US EPA RECORDS CENTER REGION 5
461224

Kevin:

These are the analytical results for the 5 samples (E01 → E05) collected in April from the Chemetco facility in Alton, IL. The slag sample (E05) had only trace levels of octa-dioxin and hepta and octa-furans. However, the sample from the polish pit (E01) showed low concentrations of all dioxins and furans (3.48 ppb 2,3,7,8-TCDD equivalents) and .11 ppb 2,3,7,8-TCDD. These levels are below the 5 ppb level of concern for dioxin in soils established by the CDC. Any questions, let me know.

Don Bruce C-7241

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION V

E: 5/12/87

T. Review of Region V CLP Data
Received for Review on 5/1/87

M. Curtis Ross, Director (SSCRL) Patrick J. Churilla
Central Regional Laboratory

O: Data User: Fit

We have reviewed the data for the following case(s).

SITE NAME: Chemetco SMO Case No. SAS 2782E

EPA Data Set No. SF 3916 No. of Samples: 5 D.U./Activity Numbers 4205 C72100

CRL No. 87FB05505 - 87FB05505

SMO Traffic No. E01 - E05

CLP Laboratory: Triangle Hrs. Required for Review: 7

Following are our findings.

1. CALIBRATION: ION RATIOS AND RESPONSE FACTORS ARE OK
2. COLUMN SEPARATION IS ACCEPTABLE
3. SURROGATE RECOVERY IS OK
4. MATRIX SPIKE AND DUPLICATE ARE IN AGREEMENT
5. THE BLANK WAS CLEAN. DETECTION LIMITS ARE OK.
6. SAMPLES ARE REPORTED ON A DRY WEIGHT BASIS. SPOT CHECK ON CALCULATIONS WAS OK.

Patrick J. Churilla
5-12-87

(X) Data are acceptable for use.
( ) Data are acceptable for use with qualifications noted above.
( ) Data are preliminary - pending verification by Contractor Laboratory.
( ) Data are unacceptable.

cc: Dr. Alfred Haeberer/Joan Fisk/Gary Ward, EPA Support Services
Ross K. Robeson, EMSL-Las Vegas
Don Trees, CLP/Sample Management Office

A FORM 1320-4 (REV 3-78)

ADDITIONAL

DATA QUALIFIERS

Contractor: Triangle Laboratories | Case SAS 2882E

Below is a summary of the out of control audits and the possible effect on the data for this case:

Please note that all values should be considered the maximum concentration present in the sample due to possible interferences. In samples E-01 → E-04, no 2,3,7,8-TCDD was found in the full isomer screening and noted as ND (not detected) at the detection limit indicated. The 2,3,7,8-TCDD/TCDF confirmatory run does indicate a positive value but below the screening run DL's. These positive values range from 10 - 110 ppT.

In general, the most contaminated dioxin/furan sample was E-01 while E-05 had only minor OCDD + Hp/OCDD positives. E01 had .11 ppB 2,3,7,8-TCDD, 16 ppB 2,3,7,8-TCDF and total TCDD = 15 ppB and total TCDF = 71 ppB. Sample E-01 was described as "Grab of Polish Pits" and E-05 as "Composite of bagged slag".

Reviewed by: Kevin B Bolger / Patrick J Chuzilla

Phone: (312) 353-4676

Date: 5/22/87

TRIANGLE LABORATORIES, INC.
4915F PROSPECTUS DRIVE
RESEARCH TRIANGLE PARK, NC 27713
919 544-5729

RECEIVED
MAY 1 1987

CASE NARRATIVE

DATE : April 25, 1987
CLIENT NO.:SAS 2882E
TLI NO.: 8701364
OBJECTIVE: ANALYSIS OF SEDIMENT,SOIL AND SLAG SAMPLES FOR THE PRESENCE OF TETRA THROUGH OCTA CHLORO-DIBENZODIOXINS AND FURANS

The samples were extracted by the enclosed protocol. On the sample data sheets the concentration is given in parts per billion (ppb). "RT" is the retention time on the gas chromatographic column in minutes and seconds, "number" is the number of isomers in the totals reported for each group, and "ratio" is the ratio observed for the M to M+2 ions for the tetra through penta chlorinated compounds and M+2 to M+4 for the hexa through octa. "DL" is the detection limit in parts per billion. For quantitation, the sum of the areas for the two masses monitored is used. When no peak is detected an area of 2 counts for each ion (total 4 counts) is used to calculate the detection limit.

The samples were spiked with 10 ng of 13C-2378-TCDD, 37Cl-2378-TCDD, 13C-2378-TCDF, 13C-123478-HxCDF, 13C-12378-PCDD, 13C-123678-HxCDD, 13C-1234678-HpCDD, and 20 ng of 13C-OCDD prior to the extraction. For GC/MS analysis, the final extract was dissolved in 20 ul of toluene containing 13C-1234-TCDD and 13C-123789-HxCDD at a concentration of 500 pg/ul to measure the recovery of the 13C- labeled internal standards.

Samples were analyzed using a VG 7070H mass spectrometer and 11-250 data system, operated in the selected ion recording mode, at a resolution of 5000. A Varian 3700 GC was employed, with a DB-5 60m x 0.32mm id fused silica capillary column. One microliter of the 20 microliters final sample volume was injected, splitless, at a column temperature of 150 deg C, and heated ballistically to 190 deg C, then programmed at 3 deg/min to 300 deg C. A continuing calibration was demonstrated by injecting a solution of the analytes at a concentration of 100 pg/ul for the tetra isomers, 500 pg/ul for the penta though hepta and 1000 pg/ul for the octa, and a constant value of 100 pg/ul for 13C-2378-TCDD, 13C-2378-TCDF, 13C-123478-HxCDF, 37Cl-2378-TCDD, 13C-12378-PCDD, 13C-123678-HxCDD, 13C-1234678-HpCDD, and 200 pg/ul 13C-OCDD. Response factors (RF) were calculated for the analytes from this continuing calibration. For the totals in each group the response factor is taken as the average of the response factors for the individual isomers listed. Note that in some cases, when only one isomer is present, there may be a discrepancy between the amount given for the individual isomer and the total, since a different response factor may be used.

Delta RF on the data sheets for the continuing calibration is the difference between the daily response factor and the response factor in the initial calibration.

Positive identification criteria for chlorinated dibenzodioxins and furans are as follows:

1) Ratio of M+ to M+2 or M+2 to M+4 is within 20% of the theoretical value, except for the tetrachloro which are taken within 13%.

Acceptable ranges of the ratios for identification of chlorine containing compounds:

M/M+2
- tetra 0.67-0.87
- penta 0.49-0.73

M+2/M+4
- hexa 0.98-1.48
- hepta 0.82-1.24
- octa 0.70-1.06

2) Retention time for analytes is within 3 seconds of the corresponding 13C internal standard or surrogate standard.

3) The identification of specific isomers that do not have corresponding 13C12-labeled standards is done by comparison of the retention time of the analyte to the nearest internal standard retention time with reference to the comparable retention times found in the continuing calibration.

4) If the retention time and the ratio are correct for identification of an isomer, the signal to noise ratio (S:N) must be greater than 2.5.

5) For confirmation of 2378-TCDD and 2378-TCDF, the samples are run on a second GC column. 2378-TCDF is not fully resolved from other TCDF isomers on a DB-5 column, so the concentration obtained from the full screen analysis is considered a maximum. The concentration of 2378-TCDF obtained on the second column is sometimes less than that indicated by the full screen analysis, because of the increased GC resolution.

6) For confirmation of 2378-TCDD and 2378-TCDF, the GC resolution from its nearest isomer must be >25% valley. The ions for masses corresponding to M-COCl are also monitored for confirmations. The column performance check solution contains the following isomers: 2347, 2348, 2378, 2367, 3467, and 1368-TCDF, and 1478, 2378, 1237, 1238, and 1234-TCDD.

Sample E05 was used for the matrix spike and matrix spike duplicate. The matrix spike was 5 ng per sample of 2378-TCDD and 2378-TCDF, 25 ng for the penta though hepta chlorinated compounds, and 50 ng for the octa. Calculations for the percent recovery and precision are enclosed.

GC/MS Conditions
(FULL SCREEN ANALYSES)

Gas Chromatography:
Instrument: Varian 3700

Capillary Column:	a.	Manufacturer	- J&W Scientific
	b.	Liquid phase	- DB-5
	c.	Length	- 60 m
	d.	I.D	- 0.25 mm
	e.	film thickness	- 0.25 microns

Carrier gas : Helium
Head pressure : 28 psi
Flow thru column: 1 to 2 ml/min.
Injection type : Splitless for 30 sec.

Initial isothermal temperature :150 deg C for 30 sec.
Initial temperature program rate: to 190 deg C ballistically
Final temperature program rate : to 300 deg C @ 3 deg/min

GC/MS Conditions
(2378-TCDD CONFIRMATION)

Gas Chromatography:
Instument: Varian 3700

Column:	a.	Manufacturer	- 1) Supelco, inc 2) J&W Scientific
	b.	Liquid phase	- 1) SP-2331 2) DB-17
	c.	Length	- 1) 30 m 2) 15 m
	d.	I.D	- 0.32 mm
	e.	film thickness	- 0.25 microns

Carrier gas : Helium
Head pressure : 14 psi
Flow thru column: 1 to 2 ml/min.
Injection type : Splitless for 30 sec.

Initial isothermal temperature :150 deg C for 30 sec.
Initial temperature program rate: to 170 deg C ballistically
Final temperature program rate : to 220 deg C @ 4 deg/min

Mass Spectrometry:

Instrument: VG Micromass 7070H
Ionization mode : EI, positive ion
Reactant gas : N/A
Resolution : 5000
Scan mode : selected ion recording
Switching mode : voltage
Reference standard: PFK

EXTRACTION OF SOIL AND ASH SAMPLES

After addition of an equal amount of anhydrous sodium sulfate and internal standard and surigate spike solution, the samples are extracted with toluene in a Soxhlet apparatus for 16 hours. The extract is chromatographed as described below.

PURIFICATION

Column A1: Silica gel with basic and acidic layers. From the bottom, these layers are: 1 g activated silica gel, 2 g silica gel with 1N sodium hydroxide (2:1), 1 g silica gel, 12 g silica gel with concentrated sulfuric acid (3:2), 2 g silica gel and 1 cm of sodium sulfate on the top.

Column A2: Directly under column 1, a 2nd column of 6 g A-948 activated alumina with 10% water. The sample is loaded onto column 1 and the column is rinsed with 90 ml hexane. Column 1 is removed and the sample is eluted from column 2 with 20 ml of 1% methylene chloride in hexane (which is set aside for PCB analysis or in case of breakthrough) and 20 ml of 20% methylene chloride in hexane (dioxin fraction).

Column B: Mixed 124 g 545 celite and 10.7 g AX-21 carbon. Prewash with : 2 ml 50% benzene/ethyl acetate, 1 ml 50% methylene chloride/cyclohexane, and 2 ml hexane. After adding 1 ml of hexane, the dioxin fraction is loaded on the column, which is then rinsed with 2 ml 50 % methylene chloride/hexane followed by 2 ml of 50% benzene/ethyl acetate. The column is inverted, and the sample is eluted with 4 ml of toluene.

The sample is evaporated to dryness with nitrogen and redissolved in 20 ul of toluene containing the recovery standards 13C12-1234-TCDD and 13C12-123789-HxCDD, for GC/MS analysis.

EXAMPLE CALCULATIONS

SURROGATE, SAMPLE, OR INTERNAL STANDARD RESPONSE FACTORS:

$$\frac{(A_s)(I_{is})}{(A_{IS})(I_S)} = RF$$

2378-TCDD

WHERE:

A_s = AREA OF SAMPLE <FROM RIC> 2297+2887 = 5184

A_{is} = AREA OF APPROPRIATE INTERNAL STANDARD 2281+2764 = 5045
13C12-2378-TCDD

I_{is} = CONCENTRATION OF INTERNAL STANDARD 100 pg/µl

I_s = CONCENTRATION OF ANALYTE 100 pg/µl

EXAMPLE: FROM FILE M870770

$$\frac{[5184][100]}{[5045][100]} = RF = 1.028$$

The areas of the 2 masses monitored for the analyte are added and compared to the sum of the areas for the 2 masses monitored for the internal standard, except for the surrogate 37Cl-TCDD where only one mass is monitored (328), which is compared to the 334 ion in the internal standard, 13C12-2378-TCDD.

SURROGATE % RECOVERY:

1). Amount of surrogate found = $\frac{(A_s)(I_{is})}{(A_{is})(RF)}$ 13C 12-TCDF

WHERE:

A_s = AREA OF SAMPLE 4336 + 5387 = 9723

A_{IS} = AREA OF INTERNAL STANDARD 3101 + 3866 = 6967
13C12-2378-TCDD

I_{IS} = AMOUNT OF INTERNAL STANDARD IN TOTAL EXTRACT 10 ng

RF = RESPONSE FACTOR FOR SURROGATE
(from Continuing Calibration m870770) 1.337

EXAMPLE: 13C12-TCDF FILE: m870771

$$\frac{(9723)(10\,ng)}{(6967)(1.337)} = 10.44\ ng$$

2). % Recovery = $\frac{[\text{Found}]}{[\text{Spiked}]} \times 100$

% Recovery (for above sample) = $\frac{[10.44]}{[10]} \times 100 =$ 104 % Recove

CALCULATIONS OF INTERNAL STANDARD RECOVERY:

1). $$\frac{(A_{IS})(I_{RS})}{(A_{RS})(RF_{IS})} = \text{Amount of Internal Standard Found}$$

13C12-2378-TCDD

WHERE:

A_{IS} = AREA FOUND FOR INTERNAL STANDARD 3101 + 3866 = 6967

A_{RS} = AREA FOUND FOR RECOVERY STANDARD (13C-1234-TCDD) 2512 + 3208 = 5720

I_{RS} = AMOUNT OF RECOVERY STANDARD IN EXTRACT 500 pg/µl

RF_{IS} = RESPONSE FACTOR FOR INTERNAL STANDARD (from Continuing Calibration m870770) 1.356

13C12 2378-TCDD FILE: m870771

$$\frac{(6967)(500\ pg/\mu l)}{(5720)(1.356)} = 449.1\ pg/\mu l$$

2). $$\% \text{ Recovery} = \frac{(\text{Amount of Int. Std. Found})}{(\text{Amount of Int. Std. Added})} \times 100$$

$$\frac{(449)}{(500)} \times 100 = 89.8\ \%\ \text{Recovery}$$

CALCULATIONS OF DETECTION LIMITS:

$$DL: = \frac{(A_S)(I_{IS}) \times 2.5}{(A_{IS})(WT.)(RF)}$$

WHERE:

A_S = Area of sample channel integrated through the center of noise across a region corresponding to the baseline peak width o the standard compound (continuing calibration)

2378-TCDD 30.8+2* = 32.8

A_{IS} = AREA OF INTERNAL STANDARD
13C12-2378-TCDD 3101 + 3866 = 6967

I_{IS} = AMOUNT OF INTERNAL STANDARD 10ng

2.5 = FACTOR FOR SIGNAL/NOISE ACCEPTANCE
i.e.: s/n $\geq$ 2.5 for acceptance of signal as statistically significant.

RF = RESPONSE FACTOR FOR ANALYTE 1.028
(from Continuing Calibration M870770)

WT.= WEIGHT OF SAMPLE 10.0 g

EXAMPLE: FILE:

$$\frac{(32.8)(10ng)}{(6967)(10g)(1.028)} \times 2.5 = .01 \text{ ppb}$$

* 2 used for background (no peak)

CALCULATIONS OF SAMPLE RESULT:

$$\text{concentration} = \frac{[A_s][I_{is}]}{[A_{is}][RF_s][W]}$$

WHERE:

A_s = AREA FOUND FOR SAMPLE 2378-TCDF 4370+5926 = 10296

A_{is} = AREA FOUND FOR INTERNAL STANDARD 2277+2619 = 4896
13C-2378-TCDD

I_{is} = AMOUNT OF INT. STD. 10 ng

RF_s = RESPONSE FACTOR FOR ANALYTE 1.499
(from Continuing Calibration M870770)

W = MASS OF SAMPLE 7.03 g

EXAMPLE: 2378-TCDF FILE: M870773

$$\frac{(10296)(10\,ng)}{(4896)(1.499)(7.03\,g)} = 1.996\,ng/g = 1.996\,ppb$$

TLI#8701364
DATE 4-24-87

TRIANGLE LABORATORIES, INC.
MATRIX SPIKE
SAMPLE # SAS2882E05 MSD

ISOMER	NATIVE (ppb)	SPIKED (ppb)	OBSERVED (ppb)	%RECOVERY
2378-TCDD	ND(.001)	.49	.56	114%
123478-PCDD	ND(.002)	2.47	2.67	108%
123478-HxCDD	ND(.002)	2.47	2.15	87%
123679-HxCDD	ND(.002)	2.47	2.44	99%
123789-HxCDD	ND(.002)	2.47	2.36	96%
1234678-HpCDD	ND(.003)	2.47	2.70	109%
OCDD	.06	4.94	5.12	102%
2378-TCDF	ND(.001)	.49	.59	120%
12378-PCDF	ND(.001)	2.47	2.54	103%
234678-PCDF	ND(.001)	2.47	2.63	107%
123478-HXCDF	ND(.001)	2.47	2.33	94%
123678-HxCDF	ND(.001)	2.47	2.33	94%
234678-HXCDF	ND(.001)	2.47	2.35	95%
123789-HXCDF	ND(.001)	2.47	2.34	95%
1234678-HpCDF	.02	2.47	2.61	105%
1234789-HpCDF	ND(.011)	2.47	2.82	114%
OCDF	.05	4.94	5.28	106%

TLI#8701364
DATE 4-24-87

TRIANGLE LABORATORIES, INC.
MATRIX SPIKE
SAMPLE # SAS2882E05 MS

ISOMER	NATIVE (ppb)	SPIKED (ppb)	OBSERVED (ppb)	%RECOVERY
2378-TCDD	ND(.001)	.49	.53	107%
123478-PCDD	ND(.002)	2.43	2.48	102%
123478-HxCDD	ND(.002)	2.43	2.27	93%
123679-HxCDD	ND(.002)	2.43	2.49	102%
123789-HxCDD	ND(.002)	2.43	2.34	96%
1234678-HpCDD	ND(.003)	2.43	2.60	107%
OCDD	.06	4.87	4.91	100%
2378-TCDF	ND(.001)	.49	.55	113%
12378-PCDF	ND(.001)	2.43	2.45	101%
234678-PCDF	ND(.001)	2.43	2.43	100%
123478-HXCDF	ND(.001)	2.43	2.39	98%
123678-HxCDF	ND(.001)	2.43	2.46	101%
234678-HXCDF	ND(.001)	2.43	2.38	98%
123789-HXCDF	ND(.001)	2.43	2.37	97%
1234678-HpCDF	.02	2.43	2.53	103%
1234789-HpCDF	ND(.011)	2.43	2.73	112%
OCDF	.05	4.87	5.18	105%

TLI#8701364
DATE 4-24-87

TRIANGLE L' ORATORIES, INC.
MATRIX SPIKE
SAMPLE # SAS2882E05 MS AND SAS2882E05 MSD

ISOMER	MS(TOTAL) (ng)	MSD(TOTAL) (ng)	AVERAGE (ng)	DEVIATION ABS	DEVIATION %DEV
2378-TCDD	5.402	5.657	5.530	.128	2%
TOTAL TCDD	5.402	5.657	5.530	.128	2%
123478-PCDD	25.470	26.970	26.220	.750	3%
TOTAL PCDD	25.470	26.970	26.220	.750	3%
123478-HxCDD	23.282	21.768	22.525	-.757	-3%
123679-HxCDD	25.603	24.683	25.143	-.460	-2%
123789-HxCDD	24.011	23.883	23.947	-.064	-0%
TOTAL HxCDD	72.835	70.172	71.503	-1.331	-2%
1234678-HpCDD	26.661	27.324	26.992	.332	1%
TOTAL HpCDD	26.661	27.324	26.992	.332	1%
OCDD	50.374	51.814	51.094	.720	1%
2378-TCDF	5.679	5.930	5.805	.126	2%
TOTAL TCDF	5.679	5.930	5.805	.126	2%
12378-PCDF	25.172	25.745	25.459	.287	1%
234678-PCDF	24.915	26.646	25.780	.865	3%
TOTAL PCDF	50.138	52.209	51.174	1.035	2%
123478-HXCDF	24.556	23.539	24.047	-.508	-2%
123678-HxCDF	25.223	23.569	24.396	-.827	-3%
234678-HXCDF	24.443	23.802	24.122	-.320	-1%
123789-HXCDF	24.289	23.661	23.975	-.314	-1%
TOTAL HxCDF	98.592	94.531	96.561	-2.031	-2%
1234678-HpCDF	25.963	26.383	26.173	.210	1%
1234789-HpCDF	28.068	28.518	28.293	.225	1%
TOTAL HpCDF	53.887	54.749	54.318	.431	1%
OCDF	53.219	53.423	53.321	.102	0%

TRIANGLE LABORATORIES, INC.
PCDD/PCDF ANALYSIS

ANALYST	MAW	FILE #	M870771
DATE	4-23-87	CONCAL #	M870770
SAMPLE WEIGHT	10.00	TLI #	8701364
SAMPLE ID	SAS 2882 TLI BLANK		

NAME	CONC. (ppb)	NUMBER	DL	RATIO	RT
2378-TCDD	ND		0.011	15.400	26.36
TOTAL TCDD	ND	0	0.011	15.400	
12378-PCDD	ND		0.016	19.500	32.30
TOTAL PCDD	ND	0	0.016	19.500	
123478-HxCDD	ND		0.002	1.000	
123678-HxCDD	ND		0.002	1.000	
123789-HxCDD	ND		0.003	1.000	
TOTAL HxCDD	ND	0	0.025	1.241	
1234678-HpCDD	ND		0.003	1.000	
TOTAL HpCDD	ND	0	0.003	1.000	
OCDD	ND		0.021	0.100	49.58
2378-TCDF	ND		0.023	0.626	25.54
TOTAL TCDF	ND	0	0.023	0.626	
12378-PCDF	ND		0.001	1.000	
23478-PCDF	ND		0.040	0.850	32.08
TOTAL PCDF	ND	0	0.032	0.850	
123478-HxCDF	ND		0.001	1.000	
123678-HxCDF	ND		0.001	1.000	
234678-HxCDF	ND		0.001	1.000	
123789-HxCDF	ND		0.058	1.613	38.00
TOTAL HxCDF	ND	0	0.052	1.613	
1234678-HpCDF	ND		0.002	1.000	
1234789-HpCDF	ND		0.002	1.000	
TOTAL HpCDF	ND	0	0.002	1.000	
OCDF	ND		0.003	1.000	

SURROGATE RESULTS SUMMARY

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
13C12-TCDF	1.04	104.38	0.805	25.56
37Cl-TCDD	1.13	112.95		26.37
13C12-HxCDF	1.03	102.91	1.218	35.58

INTERNAL STANDARDS RECOVERY RESULTS

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
2378-13C12-TCDD	0.90	89.82	0.802	26.36
13C12-PCDD	0.88	88.33	0.643	32.30
13C12-HxCDD	0.95	95.42	1.296	37.09
13C12-HpCDD	0.83	83.16	1.105	43.51
13C12-OCDD	1.53	76.60	0.905	49.58

TRIANGLE LABORATORIES, INC.
PCDD/PCDF ANALYSIS

ANALYST	JAJ/MAW	FILE #	M870774
DATE	4-23-87	CONCAL #	M870770
SAMPLE WEIGHT	5.48	TL1 #	8701364
SAMPLE ID	SAS 2882 E01		

NAME	CONC. (ppb)	NUMBER	DL	RATIO	RT
2378-TCDD	ND		0.157	0.577	26.34
TOTAL TCDD	15.108	8		0.794	
12378-PCDD	0.382			0.539	32.32
TOTAL PCDD	12.905	11		0.603	
123478-HxCDD	0.511			1.301	37.03
123678-HxCDD	1.173			1.420	37.10
123789-HxCDD	1.812			1.177	37.37
TOTAL HxCDD	16.645	7		1.267	
1234678-HpCDD	10.743			1.050	43.50
TOTAL HpCDD	20.955	2		1.027	
OCDD	20.527			0.874	49.59
2378-TCDF	15.972			0.750	25.53
TOTAL TCDF	71.147	17		0.750	
12378-PCDF	2.103			0.659	31.13
23478-PCDF	6.193			0.617	32.06
TOTAL PCDF	48.197	14		0.629	
123478-HxCDF	10.676			1.231	35.57
123678-HxCDF	3.744			1.217	36.06
234678-HxCDF	8.475			1.236	36.52
123789-HxCDF	0.675			1.188	37.58
TOTAL HxCDF	48.344	12		1.228	
1234678-HpCDF	30.401			1.007	42.22
1234789-HpCDF	8.204			0.972	44.28
TOTAL HpCDF	58.975	4		1.002	
OCDF	64.718			0.896	50.12

SURROGATE RESULTS SUMMARY

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
13C12-TCDF	1.953	107.05	0.788	25.52
37Cl-TCDD	1.998	109.51		26.34
13C12-HxCDF *	2.238	122.63	0.761	35.58

INTERNAL STANDARDS RECOVERY RESULTS

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
2378-13C12-TCDD	1.613	88.42	0.847	26.33
13C12-PCDD	1.474	80.78	0.722	32.31
13C12-HxCDD	1.670	91.49	1.223	37.10
13C12-HpCDD	1.467	80.37	1.149	43.50
13C12-OCDD	2.626	71.95	0.848	49.59

* interference

TRIANGLE LABORATORIES, INC
2,3,7,8-TCDD/TCDF ANALYSIS

ANALYST	MDC	FILE #	M870797
DATE	4/25/87	CONCAL #	M870795
SAMPLE WEIGHT	5.48	TLI #	8701364
SAMPLE ID	SAS 2882E01		

NAME	CONC (ng/g)	DL	RATIO	RT
2378-TCDF	2.55		0.814	27.27
2378-TCDD	0.11		0.838	22.54

SURROGATE RESULTS SUMMARY

NAME	CONC (ng/g)	% RECOVERY	RATIO	RT
13C12-TCDF	2.11	115.42	0.804	27.24
37Cl-TCDD	2.27	124.54		22.55

INTERNAL STANDARDS RECOVERY RESULTS

NAME	CONC (ng/g)	% RECOVERY	RATIO	RT
2378-13C12-TCDD	2.36	129.11	0.810	22.53

TRIANGLE LABORATORIES, INC.
PCDD/PCDF ANALYSIS

ANALYST	MAW	FILE #	M870773
DATE	4-23-87	CONCAL #	M870770
SAMPLE WEIGHT	7.03	TLI #	8701364
SAMPLE ID	SAS 2882 E02		

NAME	CONC. (ppb)	NUMBER	DL	RATIO	RT
2378-TCDD	ND		0.034	1.526	26.33
TOTAL TCDD	0.632	2		0.750	
12378-PCDD	0.034			0.542	32.30
TOTAL PCDD	0.989	7		0.600	
123478-HxCDD	0.063			1.051	37.03
123678-HxCDD	0.152			1.000	37.11
123789-HxCDD	0.226			1.182	37.40
TOTAL HxCDD	1.993	6		1.220	
1234678-HpCDD	1.678			1.084	43.51
TOTAL HpCDD	3.269	2		1.056	
OCDD	5.053			0.873	50.00
2378-TCDF	1.996			0.737	25.53
TOTAL TCDF	8.874	13		0.738	
12378-PCDF	0.259			0.600	31.13
23478-PCDF	0.692			0.597	32.05
TOTAL PCDF	5.049	11		0.608	
123478-HxCDF	1.426			1.396	35.57
123678-HxCDF	0.538			1.429	36.07
234678-HxCDF	1.171			1.223	36.53
123789-HxCDF	0.083			1.296	38.00
TOTAL HxCDF	6.317	10		1.305	
1234678-HpCDF	4.378			1.018	42.23
1234789-HpCDF	1.077			0.995	44.29
TOTAL HpCDF	8.406	4		1.010	
OCDF	9.532			0.888	50.12

SURROGATE RESULTS SUMMARY

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
13C12-TCDF	1.399	98.32	0.807	25.51
37Cl-TCDD	1.379	96.97		26.33
13C12-HxCDF	1.426	100.24	1.219	35.58

INTERNAL STANDARDS RECOVERY RESULTS

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
2378-13C12-TCDD	1.307	91.90	0.869	26.32
13C12-PCDD	1.237	86.99	0.647	32.30
13C12-HxCDD	1.324	93.07	1.241	37.10
13C12-HpCDD	1.204	84.62	1.063	43.51
13C12-OCDD	2.250	79.10	0.878	49.59

TRIANGLE LABORATORIES, INC
2,3,7,8-TCDD/TCDF ANALYSIS

ANALYST	MDC	FILE #	M870798
DATE	4/25/87	CONCAL #	M870795
SAMPLE WEIGHT	7.03	TLI #	8701364
SAMPLE ID	SAS 3882E02		

NAME	CONC (ng/g)	DL	RATIO	RT
2378-TCDF	0.65		0.802	26.49
2378-TCDD	0.03		0.688	22.41

SURROGATE RESULTS SUMMARY

NAME	CONC (ng/g)	% RECOVERY	RATIO	RT
13C12-TCDF	1.45	101.72	0.799	26.47
37Cl-TCDD	1.55	109.19		22.41

INTERNAL STANDARDS RECOVERY RESULTS

NAME	CONC (ng/g)	% RECOVERY	RATIO	RT
2378-13C12-TCDD	1.42	100.00	0.813	22.39

TRIANGLE LABORATORIES, INC.
PCDD/PCDF ANALYSIS

ANALYST	JAJ	FILE #	M870775
DATE	4-23-87	CONCAL #	M870770
SAMPLE WEIGHT	8.93	TLI #	8701364
SAMPLE ID	SAS 2882 E03		

NAME	CONC. (ppb)	NUMBER	DL	RATIO	RT
2378-TCDD	ND		0.021	0.822	26.36
TOTAL TCDD	0.297	6		0.794	
12378-PCDD	0.036			0.543	32.35
TOTAL PCDD	0.435	7		0.586	
123478-HxCDD	0.029			1.195	37.07
123678-HxCDD	0.091			1.132	37.16
123789-HxCDD	0.140			1.322	37.43
TOTAL HxCDD	0.988	6		1.338	
1234678-HpCDD	1.080			1.024	43.55
TOTAL HpCDD	2.071	2		1.015	
OCDD	4.291			0.882	50.04
2378-TCDF	0.669			0.772	25.53
TOTAL TCDF	3.151	15		0.749	
12378-PCDF	0.091			0.551	31.10
23478-PCDF	0.250			0.635	32.08
TOTAL PCDF	2.184	13		0.601	
123478-HxCDF	0.601			1.274	36.01
123678-HxCDF	0.211			1.442	36.11
234678-HxCDF	0.468			1.280	36.55
123789-HxCDF	ND		0.001	1.000	
TOTAL HxCDF	2.560	10		1.268	
1234678-HpCDF	1.991			0.988	42.28
1234789-HpCDF	0.349			1.042	44.33
TOTAL HpCDF	3.411	4		0.989	
OCDF	3.281			0.892	50.16

SURROGATE RESULTS SUMMARY

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
13C12-TCDF	1.198	106.98	0.762	25.53
37Cl-TCDD	1.263	112.75		26.36
13C12-HxCDF	1.095	97.75	1.193	36.02

INTERNAL STANDARDS RECOVERY RESULTS

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
2378-13C12-TCDD	1.027	91.71	0.804	26.35
13C12-PCDD	0.947	84.53	0.606	32.34
13C12-HxCDD	1.040	92.89	1.250	37.14
13C12-HpCDD	1.090	97.31	1.042	43.54
13C12-OCDD	2.200	98.23	0.898	50.03

TRIANGLE LABORATORIES, INC
2,3,7,8-TCDD/TCDF ANALYSIS

ANALYST	MDC	FILE #	M870799
DATE	4/25/87	CONCAL #	M870795
SAMPLE WEIGHT	8.93	TLI #	8701364
SAMPLE ID	SAS 2882E03		

NAME	CONC (ng/g)	DL	RATIO	RT
2378-TCDF	0.17		0.770	26.54
2378-TCDD	0.01		0.688	22.45

SURROGATE RESULTS SUMMARY

NAME	CONC (ng/g)	% RECOVERY	RATIO	RT
13C12-TCDF	1.21	108.27	0.773	26.52
37Cl-TCDD	1.30	116.32		22.45

INTERNAL STANDARDS RECOVERY RESULTS

NAME	CONC (ng/g)	% RECOVERY	RATIO	RT
2378-13C12-TCDD	1.09	97.42	0.813	22.43

TRIANGLE LABORATORIES, INC.
PCDD/PCDF ANALYSIS

ANALYST	MAW	FILE #	M870776
DATE	4-23-87	CONCAL #	M870770
SAMPLE WEIGHT	8.26	TLI #	8701364
SAMPLE ID	SAS 2882 E04		

NAME	CONC. (ppb)	NUMBER	DL	RATIO
2378-TCDD	ND		0.089	1.100
TOTAL TCDD	1.650	7		0.791
12378-PCDD	0.088			0.550
TOTAL PCDD	2.000	11		0.598
123478-HxCDD	0.125			1.186
123678-HxCDD	0.280			1.309
123789-HxCDD	0.396			1.289
TOTAL HxCDD	3.362	7		1.257
1234678-HpCDD	2.773			1.004
TOTAL HpCDD	5.487	2		1.014
OCDD	9.262			0.891
2378-TCDF	4.396			0.740
TOTAL TCDF	20.790	14		0.752
12378-PCDF	0.467			0.654
23478-PCDF	1.560			0.663
TOTAL PCDF	11.472	14		0.626
123478-HxCDF	2.499			1.243
123678-HxCDF	0.960			1.241
234678-HxCDF	2.133			1.253
123789-HxCDF	0.153			1.288
TOTAL HxCDF	11.507	12		1.244
1234678-HpCDF	7.267			0.999
1234789-HpCDF	1.736			1.023
TOTAL HpCDF	14.385	4		1.000
OCDF	17.183			0.897

SURROGATE RESULTS SUMMARY

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
13C12-TCDF	1.303	107.67	0.790	25.51
37Cl-TCDD	1.344	110.99		26.33
13C12-HxCDF	1.284	106.05	1.113	35.58

INTERNAL STANDARDS RECOVERY RESULTS

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
2378-13C12-TCDD	1.140	94.14	0.773	26.33
13C12-PCDD	1.019	84.19	0.616	32.31
13C12-HxCDD	1.066	88.05	1.361	37.10
13C12-HpCDD	1.002	82.77	1.031	43.50
13C12-OCDD	1.978	81.71	0.905	49.58

TRIANGLE LABORATORIES, INC
2,3,7,8-TCDD/TCDF ANALYSIS

ANALYST	MDC	FILE #	M870800
DATE	4/25/87	CONCAL #	M870795
SAMPLE WEIGHT	8.26	TLI #	8701364
SAMPLE ID	SAS 2882E04		

NAME	CONC (ng/g)	DL	RATIO	RT
2378-TCDF	1.41		0.787	26.53
2378-TCDD	0.04		0.745	22.42

SURROGATE RESULTS SUMMARY

NAME	CONC (ng/g)	% RECOVERY	RATIO	RT
13C12-TCDF	1.30	107.28	0.796	26.51
37Cl-TCDD	1.39	114.56		22.44

INTERNAL STANDARDS RECOVERY RESULTS

NAME	CONC (ng/g)	% RECOVERY	RATIO	RT
2378-13C12-TCDD	1.23	101.69	0.809	22.41

TRIANGLE LABORATORIES, INC.
PCDD/PCDF ANALYSIS

ANALYST	MAW/JAJ	FILE #	M870777
DATE	4-23-87	CONCAL #	M870770
SAMPLE WEIGHT	10.32	TLI #	8701364
SAMPLE ID	SAS 2882 E05		

NAME	CONC. (ppb)	NUMBER	DL	RATIO	RT
2378-TCDD	ND		0.001	1.000	
TOTAL TCDD	ND	0	0.001	1.000	
12378-PCDD	ND		0.002	1.000	
TOTAL PCDD	ND	0	0.002	1.000	
123478-HxCDD	ND		0.002	1.000	
123678-HxCDD	ND		0.002	1.000	
123789-HxCDD	ND		0.002	1.000	
TOTAL HxCDD	ND	0	0.085	1.763	
1234678-HpCDD	ND		0.003	1.000	
TOTAL HpCDD	ND	0	0.003	1.000	
OCDD	0.058			0.835	50.02
2378-TCDF	ND		0.001	1.000	
TOTAL TCDF	ND	0	0.001	1.000	
12378-PCDF	ND		0.001	1.000	
23478-PCDF	ND		0.001	1.000	
TOTAL PCDF	ND	0	0.001	1.000	
123478-HxCDF	ND		0.001	1.000	
123678-HxCDF	ND		0.001	1.000	
234678-HxCDF	ND		0.001	1.000	
123789-HxCDF	ND		0.001	1.000	
TOTAL HxCDF	ND	0	0.001	1.000	
1234678-HpCDF	0.022			0.940	42.27
1234789-HpCDF	ND		0.012	0.093	44.33
TOTAL HpCDF	0.024	1		0.940	
OCDF	0.050			0.938	50.16

SURROGATE RESULTS SUMMARY

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
13C12-TCDF	1.049	108.18	0.755	25.55
37Cl-TCDD	1.077	111.09		26.37
13C12-HxCDF	0.933	96.21	1.197	36.02

INTERNAL STANDARDS RECOVERY RESULTS

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
2378-13C12-TCDD	1.037	106.98	0.835	26.36
13C12-PCDD	0.945	97.45	0.656	32.35
13C12-HxCDD	1.090	112.45	1.266	37.14
13C12-HpCDD	1.010	104.17	1.079	43.54
13C12-OCDD	1.858	95.83	0.920	50.02

TRIANGLE LABORATORIES, INC.
PCDD/PCDF ANALYSIS

ANALYST	MAW	FILE #	M870778
DATE	4-23-87	CONCAL #	M870770
SAMPLE WEIGHT	10.27	TLI #	8701364
SAMPLE ID	SAS 2882 E05 MS		

NAME	CONC. (ppb)	NUMBER	DL	RATIO	RT
2378-TCDD	0.526			0.758	26.36
TOTAL TCDD	0.526	1		0.758	
12378-PCDD	2.480			0.604	32.35
TOTAL PCDD	2.480	1		0.604	
123478-HxCDD	2.267			1.282	37.06
123678-HxCDD	2.493			1.253	37.13
123789-HxCDD	2.338			1.282	37.43
TOTAL HxCDD	7.092	3		1.272	
1234678-HpCDD	2.596			1.021	43.55
TOTAL HpCDD	2.596	1		1.021	
OCDD	4.905			0.870	50.03
2378-TCDF	0.553			0.750	25.55
TOTAL TCDF	0.553	1		0.750	
12378-PCDF	2.451			0.621	31.16
23478-PCDF	2.426			0.640	32.08
TOTAL PCDF	4.882	2		0.628	
123478-HxCDF	2.391			1.227	36.01
123678-HxCDF	2.456			1.239	36.11
234678-HxCDF	2.380			1.247	36.56
123789-HxCDF	2.365			1.222	38.03
TOTAL HxCDF	9.600	4		1.234	
1234678-HpCDF	2.528			0.973	42.27
1234789-HpCDF	2.733			1.045	44.33
TOTAL HpCDF	5.247	2		1.007	
OCDF	5.182			0.922	50.16

SURROGATE RESULTS SUMMARY

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
13C12-TCDF	1.073	110.15	0.806	25.55
37Cl-TCDD	1.085	111.45		26.37
13C12-HxCDF	0.969	99.44	1.311	36.01

INTERNAL STANDARDS RECOVERY RESULTS

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
2378-13C12-TCDD	0.904	92.80	0.821	26.36
13C12-PCDD	0.850	87.30	0.632	32.33
13C12-HxCDD	0.898	92.15	1.185	37.13
13C12-HpCDD	0.844	86.70	1.034	43.54
13C12-OCDD	1.575	80.85	0.878	50.02

TRIANGLE LABORATORIES, INC.
PCDD/PCDF ANALYSIS

ANALYST	MAW	FILE #	M870779
DATE	4-23-87	CONCAL #	M870770
SAMPLE WEIGHT	10.12	TLI #	8701364
SAMPLE ID	SAS 2882 E05 MSD		

NAME	CONC. (ppb)	NUMBER	DL	RATIO
2378-TCDD	0.559			0.780
TOTAL TCDD	0.559	1		0.780
12378-PCDD	2.665			0.609
TOTAL PCDD	2.665	1		0.609
123478-HxCDD	2.151			1.266
123678-HxCDD	2.439			1.302
123789-HxCDD	2.360			1.280
TOTAL HxCDD	6.934	3		1.283
1234678-HpCDD	2.700			1.005
TOTAL HpCDD	2.700	1		1.005
OCDD	5.120			0.856
2378-TCDF	0.586			0.736
TOTAL TCDF	0.586	1		0.736
12378-PCDF	2.544			0.616
23478-PCDF	2.633			0.601
TOTAL PCDF	5.159	2		0.610
123478-HxCDF	2.326			1.220
123678-HxCDF	2.329			1.249
234678-HxCDF	2.352			1.239
123789-HxCDF	2.338			1.207
TOTAL HxCDF	9.341	4		1.229
1234678-HpCDF	2.607			1.021
1234789-HpCDF	2.818			0.994
TOTAL HpCDF	5.410	2		1.008
OCDF	5.279			0.937

SURROGATE RESULTS SUMMARY

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
13C12-TCDF	1.134	114.73	0.788	25.55
37Cl-TCDD	1.179	119.29		26.37
13C12-HxCDF	0.940	95.18	1.244	36.01

INTERNAL STANDARDS RECOVERY RESULTS

NAME	CONC. (ppb)	% RECOVERY	RATIO	RT
2378-13C12-TCDD	0.892	90.24	0.789	26.36
13C12-PCDD	0.822	83.17	0.599	32.34
13C12-HxCDD	0.959	97.08	1.303	37.13
13C12-HpCDD	0.916	92.69	0.983	43.54
13C12-OCDD	1.743	88.22	0.913	50.01

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION V

ESD/Central Regional Laboratory
DATA TRACKING FORM FOR CONTRACT SAMPLES

CRL Data Set No. SF3916 CERCLIS No. _____

SMO Case No. SMS288RE Site Name and Location: Chemetco

Name of Contractor or EPA Laboratory: Triangle Data User: Fit

No. of Samples: 5 Date Samples or Data Received: 5/1/87

1. Have chain-of-custody records been received? YES ✓ NO ___
2. Have Traffic Reports or packing lists been received? YES ✓ NO ___
3. If no, are Traffic Report or packing list numbers written on the chain-of-custody record? YES ✓ NO ___
4. If no, which Traffic report or packing list numbers are missing?

Are basic data forms in? YES ✓ NO ___

Number of samples claimed: 5 Number of samples received: 5

Checked by: Mylia Feliciano Date: 5/1/87

Received by Contract Project Management Section: P. Chuvillo Date: 5/4/87

Review Started: 5-11-87 Reviewer Signature: Patrick J. Chuvillo

Total time spent on review: 7 HRS Date review completed: 5-12-87

Copied (xeroxed) by: [illegible] Date: _____

Mailed to Data User by: Mylia Feliciano Date: 5/13/87

DATA USERS:

Please fill in the blanks below and return this form to: Sylvia Griffin, Data Management Coordinator, Region V, 5SCRL

Data received by: _____ Date: _____

Q.A. review received by: _____ Date: _____

Inorganic Data Complete [ ], Suitable for Intended Purposes [ ] ✓ [ ] if acceptable.
Organic Data Complete [ ], Suitable for Intended Purposes [ ] List problems below.
Dioxin Data Complete [ ], Suitable for Intended Purposes [ ]
SAS Data Complete [ ], Suitable for Intended Purposes [ ]

See Attached "Missing Data Request Form" [ ]
PROBLEMS: Please indicate reasons (if any) why data are not suitable for your uses. Other problems.

Received by Data Management Coordinator, CRL for File: Date: _____

Signature: