$\frac{100ng \times 19.80}{8.24g \times 7310.48 \times 1.533} = 0.021 \quad (\text{lab} = 0.021)$$

5. 1234678 HpCDD

$$\frac{50ng \times 69.85}{8.24g \times 2425.15 \times .750} = .233 \quad (\text{lab} = .233)$$ ✓

6. Total TCDD 1 pk

$$\frac{50ng \times 1750.71}{8.24g \times 3798.16 \times 1.240} = 2.256 \quad (\text{lab} = 2.256)$$ ✓

7. Total TCDF 11 pks.

$$\frac{50ng \times 362.98}{8.24 \times 5912.67 \times 1.292} = .288 \quad (\text{lab} = .288)$$ ✓

not used - 13C OCDD %R out

see RE

Sample 6149A-06
File 2860

CC3 = 2853

1. Retention time of Recovery stds

		CC3
$^{13}C_{12}$ 1234-TCDD	18:34	18:33
$^{13}C_{12}$ 123789 HxCDD	30:14	30:12

2. Recovery of I.S.

- $^{13}C_{12}$ 2378 TCDF ion ratio $\frac{316}{318} \rightarrow \frac{861.67}{1073.01} = 0.80$ (lab=0.80)

$$\frac{50 \times 3300.18}{1.813 \times 2014.78} = \frac{45.17}{50} \times 100 = 90.3\% \quad (\text{lab} = 90.3\%)$$

- $^{13}C_{12}$ 34678 HpCDF ion ratio $\frac{420}{422} \rightarrow \frac{1442.76}{1341.21} = 1.08$ (lab=1.08)

$$\frac{50 \times 2783.97}{1.118 \times 1249.92} = \frac{99.61}{100} \times 100 = 99.6\% \quad (\text{lab} = 99.6\%)$$

3. EDG 2378 TCDD $\frac{2.5 \times 50ng \times (.184 + .212)}{7.7g \times (247 + 303) \times 1.240} = \frac{49.50}{5251.4} = .009$ (lab = .01

4. EMPC 1234678 HpCDF $\frac{100ng \times 12.57}{7.7g \times 2783.97 \times 1.413} = \frac{1257}{30289.87} = 0.041$ (lab=0.

5. 2378 TCDF $\frac{50ng \times 11.39}{7.7g \times 3300.18 \times 1.292} = 0.017$ (lab = 0.017)

6. total TCDF 8pks $\frac{50ng \times 165.29}{7.7g \times 3300.18 \times 1.292} = 0.252$ (lab=0.252

Sample 6149A - 06RE CC3 = 2897

F.6 2907

1. Retention time of recovery standards

		CC3
13C 1234 TCDD	18:31	18:35 ✓
13C 123789 HCDD	30:11	30:16 ✓

2. Recovery of internal standards

13C 2378 TCDF $314/318 \rightarrow \frac{1988.74}{2482.22} = .80$ (lab = .80) ✓

$$\frac{4474.96 \times 50ng}{1.833 \times 2770.91} = \frac{44.05}{50} \times 100 = 88.1\%$$ (lab = 88.1%) ✓

3. EDL 2378 TCDD

$$\frac{2.5 \times 50ng \times (.406 + .423)}{7.7g \times (320 + 393) \times 1.256} = \frac{103.62}{6895.6} = .015$$

(lab = .015

4. EMPC 123789 HxCDF

$$\frac{100ng \times 11.29}{7.7g \times 3561.95 \times 1.508} = 0.027$$ (lab = 0.027) ✓

5. 2378 TCDF

$$\frac{50ng \times 18.08}{7.7g \times 4474.96 \times 1.313} = .020$$ (lab = .020) ✓

214

6. total TCDF 10pks

$$\frac{50ng \times 284.59}{7.7g \times 4474.96 \times 1.313} = 0.315$$ (lab = 0.315) ✓

↳ leave out 3pks w/o Ratio/RT fit

$$\frac{50ng \times 214.21}{7.7g \times 4474.96 \times 1.313} = 0.24$$ (lab = 0.24) ✓

Sample 6149A-07
File 2857

CC3 = 2853

1. Retention time of Recovery Stds

		CC3
$^{13}C_{12}$ 1234 TCDD	18:34	18:33 ✓
$^{13}C_{12}$ 23789 HxCDD	30:14	30:12 ✓

2. Recovery of IS

- $^{13}C_{12}$ 2378 TCDF ion ratio $\frac{316}{318} \rightarrow \frac{947.87}{1179.13} = .80$ (lab = .80) ✓

$$\frac{50 \times 3425.47}{1.813 \times 2486.86} = \frac{37.99}{50} \times 100 = 76\%$$ (lab = 76%) ✓

- $^{13}C_{12}$ 34678 HpCDF ion ratio $\frac{420}{422} \rightarrow \frac{1570.80}{1466.58} = 1.07$ (lab = 1.07) ✓

$$\frac{50 \times 3037.38}{1.118 \times 1475} = \frac{92.09}{100} \times 100 = 92.1\%$$ (lab = 92.1%) ✓

3. EDL 2378TCDD $\frac{2.5 \times 50ng \times (.346 + .385)}{15.69g \times (283 + 354) \times 1.240} = \frac{91.38}{12393.22} = .007$ (lab = .00

4. OCDD $\frac{100ng \times 10.98}{15.69g \times 1380 \times 1.426} = .036$ (lab = .036) ✓

5. total TCDD $\frac{50ng \times 1089.25}{15.69g \times 7127.00 \times 1.240} = 1.316$ (lab = 1.316) ✓

Sample 6149A-08
File 2859

CC3 = 2853

1. Retention time of Recovery Stds

		CC3
13C 1234 TCDD	18:32	18:33 ✓
13C 123789HxCDD	30:13	30:12 ✓

2. Recovery of IS

13C2378 TCDF ion ratio $\frac{316}{318} \rightarrow \frac{3163.92}{3901.57} = 0.81$ (lab = 0.81) ✓

$\frac{50ng \times 7065.49}{1.813 \times 4453.24} = \frac{43.76}{50} \times 100 = 87.5\%$ (lab = 87.5%)

13C 1234678 HpCDF ion ratio $= \frac{420}{422} \rightarrow \frac{4291.89}{4024.73} = 1.07$ (lab = 1.07) ✓

$\frac{50ng \times 8316.62}{1.118 \times 3357.92} = \frac{110.766}{100} \times 100 = 110.8\%$ (lab = 110.8%) ✓

3. EDL 123478 HxCDD $\frac{2.5 \times 50ng \times (.818 + .682)}{10.04g \times (3414674) \times 1.132} = \frac{187.5}{8876.85} = .027$ (lab = .027) ✓

4. EMPC 12378 PeCDD $\frac{50ng \times 31.16}{10.04g \times 4321.53 \times .536} = .067$ (lab = .067) ✓

5. 2378 TCDD $\frac{50ng \times 18.52}{10.04g \times 4321.53 \times 1.240} = 0.017$ (lab = 0.017) ✓ /471

6. 1234789 HpCDF $\frac{100ng \times 734.71}{10.04g \times 8316.62 \times .960} = 0.917$ (lab = 0.917) ✓

7. total HxCDD (6 pks) $\frac{50ng \times 9667.47}{10.04g \times 2911.44 \times 1.132} = 14.61$ (lab = 14.04)

→ total concentration - leave out pk at 30:14 (just outside of window - $\frac{50ng \times 9103.56}{10.04g \times 2911.44 \times 1.132} = 13.76$ (lab = 13.74)

1:20 DL

Sample 6149A-08DL CC3 = 2897

File 2905

1. Retention time of Recovery stds

		CC3
13C1234 TCDD	18:31	18:35 ✓
13C123789 HxCDD	30:12	30:16 ✓

2. Recovery of Internal Standards

13C 2378 TCDF 316/318 → $\frac{54.83}{68.36}$ .80 (lab = .80) ✓

$\frac{50ng \times 123.19}{1.833 \times 2322.21} = 1.447 \times 20 = \frac{28.94}{50} \times 100 = 57.9\%$ (lab = 57.9%) ✓

3. EDL 2378 TCDD $\frac{2.5 \times 50ng \times (.237 + .234)}{1.256 \times (\sim 30) \times 10.04g} = \frac{59.5}{378} = .157$ (lab = .562)

Column 8 P2331 1:20 PM

Sample 6149A-08DL
File 2940

CC3 = 2935

1. Retention time of recovery stds

		CC3
13C 1234TCDD	13:57	13:58 ✓
13C 123789HxCDD	23:57	23:57 ✓

2. Recovery of IS

ion ratio 332/334 → 74.30 / 101.59 = 0.73 ✓ (lab=0.7

13C 2378TCDD

$$\frac{50ng \times 175.89}{1.035 \times 4253.26} = 1.99 \times 20 = \frac{39.96}{50} \times 100 = 79.9$$

(lab=79.9)

3. EDL 2378TCDD

$$\frac{2.5 \times 50ng \times (.515 + .521)}{10.04g \times 3141.84 \times 1.233} = \frac{129.5}{321.86}$$

(lab=.297)

4. EMPC 123478HxCDF

$$\frac{100ng \times 39.91}{10.04g \times 366.62 \times 1.823} \times 20 = 11.89$$

(lab=1.112)

5. 123478HxCDD

$$\frac{50ng \times 13.60}{10.04g \times 4036.97 \times 1.186} \times 20 = .283$$

?

(lab=.684)

42

Sample 6149A-09
File 2861

CC3 = 28.53

1. Retention time of recovery standards

		CC3
13C 1234 TCDD	18:34	18:33 ✓
13C 123789 HxCDD	30:14	30:12 ✓

2. Recovery of I.S.

- 13C 2378 TCDF ion ratio $\frac{316}{318} \rightarrow \frac{1592.67}{1971.30} = 0.81$ (lab = 0.81) ✓

$$\frac{50ng \times 3563.97}{1.813 \times 2820.12} = \frac{34.85}{50} \times 100 = 69.7\%$$ (lab = 69.7 %) ✓

- 13C 1234678 HpCDF ion ratio $\frac{424}{422} \rightarrow \frac{1903.67}{1776.73} = 1.07$ (lab = 1.07) ✓

$$\frac{50ng \times 3680.40}{1.118 \times 1848.61} = \frac{89.0}{100} \times 100 = 89.0\%$$ (lab = 89.0 %) ✓

3. EDL 2378 TCDD $\frac{2.5 \times 50ng \times (4124 + 4126)}{10.14g \times (320 + 404) \times 1.240} = \frac{104.75}{9103.29} = .012$ (lab = .013) ✓

4. 1234678 HpCDF $\frac{100ng \times 8.64}{10.14g \times 3680.40 \times 1.413} = .016$ (lab = .016) ✓

5. total HpCDD 2pks $\frac{50ng \times 27.15}{10.14g \times 15\,30.16 \times .750} = 0.12$ (lab = 0.12) ✓

6. total TCDF 7pks $\frac{50ng \times 80.59}{10.14g \times 3563.97 \times 1.292} = \frac{4029.5}{46,691.14} = .086$

(lab = .123)

total = 0.04
2,3,7,8 TCDF 0.007

The laboratory was requested to provide an explanation for their reported result of 0.123 for total TCDF. They replied that 7 pks were used in the quantitation. However, upon recalculation, the validator still gets 0.086. The value 0.086 will be reported on the data summary tables.

the lab confirmed the validator's calculation and will resubmit forms

Sample 6149A - 04S CC3 = 2853
File 2855

1. Retention time of Recovery stds CC3

13C1234 TCDD	18:33	18:33 ✓
13C 123789 HxCDD	30:14	30:12 ✓

2. Recovery of I.S.

13C 2378 TCDF ion ratio $\frac{316}{318} = \frac{2856.78}{3572.04} = 0.80$ ✓

$$\frac{50ng \times 6428.82}{1.813 \times 4104.52} = \frac{43.20}{50} \times 100 = 86.4\%$$

Sample 6149A-04D CC3 - 2853
File 2856

1. Retention time of Recovery Stds

		CC3
13C1234 TCDD	18:33	18:33 ✓
13C123789 HxCDD	30:14	30:12 ✓

2. Recovery of I.S.

13C2378 TCDF Ion ratio $\frac{316}{318} = \frac{2205.40}{2746.60} = .80$ ✓

$$\frac{50ng \times 4952}{1.813 \times 3631.59} = \frac{37.61}{50} \times 100 = 75.2\%$$

In Reference to Case No(s):
6149A

Contract Laboratory Program
REGIONAL/LABORATORY COMMUNICATION SYSTEM
Telephone Record Log

Date of Call: 7/26/91

Laboratory Name: Triangle Laboratories

Lab Contact: Marie Vasiadou

Region: I

Regional Contact: Weston/ESAT

Call Initiated By: ___ Laboratory ✓ Region

In reference to data for the following sample number(s):

- 6149A-09
- missing raw data for window defining mix File 2852

Summary of Questions/Issues Discussed:

- laboratory was asked to recheck EDL EMPC calculation for total TCDF for sample 6149A-09
- laboratory was asked to submit raw data for window-defining mix File 2852

Summary of Resolution:

- laboratory rechecked calculations and will submit corrected forms on July 29-30, 1991.
- laboratory will also submit raw data for window-defining mix.

Signature [signature] Date 7/26/91

Distribution: (1) Lab Copy, (2) Region Copy, (3) SMO Copy

In Reference to Case No(s):
6149A

Contract Laboratory Program
REGIONAL/LABORATORY COMMUNICATION SYSTEM
Telephone Record Log

Date of Call: 7/11/91

Laboratory Name: Triangle Laboratories

Lab Contact: Marie Vasliadou

Region: I

Regional Contact: Weston/ESAT

Call Initiated By: ___ Laboratory ✓ Region

In reference to data for the following sample number(s):

Summary of Questions/Issues Discussed:

1. Need summary of area counts for CC3 and IC standards in order to calculate response factors and to confirm recovery standards
2. Need raw data for window defining mix File 2852 analyzed on 4/3/91
3. TEF value for 23478 PeCDF is listed as 0.05 - is this correct?
4. Total TEF values calculated using EDL values
5. cannot confirm total TCDF value for sample 6149A-09
6. cannot confirm concentration values for 6149A-08DL

Summary of Resolution:

1. lab will fedex summary forms by 7/15-7/16
2. " " " raw data " " "
3. lab stated that there is discrepancy in TEF value in 12/90 SOW
4. lab will submit calculations
5.

Signature: [signature] Date: 7/11/91

Distribution: (1) Lab Copy, (2) Region Copy, (3) SMO Copy

→ there is a discrepancy in 12/90 SOW - Form I PCDD-2 lists TEF as 0.05
P: D-56 lists TEF as 0.5
lab will change their form and [illegible]