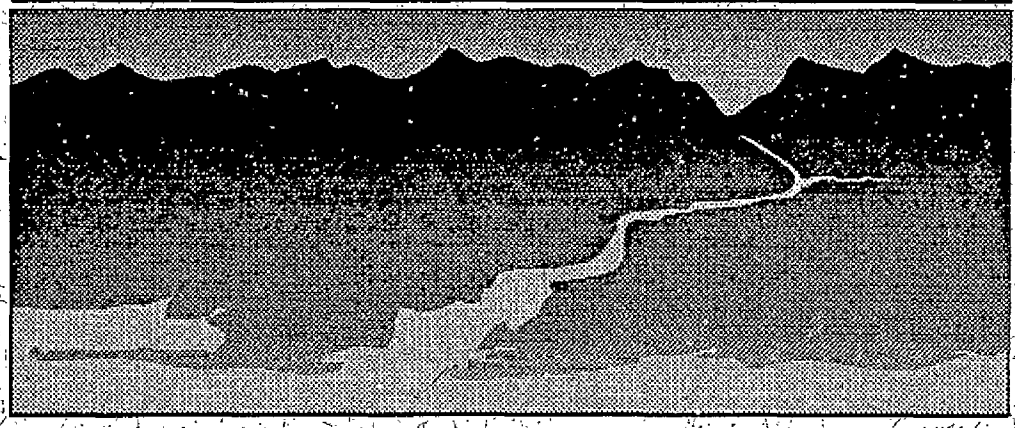
LOWER COLUMBIA RIVER



BI-STATE PROGRAM

RECONNAISSANCE SURVEY OF THE LOWER COLUMBIA RIVER

LABORATORY DATA REPORT
VOLUME 7: DIOXIN AND FURAN DATA FOR SEDIMENT

APRIL, 1992

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In Association With:

KEYSTONE/NEA

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VOLUME 7

DIOXIN/FURAN DATA - SEDIMENT

SECTION A. SAMPLES D28, D24, D26,
D30, D4, D10, D11, D45

SECTION B. SAMPLES D35, D38, D40, D40 DUP
D5, D8, D6, D6 DUP

SECTION C. SAMPLED D14, D15, D16, D16 MS,
D16 MSD, D18, D19, D20, D23

SECTION A. SAMPLES D28, D24, D26, D30, D4, D10, D11, D45

ANALYSIS OF SEDIMENTS

For The Presence of

PCDD's AND PCDF's
By
HIGH RESOLUTION GAS CHROMATOGRAPHY
HIGH RESOLUTION MASS SPECTROMETRY

KEYSTONE/NEA
ENVIRONMENTAL RESOURCES

CASE NARRATIVE

CASE NARRATIVE

I. SAMPLE DESCRIPTION

Four sand samples were received under Chain-of-Custody on October 1, 1991 and October 9, 1991. The samples were in good condition upon receipt, and were stored in a refrigerator maintained at 4°C until analysis. The samples were extracted on October 14, 1991, and analyzed on a DB-5 column on December 4, 1991. Confirmation analyses were on a DB-225 column on December 11, 1991.

Two laboratory method blanks and Two Precision and Recovery (PAR) samples were also analyzed with these sample sets.

II. ANALYSIS REQUEST

The analytical test requested for this sample set was as follows:

LAB ID NUMBER	ANALYSIS	DETECTION LIMIT
91TT01OC01	EPA Method 1613x	1 ppt (tetras)
91TT09OC01	EPA Method 1613x	1 ppt (tetras)

III. SAMPLE ANALYSIS SUMMARY

A. Background

Keystone/NEA's Center for Analytical Mass Spectrometry has analyzed this set of samples by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) according to EPA Method 1613x. Deviations from the promulgated Method 1613 are described below.

B. Analytical Methodology

The extraction, sample clean-up, and instrumental analyses were done by EPA Method 1613. All instrument calibration solutions (CS1 through CS5) were prepared and certified by an independent laboratory (Cambridge Isotope Labs), and conform to EPA Method 1613 levels. The spiking levels for Internal Standard, Recovery Standard, and native analytes are identical to those specified in EPA Method 1613.

Slight modifications have been made to EPA Method 1613 to improve efficiency and accuracy during the data validation steps, and to reduce the occurence of sample contamination with native 2378-TCDD. The modifications included here are consistent with procedures outlined in other EPA methods (Method 8280, Method 8290, Method 23, SAS CLP work, etc.), or have been suggested by NCASI (Method 90.01). The modifications are outlined below:

Clean-Up Recovery Standard Spiking Levels EPA Method 1613 calls for spiking the sample extracts with 800 pg of 37Cl-2378-TCDD immediately prior to the clean-up procedure. That level has been reduced to 200 pg, as suggested by NCASI Method 90.01. The purpose of this change is to reduce the occurence of native contamination in the 322 channel.

Standard Preparation and Spiking To prevent changes in concentration due to solvent losses, the standards for these analyses have been prepared in tetradecane. Internal Standards and PAR solutions are dissolved in acetone immediately prior to spiking an aqueous matrix.

ConCal Acceptance Criteria EPA Method 1613 lists separate and different acceptance criteria for each of the seventeen native analytes, for the fifteen Internal Standards, and for the Clean-Up Recovey Standard. Those acceptance criteria have been simplified by adopting EPA Method 8290 acceptance criteria of ±20% for the continuing calibration. The purpose of this change is to make the acceptance criteria for the continuing calibration the same as the acceptance criteria for the initial calibration.

Reporting Sample specific Estimated Detection Limits (EDLs), analyte concentrations below the LMCL, and Estimated Maximum Possible Concentrations (EMPCs) have been calculated and reported according to standard EPA methods. (Method 1613 does not specify how these values should be calculated and/or reported, but instead reports only the Lower Method Calibration Limits, LMCL.) In addition, analyte recoveries in the PAR samples are reported as the total amount of analyte recovered from the original sample, rather than as a concentration in the final extract.

C. Calculations and Reporting

Positive Identification. Where a peak has been positively identified as one of the 2378-substituted PCDD/PCDF isomers by passing all the QA criteria (retention times, analyte isotope ratios, and signal-to-noise), a concentration has been calculated in the usual manner and reported in

the attached tables. In cases where the reported concentration falls below the LMCL, it should be considered an estimate only.

Estimated Maximum Possible Concentration Where a peak has passed all the QA criteria except for the analyte isotope ratios, there may be co-eluting contaminants or other chemical interferences. In such cases, a concentration has been calculated in the usual manner, but reported as an Estimated Maximum Possible Concentration (EMPC).

Analyte Not Detected Where the Chromatogram is characterized by the absence of peaks in both native channels (at the appropriate retention times), or where a peak is present in one or both channels, but does not pass the signal-to-noise criteria of 2.5:1, the analyte cannot be positively identified and may be reported as Not Detected at or above the sample specific Estimated Detection Limit (ND/EDL). A data-review specialist has inspected each one individually and calculated an EDL based on the reporting requirements specified in EPA method 8290. Hard copies of the calculations are included in the sample data packet.

Calibration Limits A series of three Lower Method Calibration Limits (LMCLs) and three Upper Method Calibration Limits (UMCLs) have been calculated based on a sample size of 10 grams. The equations used are as follows:

(1) $$LMCL = \frac{(Lowest\ Instrument\ Calibration\ Pt) \times (Final\ Volume)}{(Sample\ Size)}$$

(2) $$UMCL = \frac{(Highest\ Instrument\ Calibration\ Pt) \times (Final\ Volume)}{(Sample\ Size)}$$

The Lowest and Highest Instrument Calibration Points (LICPs and HICPs) vary with each homologue group. For a 10 gram sample, the LMCL and UMCL are:

Homologue Group	LICP/HICP	LMCL	UMCL
Tetra	0.5/200 pg/µL	1 pg/g	400 pg/g
Penta, Hexa, Hepta	2.5/1,000 pg/µL	5 pg/g	2,000 pg/g
Octa	5.0/2,000 pg/µL	10 pg/g	4,000 pg/g

NOTE: pg/g = ppt

When the sample size is something other than 10 grams, the LMCL and UMCL values vary accordingly. For example, with a 20 gram sample, the LMCL for 2378-TCDD would be 0.5 ppt.

D. Results

General Sediment results are based on the initial weight of the sample (approximately 20g to 30g). All of the reported results are rounded to three significant figures. Laboratory Method Blank results are also based on a sample size of 20 g or 30g. Results for the PAR sample are on a per-sample basis; no correction has been made for sample size. Reported results for the 2378-TCDF are from a DB-225 column. All other results are from a DB-5 column.

Sediment Sample Results Most of the analyte concentrations were below or near the Lower Method Calibration Limit for the individual homologue groups. Sample specific EDLs and EMPCs have been calculated, but where they fall below the LMCL, they should be considered ESTIMATES ONLY. Samples D24 and D10 had quantifiable levels of 1234678-HpCDD, 188 ppt and 132 ppt respectively. Samples with concentrations of 2378-TCDF above the LMCL for a 20 gram sample were analyzed on a DB-225 column. Those results are flagged with an asterisk (*).

IV. QUALITY CONTROL

A. Project Quality Control

No special quality control measures were required or requested for this set of samples.

B. Instrument Quality Control

Conventional instrument quality control measures were applied for the analysis of these samples. The HRGC and HRMS systems' initial calibrations were verified immediately prior to and following analysis by injection of appropriate standards. One instrument blank was run prior to the laboratory Method Blank. All relevant instrument performance criteria were met. Documentation of initial and continuing calibrations, and GC and MS resolution checks can be found in the "QUALITY CONTROL DOCUMENTS" section of this report.

C. Laboratory Quality Control

Laboratory Method Blank One method blank was analyzed with each set of samples to test for laboratory contamination. Their treatment in the laboratory was identical in all respects to that of the actual samples. The data are included in the "QUALITY CONTROL DOCUMENTS" section of this report.

Both laboratory method blanks "91TT01OC01-MB" and "91TT09OC01-MB" were Non-Detect for all PCDD and PCDF isomers at the LMCL. For the 20 gram method blank the LMCLs would be 0.5 ppt (tetras), 2.5 ppt (pentas, hexas, heptas), and 5.0 ppt (octas). Many of the analytes, however, had sample specific EDL's significantly lower than the LMCL, ranging from 0.15 ppt to 0.75 ppt. A few analytes were present at levels significantly below the LMCL for their particular homologue group, and would not normally be reported under method 1613, but are included for your review.

Precision and Recovery Samples Table 4 in the "SAMPLE ANALYSIS SUMMARY" section of this report lists the levels (in pg) of analyte detected in the PAR samples. The detected levels are compared to the spiked levels, and a Percent Recovery is reported as well. The Percent Recovery for the various analytes is a measure of laboratory accuracy, and ranges from 34% to 112%. The Relative Percent Difference between the two PAR samples is also reported in Table 4. These values are a measure of laboratory precision and are all within 6%, except for 123789-HxCDD which has a value of -39 percent. The cause of this excessive deviation is currently under investigation.

D. Quality Control Review

All of the data has been reviewed by the scientist performing the analysis, by the Director of the Center for Analytical Mass Spectrometry, and the Quality Assurance Officer. All of the quality control and sample-specific information in the package is complete and meets or exceeds the minimum requirements for acceptability.

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Jeff Sprenger Date
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Keystone/NEA

KEYSTONE/NEA
ENVIRONMENTAL RESOURCES

SAMPLE ANALYSIS SUMMARY

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received: October 1 and October 9, 1991
Client name: TetraTech
Laboratory Project Number: 91TT01OC01 and 91TT09OC01
Customer Project Number:

MS File Number:	04DEC91LCB2011	04DEC91LCB2021	04DEC91LCB2031
Keystone/NEA Number:	91TT01OC01-MB	91TT01OC01-01	91TT01OC01-02
Customer Number:		D28	D24
Sample Description:	Method Blank	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	ND/EDL=0.17	EMPC=0.18	EMPC=0.26
12378-PeCDD	ND/EDL=0.28	EMPC=0.21	EMPC=3.38
123478-HxCDD	ND/EDL=0.30	0.65	1.37
123678-HxCDD	ND/EDL=0.25	1.61	5.29
123789-HxCDD	ND/EDL=0.27	1.13	2.52
1234678-HpCDD	EMPC=0.66	41.4	188
OCDD	3.76	369	1480
Furans			
2378-TCDF	EMPC=0.32	1.44*	3.23*
12378-PeCDF	ND/EDL=0.22	EMPC=0.26	1.14
23478-PeCDF	ND/EDL=0.20	0.32	0.83
123478-HxCDF	ND/EDL=0.62	EMPC=0.74	2.18
123678-HxCDF	ND/EDL=0.63	0.43	0.91
234678-HxCDF	ND/EDL=0.75	EMPC=0.44	0.65
123789-HxCDF	ND/EDL=0.74	ND/EDL=0.24	0.09
1234678-HpCDF	0.82	4.30	13.05
1234789-HpCDF	EMPC=0.33	0.37	1.14
OCDF	EMPC=1.52	9.84	36.56

Notes:
1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. Concentrations marked with an asterisk (*) are from a DB-225 column.

Table 1a

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 1 and October 9, 1991
Client name:	TetraTech
Laboratory Project Number:	91TT01OC01 and 91TT09OC01
Customer Project Number:	

MS File Number:	04DEC91LCB2041	04DEC91LCB2051
Keystone/NEA Number:	91TT01OC01-03	91TT01OC01-04
Customer Number:	D26	D30
Sample Description:	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)
Dioxins		
2378-TCDD	ND/EDL=0.10	0.12
12378-PeCDD	ND/EDL=0.12	0.09
123478-HxCDD	EMPC=0.10	EMPC=0.17
123678-HxCDD	0.61	0.82
123789-HxCDD	0.44	EMPC=0.57
1234678-HpCDD	6.38	23.03
OCDD	53.76	221
Furans		
2378-TCDF	0.67	1.72*
12378-PeCDF	EMPC=0.24	EMPC=0.19
23478-PeCDF	0.20	0.16
123478-HxCDF	0.70	0.37
123678-HxCDF	0.23	0.16
234678-HxCDF	EMPC=0.38	0.37
123789-HxCDF	EMPC=0.08	EMPC=0.10
1234678-HpCDF	1.67	2.37
1234789-HpCDF	0.35	EMPC=0.12
OCDF	3.58	6.89

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. Concentrations marked with an asterisk (*) are from a DB-225 column.

Table 1b

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 1 and October 9, 1991
Client name:	TetraTech
Laboratory Project Number:	91TT01OC01 and 91TT09OC01
Customer Project Number:	

MS File Number:	04DEC91LCB2071	04DEC91LCB2081	04DEC91LCB2091
Keystone/NEA Number:	91TT09OC01-MB	91TT09OC01-01	91TT09OC01-02
Customer Number:		D4	D10
Sample Description:	Method Blank	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	ND/EDL=0.08	0.23	EMPC=0.26
12378-PeCDD	ND/EDL=0.13	EMPC=0.22	0.52
123478-HxCDD	ND/EDL=0.22	0.51	1.92
123678-HxCDD	ND/EDL=0.19	1.91	5.95
123789-HxCDD	ND/EDL=0.21	1.58	5.04
1234678-HpCDD	1.84	26.2	132
OCDD	11.7	272	768
Furans			
2378-TCDF	EMPC=0.15	2.06*	2.09*
12378-PeCDF	ND/EDL=0.17	EMPC=0.30	EMPC=0.69
23478-PeCDF	ND/EDL=0.15	EMPC=0.30	EMPC=0.43
123478-HxCDF	0.35	EMPC=0.67	1.75
123678-HxCDF	EMPC=0.17	0.27	EMPC=1.41
234678-HxCDF	0.33	EMPC=0.66	1.40
123789-HxCDF	EMPC=0.04	EMPC=0.07	EMPC=0.08
1234678-HpCDF	0.97	4.65	14.8
1234789-HpCDF	EMPC=0.32	0.31	1.19
OCDF	2.55	15.1	34.6

Notes:
1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. Concentrations marked with an asterisk (*) are from a DB-225 column.

Table 1c

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 1 and October 9, 1991
Client name:	TetraTech
Laboratory Project Number:	91TT01OC01 and 91TT09OC01
Customer Project Number:	

MS File Number:	04DEC91LCB2101	04DEC91LCB2111
Keystone/NEA Number:	91TT09OC01-03	91TT09OC01-04
Customer Number:	D11	D45
Sample Description:	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)
Dioxins		
2378-TCDD	0.22	0.25
12378-PeCDD	0.12	0.16
123478-HxCDD	0.38	EMPC=0.40
123678-HxCDD	1.43	1.43
123789-HxCDD	1.19	0.94
1234678-HpCDD	23.8	27.1
OCDD	217	244
Furans		
2378-TCDF	1.93*	1.96*
12378-PeCDF	EMPC=0.36	EMPC=0.25
23478-PeCDF	0.24	EMPC=0.27
123478-HxCDF	EMPC=0.51	0.54
123678-HxCDF	EMPC=0.21	EMPC=0.28
234678-HxCDF	0.16	EMPC=0.30
123789-HxCDF	EMPC=1.87	ND/EDL=0.18
1234678-HpCDF	2.83	2.91
1234789-HpCDF	EMPC=0.31	0.25
OCDF	6.76	8.22

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. Concentrations marked with an asterisk (*) are from a DB-225 column.

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	October 1 and October 9, 1991
Client name:	TetraTech
Laboratory Project Number:	91TT01OC01 and 91TT09OC01
Customer Project Number:	

MS File Number:	04DEC91LCB2011	04DEC91LCB2021	04DEC91LCB2031
Keystone/NEA Number:	91TT01OC01-MB	91TT01OC01-01	91TT01OC01-02
Customer Number:		D28	D24
Sample Description:	Method Blank	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	ND/EDL=0.17	0.97	2.94
Total PeCDD	ND/EDL=0.28	0.48	2.24
Total HxCDD	ND/EDL=0.25	13.31	54.67
Total HpCDD	0.41	80.36	378
Furans			
Total TCDF	0.32	5.73	11.21
Total PeCDF	ND/EDL=0.20	2.17	7.66
Total HxCDF	ND/EDL=0.62	11.79	23.55
Total HpCDF	0.94	17.67	45.35

Note:
1. ND/EDL = Analyte Not Detected at or above sample specific Estimated Detection Limit.

Table 2a

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	October 1 and October 9, 1991
Client name:	TetraTech
Laboratory Project Number:	91TT01OC01 and 91TT09OC01
Customer Project Number:	

MS File Number:	04DEC91LCB2041	04DEC91LCB2051
Keystone/NEA Number:	91TT01OC01-03	91TT01OC01-04
Customer Number:	D26	D30
Sample Description:	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)
Dioxins		
Total TCDD	0.55	0.48
Total PeCDD	0.24	0.40
Total HxCDD	4.71	7.42
Total HpCDD	11.2	45.38
Furans		
Total TCDF	1.76	4.59
Total PeCDF	8.97	1.11
Total HxCDF	2.06	4.17
Total HpCDF	3.47	7.54

Note:
1. ND/EDL = Analyte Not Detected at or above sample specific Estimated Detection Limit.

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	October 1 and October 9, 1991
Client name:	TetraTech
Laboratory Project Number:	91TT01OC01 and 91TT09OC01
Customer Project Number:	

MS File Number:	04DEC91LCB2071	04DEC91LCB2081	04DEC91LCB2091
Keystone/NEA Number:	91TT09OC01-MB	91TT09OC01-01	91TT09OC01-02
Customer Number:		D4	D10
Sample Description:	Method Blank	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	ND/EDL=0.08	0.71	1.24
Total PeCDD	ND/EDL=0.13	0.12	2.60
Total HxCDD	ND/EDL=0.19	16.8	47.3
Total HpCDD	2.62	55.2	211
Furans			
Total TCDF	ND/EDL=0.15	6.79	7.72
Total PeCDF	ND/EDL=0.15	1.20	10.1
Total HxCDF	1.03	5.29	28.6
Total HpCDF	1.89	14.1	52.5

Note:
1. ND/EDL = Analyte Not Detected at or above sample specific Estimated Detection Limit.

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	October 1 and October 9, 1991
Client name:	TetraTech
Laboratory Project Number:	91TT01OC01 and 91TT09OC01
Customer Project Number:	

MS File Number:	04DEC91LCB2101	04DEC91LCB2111
Keystone/NEA Number:	91TT09OC01-03	91TT09OC01-04
Customer Number:	D11	D45
Sample Description:	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)
Dioxins		
Total TCDD	0.92	1.06
Total PeCDD	0.37	0.67
Total HxCDD	14.4	12.8
Total HpCDD	46.1	53.7
Furans		
Total TCDF	5.96	6.79
Total PeCDF	2.91	2.65
Total HxCDF	2.48	4.64
Total HpCDF	8.14	8.54

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received: October 1 and 9,1991
Client name: TetraTech
Laboratory Project Number: 91TT01OC01 and 91TT09OC01
Customer Project Number:

MS File Number:	04DEC91LCB2011	04DEC91LCB2021	04DEC91LCB2031
Keystone/NEA Number:	91TT01OC01-MB	91TT01OC01-01	91TT01OC01-02
Customer Number:		D28	D24
Sample Description:	Method Blank	Sediment	Sediment
Units	%	%	%
Dioxins			
13C-2378-TCDD	83	91	94
13C-12378-PeCDD	100	112	116
13C-123478-HxCDD	76	91	82
13C-123678-HxCDD	81	67	84
13C-1234678-HpCDD	97	96	110
13C-OCDD	74	86	116
Furans			
13C-2378-TCDF	83	83*	88*
13C-12378-PeCDF	75	82	80
13C-23478-PeCDF	81	88	86
13C-123478-HxCDF	72	72	74
13C-123678-HxCDF	66	63	65
13C-234678-HxCDF	65	62	54
13C-123789-HxCDF	84	85	95
13C-1234678-HpCDF	79	75	83
13C-1234789-HpCDF	90	94	104
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	85	99	78

Notes:
1. Recoveries marked with an asterisk (*) are from a DB-225 column.

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	October 1 and 9,1991
Client name:	TetraTech
Laboratory Project Number:	91TT01OC01 and 91TT09OC01
Customer Project Number:	

MS File Number:	04DEC91LCB2041	04DEC91LCB2051
Keystone/NEA Number:	91TT01OC01-03	91TT01OC01-04
Customer Number:	D26	D30
Sample Description:	Sediment	Sediment
Units	%	%
Dioxins		
13C-2378-TCDD	91	89
13C-12378-PeCDD	117	112
13C-123478-HxCDD	84	87
13C-123678-HxCDD	79	68
13C-1234789-HpCDD	106	97
13C-OCDD	97	86
Furans		
13C-2378-TCDF	91	81*
13C-12378-PeCDF	83	78
13C-23478-PeCDF	94	91
13C-123478-HxCDF	74	73
13C-123678-HxCDF	65	62
13C-234678-HxCDF	70	48
13C-123789-HxCDF	89	85
13C-1234678-HpCDF	82	78
13C-1234789-HpCDF	103	93
Clean-Up Recovery Standard		
37Cl4-2378-TCDD	94	96

Notes:
1. Recoveries marked with an asterisk (*) are from a DB-225 column.

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received: October 1 and 9,1991
Client name: TetraTech
Laboratory Project Number: 91TT01OC01 and 91TT09OC01
Customer Project Number:

MS File Number:	04DEC91LCB2071	04DEC91LCB2081	04DEC91LCB2091
Keystone/NEA Number:	91TT09OC01-MB	91TT09OC01-01	91TT09OC01-02
Customer Number:		D4	D10
Sample Description:	Method Blank	Sediment	Sediment
Units	%	%	%
Dioxins			
13C-2378-TCDD	84	74	88
13C-12378-PeCDD	102	88	112
13C-123478-HxCDD	82	77	99
13C-123678-HxCDD	75	47	52
13C-1234678-HpCDD	92	73	95
13C-OCDD	80	70	94
Furans			
13C-2378-TCDF	84	65*	76*
13C-12378-PeCDF	74	63	78
13C-23478-PeCDF	84	69	89
13C-123478-HxCDF	71	55	67
13C-123678-HxCDF	64	47	58
13C-234678-HxCDF	45	29	40
13C-123789-HxCDF	85	67	84
13C-1234678-HpCDF	78	57	74
13C-1234789-HpCDF	91	72	93
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	88	80	99

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	October 1 and 9,1991
Client name:	TetraTech
Laboratory Project Number:	91TT01OC01 and 91TT09OC01
Customer Project Number:	

MS File Number:	04DEC91LCB2101	04DEC91LCB2111
Keystone/NEA Number:	91TT09OC01-03	91TT09OC01-04
Customer Number:	D11	D45
Sample Description:	Sediment	Sediment
Units	%	%
Dioxins		
13C-2378-TCDD	86	84
13C-12378-PeCDD	107	102
13C-123478-HxCDD	97	82
13C-123678-HxCDD	51	62
13C-1234789-HpCDD	94	90
13C-OCDD	93	87
Furans		
13C-2378-TCDF	78*	76*
13C-12378-PeCDF	75	72
13C-23478-PeCDF	82	79
13C-123478-HxCDF	67	63
13C-123678-HxCDF	56	56
13C-234678-HxCDF	47	39
13C-123789-HxCDF	81	78
13C-1234678-HpCDF	73	69
13C-1234789-HpCDF	90	87
Clean-Up Recovery Standard		
37Cl4-2378-TCDD	92	88

SUMMARY OF ANALYTICAL RESULTS
Precision and Recovery Samples

Date received: 1-Oct-91
Client name: Tetra Tech
Laboratory Project Number: 91TT01-09OC01
Customer Project Number:

MS File Number:		04DEC91LCB2061			04DEC91LCB2121	
Keystone/NEA Number:		91TT01OC01-PAR			91TT09OC01-PAR	
Sample Description:	Spiked Levels	Measured Levels	Percent Recy	Measured Levels	Percent Recy	RPD
Units	pg	pg	%	pg	%	%
Dioxins						
2378-TCDD	200	206	103	211	106	-3
12378-PeCDD	1079	845	78	843	78	0
123478-HxCDD	904	1053	117	1003	111	5
123678-HxCDD	888	987	111	1023	115	-4
123789-HxCDD	783	738	94	1091	139	-39
1234678-HpCDD	1012	975	96	1007	99	-3
OCDD	1909	2065	108	2117	111	-2
Furans						
2378-TCDF	188	191	101	183	98	4
12378-PeCDF	931	1141	123	1074	115	6
23478-PeCDF	880	1049	119	1065	121	-1
123478-HxCDF	950	1086	114	1057	111	3
123678-HxCDF	934	1050	112	1072	115	-2
234678-HxCDF	904	1055	117	1029	114	2
123789-HxCDF	960	973	101	969	101	0
1234678-HpCDF	897	1099	122	1047	117	5
1234789-HpCDF	948	1025	108	1039	110	-1
OCDF	1842	2064	112	1956	106	5

SECTION B. SAMPLES D35, D38, D40, D40 DUP
D5, D8, D6, D6 DUP

ANALYSIS OF SEDIMENT

For The Presence of

PCDD's AND PCDF's
By
HIGH RESOLUTION GAS CHROMATOGRAPHY
HIGH RESOLUTION MASS SPECTROMETRY

KEYSTONE/NEA
ENVIRONMENTAL RESOURCES

CASE NARRATIVE

CASE NARRATIVE

I. SAMPLE DESCRIPTION

A total of six sediment samples were received under Chain-of-Custody on October 1, 1991 and October 9, 1991. The samples were in good condition upon receipt, and were stored in a refrigerator maintained at 4°C until analysis. The samples were extracted in two sets, one on October 14, 1991, and the second on October 17, 1991. They were analyzed on a DB-5 column on December 13, 1991. Confirmation analyses were on a DB-225 column on December 12, 1991.

One laboratory method blank and one Precision and Recovery (PAR) sample were also analyzed with each of these sample sets. One sample from each set was extracted in duplicate as a measure of laboratory precision.

II. ANALYSIS REQUEST

The analytical test requested for this sample set was as follows:

LAB ID NUMBER	ANALYSIS	DETECTION LIMIT
91TT27SP01	EPA Method 1613x	1 ppt (tetras)
91TT15OC01	EPA Method 1613x	1 ppt (tetras)

III. SAMPLE ANALYSIS SUMMARY

A. Background

Keystone/NEA's Center for Analytical Mass Spectrometry has analyzed this set of samples by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) according to EPA Method 1613x. Deviations from the promulgated Method 1613 are described below.

B. Analytical Methodology

The extraction, sample clean-up, and instrumental analyses were done by EPA Method 1613. All instrument calibration solutions (CS1 through CS5) were prepared and certified by an independent laboratory (Cambridge Isotope Labs), and conform to EPA Method 1613 levels. The

spiking levels for Internal Standard, Recovery Standard, and native analytes are identical to those specified in EPA Method 1613.

Slight modifications have been made to EPA Method 1613 to improve efficiency and accuracy during the data validation steps, and to reduce the occurence of sample contamination with native 2378-TCDD. The modifications included here are consistent with procedures outlined in other EPA methods (Method 8280, Method 8290, Method 23, SAS CLP work, etc.), or have been suggested by NCASI (Method 90.01). The modifications are outlined below:

Clean-Up Recovery Standard Spiking Levels EPA Method 1613 calls for spiking the sample extracts with 800 pg of 37Cl-2378-TCDD immediately prior to the clean-up procedure. That level has been reduced to 200 pg, as suggested by NCASI Method 90.01. The purpose of this change is to reduce the occurence of native contamination in the 322 channel.

Standard Preparation and Spiking To prevent changes in concentration due to solvent losses, the standards for these analyses have been prepared in tetradecane. Internal Standards and PAR solutions are dissolved in acetone immediately prior to spiking an aqueous matrix.

ConCal Acceptance Criteria EPA Method 1613 lists separate and different acceptance criteria for each of the seventeen native analytes, for the fifteen Internal Standards, and for the Clean-Up Recovery Standard. Those acceptance criteria have been simplified by adopting EPA Method 8290 acceptance criteria of ±20% for the continuing calibration. The purpose of this change is to make the acceptance criteria for the continuing calibration the same as the acceptance criteria for the initial calibration.

Reporting Sample specific Estimated Detection Limits (EDLs), analyte concentrations below the LMCL, and Estimated Maximum Possible Concentrations (EMPCs) have been calculated and reported according to standard EPA methods. (Method 1613 does not specify how these values should be calculated and/or reported, but instead reports only the Lower Method Calibration Limits, LMCL.) In addition, analyte recoveries in the PAR samples are reported as the total amount of analyte recovered from the original sample, rather than as a concentration in the final extract.

C. Calculations and Reporting

Positive Identification Where a peak has been positively identified as one of the 2378-substituted PCDD/PCDF isomers by passing all the QA criteria (retention times, analyte isotope

ratios, and signal-to-noise), a concentration has been calculated in the usual manner and reported in the attached tables. In cases where the reported concentration falls below the LMCL, it should be considered an estimate only.

Estimated Maximum Possible Concentration Where a peak has passed all the QA criteria except for the analyte isotope ratios, there may be co-eluting contaminants or other chemical interferences. In such cases, a concentration has been calculated in the usual manner, but reported as an Estimated Maximum Possible Concentration (EMPC).

Analyte Not Detected Where the Chromatogram is characterized by the absence of peaks in both native channels (at the appropriate retention times), or where a peak is present in one or both channels, but does not pass the signal-to-noise criteria of 2.5:1, the analyte cannot be positively identified and may be reported as Not Detected at or above the sample specific Estimated Detection Limit (ND/EDL). A data-review specialist has inspected each one individually and calculated an EDL based on the reporting requirements specified in EPA method 8290. Hard copies of the calculations are included in the sample data packet.

Calibration Limits A series of three Lower Method Calibration Limits (LMCLs) and three Upper Method Calibration Limits (UMCLs) have been calculated based on a sample size of 10 grams. The equations used are as follows:

(1) $$LMCL = \frac{(\text{Lowest Instrument Calibration Pt}) \times (\text{Final Volume})}{(\text{Sample Size})}$$

(2) $$UMCL = \frac{(\text{Highest Instrument Calibration Pt}) \times (\text{Final Volume})}{(\text{Sample Size})}$$

The Lowest and Highest Instrument Calibration Points (LICPs and HICPs) vary with each homologue group. For a 10 gram sample, the LMCL and UMCL are:

Homologue Group	LICP/HICP	LMCL	UMCL
Tetra	0.5/200 pg/µL	1.0 pg/g	400 pg/g
Penta, Hexa, Hepta	2.5/1,000 pg/µL	5.0 pg/g	2,000 pg/g
Octa	5.0/2,000 pg/µL	10.0 pg/g	4,000 pg/g

NOTE: pg/g = ppt

When the sample size is something other than 10 grams, the LMCL and UMCL values vary accordingly. For example, for a 20 gram sample, the LMCL for 2378-TCDD would be 0.5 ppt.

D. Results

General Sediment results are based on the initial weight of the sample (approximately 20 to 30 grams). All of the reported results are rounded to three significant figures. Laboratory Method Blank results are also based on a sample size of 20 grams. Results for the PAR samples are on a per-sample basis; no correction has been made for sample size. Reported results for the 2378-TCDF are from a DB-225 column. All other results are from a DB-5 column.

Sediment Sample Results None of the six sediment samples contained any of the 2378-substituted isomers at concentrations exceeding the calibration range of the instrument. Many analytes were detected at or below the Lower Method Calibration Limit, and those concentrations should be considered estimates only. Otherwise, these samples posed no significant analytical difficulty, and contain the seventeen 2378-substituted isomers at concentrations well within the analytical range for this method. (See Tables 1a - 1d.)

IV. QUALITY CONTROL

A. Project Quality Control

No special quality control measures were required or requested for this set of samples.

B. Instrument Quality Control

Conventional instrument quality control measures were applied for the analysis of these samples. The HRGC and HRMS systems' initial calibrations were verified immediately prior to and following analysis by injection of appropriate standards. One instrument blank was run prior to the laboratory Method Blank. All relevant instrument performance criteria were met. Documentation of initial and continuing calibrations, and GC and MS resolution checks can be found in the "QUALITY CONTROL DOCUMENTS" section of this report.

C. Laboratory Quality Control

Laboratory Method Blank One method blank was analyzed with each set of samples to test for laboratory contamination. Their treatment in the laboratory was identical in all respects to that of

the actual samples. The data are included in the "QUALITY CONTROL DOCUMENTS" section of this report.

With one exception, both laboratory method blanks "91TT27SP01-MB" and "91TT15OC01-MB" were Non-Detect for all PCDD and PCDF isomers at the LMCL for a 20 gram sample of 0.5 ppt (tetras), 2.5 ppt (pentas, hexas, heptas), and 5.0 ppt (octas). Method blank 91TT15OC01-MB contained 9.69 ppt OCDD. This is approximately twice the LMCL for a 20 gram sample.

Many of the analytes had sample specific EDL's significantly lower than the LMCL, ranging from 0.07 ppt to 0.22 ppt. A few analytes were present at levels significantly below the LMCL for their particular homologue group, and would not normally be reported under method 1613, but are included for your review.

Precision and Recovery Samples Table 4 in the "SAMPLE ANALYSIS SUMMARY" section of this report lists the levels (in pg) of analyte detected in the two PAR samples. The detected levels are compared to the spiked levels, and a Percent Recovery is reported as well. The Percent Recovery for the various analytes is a measure of laboratory accuracy, and ranges from 84% to 134%. The Relative Percent Difference between the two PAR samples is also reported in Table 4. These values are a measure of laboratory precision and are all within 17%, except for 123789-HxCDD which has a value of -25 percent.

Duplicate Sample Results Two of the six samples were extracted in duplicate as a measure of laboratory precision. Results of the duplicate analyses are included in Tables 1b and 1d.

D. Quality Control Review

All of the data has been reviewed by the scientist performing the analysis, by the Director of the Center for Analytical Mass Spectrometry, and the Quality Assurance Officer. All of the quality control and sample-specific information in the package is complete and meets or exceeds the minimum requirements for acceptability.

Laura Chambers 12/20/91
Laura Chambers Date
Sr. Scientist
Center for Analytical Mass Spectrometry

Peggy L. Meek 12/18/91
Peggy L. Meek Date
Wet lab Supervisor
Center for Analytical Mass Spectrometry

12.27.91.
William H. Chambers Date
Director
Center for Analytical Mass Spectrometry

J. Sprenger 12/27/91
Jeff Sprenger Date
QA Officer
Keystone/NEA

KEYSTONE/NEA
ENVIRONMENTAL RESOURCES

SAMPLE ANALYSIS SUMMARY

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received: September 27 and October 15, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT27SP01 and 91TT15OC01
Customer Project Number:

MS File Number:	13DEC91LCB2031	13DEC91LCB2041	13DEC91LCB2051
Keystone/NEA Number:	91TT27SP01-MB	91TT27SP01-01	91TT27SP01-02
Customer Number:		D35	D38
Sample Description:	Method Blank	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	ND/EDL=0.13	0.28	ND/EDL=0.09
12378-PeCDD	ND/EDL=0.13	ND/EDL=0.13	ND/EDL=0.10
123478-HxCDD	ND/EDL=0.17	0.40	ND/EDL=0.17
123678-HxCDD	ND/EDL=0.14	1.39	EMPC=0.14
123789-HxCDD	ND/EDL=0.19	1.00	0.10
1234678-HpCDD	0.71	20.0	0.90
OCDD	4.54	193	6.76
Furans			
2378-TCDF	ND/EDL=0.08	2.94*	0.06*
12378-PeCDF	ND/EDL=0.13	1.14	ND/EDL=0.07
23478-PeCDF	ND/EDL=0.12	0.18	ND/EDL=0.07
123478-HxCDF	EMPC=0.40	2.99	0.31
123678-HxCDF	0.14	0.94	EMPC=0.11
234678-HxCDF	EMPC=0.38	1.02	EMPC=0.24
123789-HxCDF	EMPC=0.10	0.22	ND/EDL=0.10
1234678-HpCDF	0.71	6.46	0.51
1234789-HpCDF	EMPC=0.25	1.76	0.15
OCDF	1.13	16.9	1.19

Notes:
1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. Concentrations marked with an asterisk (*) are from a DB-225 column.

Table 1a

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	September 27 and October 15, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT27SP01 and 91TT15OC01
Customer Project Number:	

MS File Number:	13DEC91LCB2061	13DEC91LCB2071
Keystone/NEA Number:	91TT27SP01-03	91TT27SP01-03d
Customer Number:	D40	D40
Sample Description:	Sediment	Sediment (Duplicate)
Units	pg/g (ppt)	pg/g (ppt)
Dioxins		
2378-TCDD	EMPC=0.21	0.17
12378-PeCDD	0.18	EMPC=0.13
123478-HxCDD	EMPC=0.27	EMPC=0.20
123678-HxCDD	0.59	0.42
123789-HxCDD	0.84	EMPC=0.59
1234678-HpCDD	9.25	6.41
OCDD	71.5	64.6
Furans		
2378-TCDF	0.98*	0.65*
12378-PeCDF	0.94	0.32
23478-PeCDF	0.69	EMPC=0.28
123478-HxCDF	2.78	0.76
123678-HxCDF	1.06	0.3
234678-HxCDF	1.25	0.53
123789-HxCDF	EMPC=0.15	0.22
1234678-HpCDF	6.38	2.08
1234789-HpCDF	1.61	0.50
OCDF	12.5	5.14

Notes:
1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. Concentrations marked with an asterisk (*) are from a DB-225 column.

Table 1b

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	September 27 and October 15, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT27SP01 and 91TT15OC01
Customer Project Number:	

MS File Number:	13DEC91LCB2081	13DEC91LCB2091	13DEC91LCB2101
Keystone/NEA Number:	91TT15OC01-MB	91TT15OC01-01	91TT15OC01-02
Customer Number:		D5	D8
Sample Description:	Method Blank	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	ND/EDL=0.07	0.12	0.16
12378-PeCDD	ND/EDL=0.08	EMPC=0.17	EMPC=0.14
123478-HxCDD	ND/EDL=0.15	0.15	0.19
123678-HxCDD	ND/EDL=0.13	EMPC=0.78	0.59
123789-HxCDD	ND/EDL=0.14	0.58	0.37
1234678-HpCDD	1.22	12.6	5.93
OCDD	9.69	159	45.9
Furans			
2378-TCDF	0.23	1.23*	0.96*
12378-PeCDF	ND/EDL-0.08	0.79	0.24
23478-PeCDF	ND/EDL-0.08	0.54	EMPC=0.16
123478-HxCDF	ND/EDL=0.14	1.69	0.42
123678-HxCDF	ND/EDL=0.13	0.63	0.14
234678-HxCDF	EMPC=0.28	EMPC=0.86	0.43
123789-HxCDF	ND/EDL=0.16	EMPC=0.10	ND/EDL=0.19
1234678-HpCDF	0.42	4.50	1.52
1234789-HpCDF	ND/EDL=0.22	1.14	0.25
OCDF	EMPC=1.03	14.9	EMPC=4.48

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. Concentrations marked with an asterisk (*) are from a DB-225 column.

Table 1c

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	September 27 and October 15, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT27SP01 and 91TT15OC01
Customer Project Number:	

MS File Number:	13DEC91LCB2111	13DEC91LCB2121
Keystone/NEA Number:	91TT15OC01-03	91TT15OC01-03d
Customer Number:	D6	D6
Sample Description:	Sediment	Sediment (Duplicate)
Units	pg/g (ppt)	pg/g (ppt)
Dioxins		
2378-TCDD	0.15	0.17
12378-PeCDD	0.16	EMPC=0.19
123478-HxCDD	EMPC=0.17	0.19
123678-HxCDD	1.14	1.98
123789-HxCDD	0.74	EMPC=1.04
1234678-HpCDD	8.75	10.1
OCDD	64.6	57.9
Furans		
2378-TCDF	1.25*	1.33*
12378-PeCDF	EMPC=0.24	0.50
23478-PeCDF	0.20	EMPC=0.25
123478-HxCDF	0.37	2.09
123678-HxCDF	0.17	0.50
234678-HxCDF	0.30	EMPC=0.54
123789-HxCDF	ND/EDL=0.21	ND/EDL=0.20
1234678-HpCDF	2.24	4.31
1234789-HpCDF	ND/EDL=0.42	0.66
OCDF	4.64	6.27

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. Concentrations marked with an asterisk (*) are from a DB-225 column.

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received: September 27 and October 15, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT27SP01 and 91TT15OC01
Customer Project Number:

MS File Number:	13DEC91LCB2031	13DEC91LCB2041	13DEC91LCB2051
Keystone/NEA Number:	91TT27SP01-MB	91TT27SP01-01	91TT27SP01-02
Customer Number:		D35	D38
Sample Description:	Method Blank	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	0.37	2.50	0.87
Total PeCDD	ND/EDL=0.13	ND/EDL=0.13	ND/EDL=0.10
Total HxCDD	ND/EDL=0.14	18.7	1.46
Total HpCDD	1.11	67.2	1.68
Furans			
Total TCDF	0.31	9.62	0.18
Total PeCDF	ND/EDL=0.12	2.34	ND/EDL=0.07
Total HxCDF	0.17	19.2	0.65
Total HpCDF	1.21	27.3	1.05

Note:
1. ND/EDL = Analyte Not Detected at or above sample specific Estimated Detection Limit.

Table 2a

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	September 27 and October 15, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT27SP01 and 91TT15OC01
Customer Project Number:	

MS File Number:	13DEC91LCB2061	13DEC91LCB2071
Keystone/NEA Number:	91TT27SP01-03	91TT27SP01-03d
Customer Number:	D40	D40
Sample Description:	Sediment	Sediment (Duplicate)
Units	pg/g (ppt)	pg/g (ppt)
Dioxins		
Total TCDD	0.43	1.10
Total PeCDD	1.33	0.45
Total HxCDD	12.4	5.74
Total HpCDD	27.3	19.4
Furans		
Total TCDF	7.38	4.43
Total PeCDF	5.73	1.22
Total HxCDF	13.4	3.64
Total HpCDF	17.4	4.66

Note:
1. ND/EDL = Analyte Not Detected at or above sample specific Estimated Detection Limit.

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	September 27 and October 15, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT27SP01 and 91TT15OC01
Customer Project Number:	

MS File Number:	13DEC91LCB2081	13DEC91LCB2091	13DEC91LCB2101
Keystone/NEA Number:	91TT15OC01-MB	91TT15OC01-01	91TT15OC01-02
Customer Number:		D5	D8
Sample Description:	Method Blank	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	0.44	0.33	0.76
Total PeCDD	ND/EDL=0.08	0.68	0.20
Total HxCDD	ND/EDL=0.13	3.93	2.36
Total HpCDD	2.02	23.1	12.2
Furans			
Total TCDF	0.23	2.89	2.55
Total PeCDF	ND/EDL=0.08	3.61	1.40
Total HxCDF	ND/EDL=0.13	6.81	2.28
Total HpCDF	0.52	9.99	4.55

Note:
1. ND/EDL = Analyte Not Detected at or above sample specific Estimated Detection Limit.

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	September 27 and October 15, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT27SP01 and 91TT15OC01
Customer Project Number:	

MS File Number:	13DEC91LCB2111	13DEC91LCB2121
Keystone/NEA Number:	91TT15OC01-03	91TT15OC01-03d
Customer Number:	D6	D6
Sample Description:	Sediment	Sediment (Duplicate)
Units	pg/g (ppt)	pg/g (ppt)
Dioxins		
Total TCDD	0.79	0.76
Total PeCDD	0.10	0.65
Total HxCDD	7.62	12.0
Total HpCDD	18.2	20.7
Furans		
Total TCDF	4.5	5.24
Total PeCDF	1.21	1.54
Total HxCDF	3.08	6.51
Total HpCDF	6.91	11.3

Note:
1. ND/EDL = Analyte Not Detected at or above sample specific Estimated Detection Limit.

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	September 27 and October 15, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT27SP01 and 91TT15OC01
Customer Project Number:	

MS File Number:	13DEC91LCB2031	13DEC91LCB2041	13DEC91LCB2051
Keystone/NEA Number:	91TT27SP01-MB	91TT27SP01-01	91TT27SP01-02
Customer Number:		D35	D38
Sample Description:	Method Blank	Sediment	Sediment
Units	%	%	%
Dioxins			
13C-2378-TCDD	82	88	76
13C-12378-PeCDD	103	115	105
13C-123478-HxCDD	74	78	65
13C-123678-HxCDD	76	80	71
13C-1234678-HpCDD	106	102	82
13C-OCDD	80	88	54
Furans			
13C-2378-TCDF	96	82*	67*
13C-12378-PeCDF	87	97	85
13C-23478-PeCDF	95	60	90
13C-123478-HxCDF	76	75	68
13C-123678-HxCDF	70	70	62
13C-234678-HxCDF	52	54	49
13C-123789-HxCDF	88	92	81
13C-1234678-HpCDF	88	84	71
13C-1234789-HpCDF	101	100	82
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	87	95	82

Notes:
1. Recoveries highlighted with an asterisk (*) are reported from the DB-225 column.

SUMMARY OF ANALYTICAL RESULTS

Internal Standard Recoveries

Date received: September 27 and October 15, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT27SP01 and 91TT15OC01
Customer Project Number:

MS File Number:	13DEC91LCB2061	13DEC91LCB2071	13DEC91LCB2141
Keystone/NEA Number:	91TT27SP01-03	91TT27SP01-03d	91TT27SP01-PAR
Customer Number:	D40	D40	
Sample Description:	Sediment	Sediment (Duplicate)	PAR Sample
Units	%	%	%
Dioxins			
13C-2378-TCDD	82	84	74
13C-12378-PeCDD	113	114	97
13C-123478-HxCDD	77	78	64
13C-123678-HxCDD	72	73	77
13C-1234789-HpCDD	92	88	80
13C-OCDD	65	63	60
Furans			
13C-2378-TCDF	75*	73*	87
13C-12378-PeCDF	90	90	79
13C-23478-PeCDF	97	96	84
13C-123478-HxCDF	78	77	70
13C-123678-HxCDF	68	68	65
13C-234678-HxCDF	59	49	40
13C-123789-HxCDF	90	89	81
13C-1234678-HpCDF	80	76	71
13C-1234789-HpCDF	91	85	76
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	93	94	90

Notes:
1. Recoveries highlighted with an asterisk (*) are reported from the DB-225 column.

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	September 27 and October 15, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT27SP01 and 91TT15OC01
Customer Project Number:	

MS File Number:	13DEC91LCB2081	13DEC91LCB2091	13DEC91LCB2101
Keystone/NEA Number:	91TT15OC01-MB	91TT15OC01-01	91TT15OC01-02
Customer Number:		D5	D8
Sample Description:	Method Blank	Sediment	Sediment
Units	%	%	%
Dioxins			
13C-2378-TCDD	73	98	86
13C-12378-PeCDD	116	119	115
13C-123478-HxCDD	73	80	77
13C-123678-HxCDD	87	88	80
13C-1234678-HpCDD	83	87	78
13C-OCDD	76	83	76
Furans			
13C-2378-TCDF	101	86*	78*
13C-12378-PeCDF	95	98	92
13C-23478-PeCDF	101	103	99
13C-123478-HxCDF	78	87	81
13C-123678-HxCDF	75	80	72
13C-234678-HxCDF	50	58	44
13C-123789-HxCDF	95	97	93
13C-1234678-HpCDF	77	87	75
13C-1234789-HpCDF	84	89	82
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	67	103	92

Notes:
1. Recoveries highlighted with an asterisk (*) are reported from the DB-225 column.

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	September 27 and October 15, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT27SP01 and 91TT15OC01
Customer Project Number:	

MS File Number:	13DEC91LCB2111	13DEC91LCB2121	13DEC91LCB2151
Keystone/NEA Number:	91TT15OC01-03	91TT15OC01-03d	91TT15OC01-PAR
Customer Number:	D6	D6	
Sample Description:	Sediment	Sediment (Duplicate)	PAR Sample
Units	%	%	%
Dioxins			
13C-2378-TCDD	88	94	89
13C-12378-PeCDD	114	120	111
13C-123478-HxCDD	72	74	82
13C-123678-HxCDD	85	67	78
13C-1234789-HpCDD	80	82	78
13C-OCDD	80	77	71
Furans			
13C-2378-TCDF	79*	85*	102
13C-12378-PeCDF	92	96	94
13C-23478-PeCDF	100	106	99
13C-123478-HxCDF	78	81	82
13C-123678-HxCDF	73	75	75
13C-234678-HxCDF	54	62	52
13C-123789-HxCDF	92	96	91
13C-1234678-HpCDF	78	79	77
13C-1234789-HpCDF	81	83	81
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	93	101	101

Notes:
1. Recoveries highlighted with an asterisk (*) are reported from the DB-225 column.

SUMMARY OF ANALYTICAL RESULTS
Precision and Recovery Samples

Date received: September 27 and October 15, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT27SP01 and 91TT15OC01
Customer Project Number:
Invoice Number:

MS File Number:	13DEC91LCB2141			13DEC91LCB2151		
Keystone/NEA Number:	91TT27SP01-PAR			91TT15OC01-PAR		
Sample Description:	Spiked Levels	Measured Levels	Percent Recy	Measured Levels	Percent Recy	RPD
Units	pg	pg	%	pg	%	%
Dioxins						
2378-TCDD	200	238	119	222	111	7
12378-PeCDD	1079	995	92	902	84	10
123478-HxCDD	904	1208	134	1015	112	17
123678-HxCDD	888	1101	124	1087	122	1
123789-HxCDD	783	805	103	1030	132	-25
1234678-HpCDD	1012	1084	107	990	98	9
OCDD	1909	2248	118	2062	108	9
Furans						
2378-TCDF	188	202	107	190	101	6
12378-PeCDF	931	1226	132	1111	119	10
23478-PeCDF	880	1164	132	1028	117	12
123478-HxCDF	950	1128	119	1000	105	12
123678-HxCDF	934	1137	122	1066	114	6
234678-HxCDF	904	1089	120	993	110	9
123789-HxCDF	960	1026	107	935	97	9
1234678-HpCDF	897	1172	131	1099	123	6
1234789-HpCDF	948	1180	124	1071	113	10
OCDF	1842	2352	128	2182	118	7

SECTION C. SAMPLED D14, D15, D16, D16 MS, D16 MSD, D18, D19, D20, D23

ANALYSIS OF SEDIMENT

For The Presence of

PCDD's AND PCDF's
By
HIGH RESOLUTION GAS CHROMATOGRAPHY
HIGH RESOLUTION MASS SPECTROMETRY

KEYSTONE/NEA
ENVIRONMENTAL RESOURCES

CASE NARRATIVE

CASE NARRATIVE

I. SAMPLE DESCRIPTION

Seven sediment samples were received under Chain-of-Custody on October 8, 1991. The samples were in good condition upon receipt, and were stored in a refrigerator maintained at 4°C until analysis. The samples were extracted in two sets, one on October 15, 1991, and the second on October 17, 1991. They were analyzed on a DB-5 column on December 16, 1991. Confirmation analyses were on a DB-225 column on December 12, 1991.

Two laboratory method blanks and one Precision and Recovery (PAR) sample were also analyzed with each of these sample sets. One sample, D16, was extracted with a matrix spike and a matrix spike duplicate as a measure of laboratory precision and accuracy.

II. ANALYSIS REQUEST

The analytical test requested for this sample set was as follows:

LAB ID NUMBER	ANALYSIS	DETECTION LIMIT
91TT08OC01	EPA Method 1613x	1 ppt (tetras)

III. SAMPLE ANALYSIS SUMMARY

A. Background

Keystone/NEA's Center for Analytical Mass Spectrometry has analyzed this set of samples by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) according to EPA Method 1613x. Deviations from the promulgated Method 1613 are described below.

B. Analytical Methodology

The extraction, sample clean-up, and instrumental analyses were done by EPA Method 1613. All instrument calibration solutions (CS1 through CS5) were prepared and certified by an independent laboratory (Cambridge Isotope Labs), and conform to EPA Method 1613 levels. The

spiking levels for Internal Standard, Recovery Standard, and native analytes are identical to those specified in EPA Method 1613.

Slight modifications have been made to EPA Method 1613 to improve efficiency and accuracy during the data validation steps, and to reduce the occurence of sample contamination with native 2378-TCDD. The modifications included here are consistent with procedures outlined in other EPA methods (Method 8280, Method 8290, Method 23, SAS CLP work, etc.), or have been suggested by NCASI (Method 90.01). The modifications are outlined below:

Clean-Up Recovery Standard Spiking Levels EPA Method 1613 calls for spiking the sample extracts with 800 pg of 37Cl-2378-TCDD immediately prior to the clean-up procedure. That level has been reduced to 200 pg, as suggested by NCASI Method 90.01. The purpose of this change is to reduce the occurence of native contamination in the 322 channel.

Standard Preparation and Spiking To prevent changes in concentration due to solvent losses, the standards for these analyses have been prepared in tetradecane. Internal Standards and PAR solutions are dissolved in acetone immediately prior to spiking an aqueous matrix.

ConCal Acceptance Criteria EPA Method 1613 lists separate and different acceptance criteria for each of the seventeen native analytes, for the fifteen Internal Standards, and for the Clean-Up Recovery Standard. Those acceptance criteria have been simplified by adopting EPA Method 8290 acceptance criteria of ±20% for the continuing calibration. The purpose of this change is to make the acceptance criteria for the continuing calibration the same as the acceptance criteria for the initial calibration.

Reporting Sample specific Estimated Detection Limits (EDLs), analyte concentrations below the LMCL, and Estimated Maximum Possible Concentrations (EMPCs) have been calculated and reported according to standard EPA methods. (Method 1613 does not specify how these values should be calculated and/or reported, but instead reports only the Lower Method Calibration Limits, LMCL.) In addition, analyte recoveries in the PAR samples are reported as the total amount of analyte recovered from the original sample, rather than as a concentration in the final extract.

C. Calculations and Reporting

Positive Identification Where a peak has been positively identified as one of the 2378-substituted PCDD/PCDF isomers by passing all the QA criteria (retention times, analyte isotope

ratios, and signal-to-noise), a concentration has been calculated in the usual manner and reported in the attached tables. In cases where the reported concentration falls below the LMCL, it should be considered an estimate only.

Estimated Maximum Possible Concentration Where a peak has passed all the QA criteria except for the analyte isotope ratios, there may be co-eluting contaminants or other chemical interferences. In such cases, a concentration has been calculated in the usual manner, but reported as an Estimated Maximum Possible Concentration (EMPC).

Analyte Not Detected Where the Chromatogram is characterized by the absence of peaks in both native channels (at the appropriate retention times), or where a peak is present in one or both channels, but does not pass the signal-to-noise criteria of 2.5:1, the analyte cannot be positively identified and may be reported as Not Detected at or above the sample specific Estimated Detection Limit (ND/EDL). A data-review specialist has inspected each one individually and calculated an EDL based on the reporting requirements specified in EPA method 8290. Hard copies of the calculations are included in the sample data packet.

Calibration Limits A series of three Lower Method Calibration Limits (LMCLs) and three Upper Method Calibration Limits (UMCLs) have been calculated based on a sample size of 10 grams. The equations used are as follows:

(1) $$LMCL = \frac{(Lowest\ Instrument\ Calibration\ Pt) \times (Final\ Volume)}{(Sample\ Size)}$$

(2) $$UMCL = \frac{(Highest\ Instrument\ Calibration\ Pt) \times (Final\ Volume)}{(Sample\ Size)}$$

The Lowest and Highest Instrument Calibration Points (LICPs and HICPs) vary with each homologue group. For a 10 gram sample, the LMCL and UMCL are:

Homologue Group	LICP/HICP	LMCL	UMCL
Tetra	0.5/200 pg/µL	1.0 pg/g	400 pg/g
Penta, Hexa, Hepta	2.5/1,000 pg/µL	5.0 pg/g	2,000 pg/g
Octa	5.0/2,000 pg/µL	10.0 pg/g	4,000 pg/g

NOTE: pg/g = ppt

When the sample size is something other than 10 grams, the LMCL and UMCL values vary accordingly. For example, for a 20 gram sample, the LMCL for 2378-TCDD would be 0.5 ppt.

D. Results

General Sediment results are based on the initial weight of the sample (approximately 20 to 30 grams). All of the reported results are rounded to three significant figures. Laboratory Method Blank results are also based on a sample size of 20 grams. Results for the PAR samples are on a per-sample basis; no correction has been made for sample size. Reported results for the 2378-TCDF are from a DB-225 column. All other results are from a DB-5 column.

Sediment Sample Results None of the seven sediment samples contained any of the 2378-substituted isomers at concentrations exceeding the calibration range of the instrument. Many analytes were detected at or below the Lower Method Calibration Limit, and those concentrations should be considered estimates only. Otherwise, these samples posed no significant analytical difficulty, and contain the seventeen 2378-substituted isomers at concentrations well within the analytical range for this method. (See Tables 1a - 1d.)

IV. QUALITY CONTROL

A. Project Quality Control

Project quality control for this set of samples included duplicate matrix spikes of one of the seven samples, D16.

B. Instrument Quality Control

Conventional instrument quality control measures were applied for the analysis of these samples. The HRGC and HRMS systems' initial calibrations were verified immediately prior to and following analysis by injection of appropriate standards. One instrument blank was run prior to the laboratory Method Blanks. All relevant instrument performance criteria were met. Documentation of initial and continuing calibrations, and GC and MS resolution checks can be found in the "QUALITY CONTROL DOCUMENTS" section of this report.

C. Laboratory Quality Control

Laboratory Method Blanks One method blank was analyzed with each set of samples to test for laboratory contamination. Their treatment in the laboratory was identical in all respects to that of

the actual samples. The data are included in the "QUALITY CONTROL DOCUMENTS" section of this report.

With one exception, both laboratory method blanks "91TT08OC01-MB1" and "91TT08OC01-MB2" were Non-Detect for all PCDD and PCDF isomers at the LMCL of 0.5 ppt (tetras), 2.5 ppt (pentas, hexas, heptas), and 5.0 ppt (octas). Both method blanks contained OCDD above the LMCL. Method blank #1 contained a level of OCDD very near the LMCL, 5.75 ppt. This is not an unusual concentration for this analyte. Method blank #2 contained 29.1 ppt OCDD. This is approximately 6 times the LMCL for that analyte, and should be considered when reviewing the data.

Many of the analytes had sample specific EDL's significantly lower than the LMCL, ranging from 0.10 ppt to 0.94 ppt. A few analytes were present at levels significantly below the LMCL for their particular homologue group, and would not normally be reported under method 1613, but are included for your review.

Precision and Recovery Samples Table 4 in the "SAMPLE ANALYSIS SUMMARY" section of this report lists the levels (in pg) of analyte detected in the PAR samples. The detected levels are compared to the spiked levels, and a Percent Recovery is reported as well. The Percent Recovery for the various analytes is a measure of laboratory accuracy, and ranges from 93% to 136%.

Matrix Spike Sample Results The results of the matrix spike and matrix spike duplicate are in Tables 5a and 5b. One analyte, 123789-HxCDD had percent deviations of 68% and 76% in the two matrix spike samples. However, the duplicate analyses had excellent reproducibility, with a relative percent difference of only 8%.

D. Quality Control Review

All of the data has been reviewed by the scientist performing the analysis, by the Director of the Center for Analytical Mass Spectrometry, and the Quality Assurance Officer. All of the quality control and sample-specific information in the package is complete and meets or exceeds the minimum requirements for acceptability.

Laura Chambers 12/30/91
Laura Chambers Date
Sr. Scientist
Center for Analytical Mass Spectrometry

Peggy Meek 12/30/91
Peggy L. Meek Date
Wet lab Supervisor
Center for Analytical Mass Spectrometry

Patrick Buddress 12/31/91
Patrick Buddress Date
Associate Director
Center for Analytical Mass Spectrometry

J. Sprenger 12/31/91
Jeff Sprenger Date
QA Officer
Keystone/NEA

KEYSTONE/NEA
ENVIRONMENTAL RESOURCES

SAMPLE ANALYSIS SUMMARY

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 8, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT08OC01
Customer Project Number:	

MS File Number:	16DEC91LCB3071	16DEC91LCB3081	16DEC91LCB3091
Keystone/NEA Number:	91TT08OC01-MB2	91TT08OC01-01	91TT08OC01-02
Customer Number:		D14	D15
Sample Description:	Method Blank	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	ND/EDL=0.72	0.19	0.17
12378-PeCDD	ND/EDL=0.38	0.23	0.16
123478-HxCDD	EMPC=0.10	EMPC=0.40	EMPC=0.26
123678-HxCDD	0.27	1.21	0.99
123789-HxCDD	0.25	1.00	0.83
1234678-HpCDD	2.66	12.7	12.1
OCDD	29.1	103	105
Furans			
2378-TCDF	0.05	1.17*	1.34*
12378-PeCDF	ND/EDL=0.94	0.27	0.29
23478-PeCDF	ND/EDL=0.89	0.24	0.23
123478-HxCDF	0.18	0.61	0.73
123678-HxCDF	0.10	EMPC=0.23	0.31
234678-HxCDF	EMPC=0.28	0.36	0.43
123789-HxCDF	ND/EDL=0.12	EMPC=0.14	0.18
1234678-HpCDF	0.84	2.75	3.12
1234789-HpCDF	0.23	EMPC=0.25	EMPC=0.45
OCDF	3.07	7.86	9.45

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. Concentrations marked with an asterisk (*) are from a DB-225 column.

Table 1a

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 8, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT08OC01
Customer Project Number:	

MS File Number:	13DEC91LCB3101	13DEC91LCB3111	13DEC91LCB3121
Keystone/NEA Number:	91TT08OC01-03	91TT08OC01-03MS	91TT08OC01-03MSd
Customer Number:	D16	D16	D16
Sample Description:	Sediment	Sediment plus Matrix Spike	Sediment Matrix Spike Dup
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	0.35	7.32	6.79
12378-PeCDD	0.23	28.3	27.3
123478-HxCDD	0.74	34.4	31.9
123678-HxCDD	1.67	35.3	31.3
123789-HxCDD	1.59	44.3	40.8
1234678-HpCDD	28.8	53.8	49.5
OCDD	303	294	255
Furans			
2378-TCDF	2.87*	9.31*	8.25*
12378-PeCDF	0.57	34.8	32.4
23478-PeCDF	0.49	33.4	30.4
123478-HxCDF	1.14	35.4	29.6
123678-HxCDF	EMPC=0.37	33.4	34.5
234678-HxCDF	0.61	32.3	29.6
123789-HxCDF	EMPC=0.27	30.1	27.9
1234678-HpCDF	5.14	38.4	32.5
1234789-HpCDF	0.75	32.7	30.5
OCDF	8.61	77.0	62.0

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. Concentrations marked with an asterisk (*) are from a DB-225 column.

Table 1b

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 8, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT08OC01
Customer Project Number:	

MS File Number:	16DEC91LCB3011	16DEC91LCB3021	16DEC91LCB3031
Keystone/NEA Number:	91TT08OC01-MB1	91TT08OC01-04	91TT08OC01-05
Customer Number:		D18	D19
Sample Description:	Method Blank	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	ND/EDL=0.05	0.13	EMPC=0.07
12378-PeCDD	ND/EDL=0.10	0.20	ND/EDL=0.08
123478-HxCDD	ND/EDL=0.11	0.49	0.15
123678-HxCDD	EMPC=0.17	1.93	0.44
123789-HxCDD	0.15	2.39	0.20
1234678-HpCDD	0.07	27.3	16.5
OCDD	5.75	219	129
Furans			
2378-TCDF	0.13	1.30*	0.82*
12378-PeCDF	0.14	1.37	EMPC=0.31
23478-PeCDF	EMPC=0.13	1.46	0.28
123478-HxCDF	0.40	7.47	0.60
123678-HxCDF	EMPC=0.16	2.22	EMPC=0.27
234678-HxCDF	EMPC=0.36	6.21	0.30
123789-HxCDF	EMPC=0.15	EMPC=7.21	EMPC=0.07
1234678-HpCDF	0.92	27.8	2.06
1234789-HpCDF	0.28	15.5	0.31
OCDF	2.43	128	6.15

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. Concentrations marked with an asterisk (*) are from a DB-225 column.

Table 1c

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 8, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT08OC01
Customer Project Number:	

MS File Number:	16DEC91LCB3041	16DEC91LCB3051
Keystone/NEA Number:	91TT08OC01-06	91TT08OC01-07
Customer Number:	D20	D23
Sample Description:	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)
Dioxins		
2378-TCDD	0.24	0.19
12378-PeCDD	0.12	0.13
123478-HxCDD	0.31	EMPC=0.15
123678-HxCDD	1.48	1.02
123789-HxCDD	EMPC=0.89	0.58
1234678-HpCDD	54.3	15.4
OCDD	566	139
Furans		
2378-TCDF	2.07*	1.92*
12378-PeCDF	EMPC=0.17	0.19
23478-PeCDF	0.28	0.21
123478-HxCDF	0.61	0.43
123678-HxCDF	0.25	0.18
234678-HxCDF	EMPC=0.55	0.47
123789-HxCDF	EMPC=0.16	EMPC=0.15
1234678-HpCDF	3.42	2.45
1234789-HpCDF	EMPC=0.37	0.28
OCDF	12.5	6.30

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. Concentrations marked with an asterisk (*) are from a DB-225 column.

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received: October 8, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT08OC01
Customer Project Number:

MS File Number:	16DEC91LCB3071	16DEC91LCB3081	16DEC91LCB3091
Keystone/NEA Number:	91TT08OC01-MB2	91TT08OC01-01	91TT08OC01-02
Customer Number:		D14	D15
Sample Description:	Method Blank	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	0.12	1.60	1.48
Total PeCDD	ND/EDL=0.38	1.01	1.01
Total HxCDD	1.08	8.95	10.8
Total HpCDD	4.59	24.3	24.3
Furans			
Total TCDF	0.12	8.53	7.66
Total PeCDF	ND/EDL=0.89	3.71	7.84
Total HxCDF	7.33	5.20	5.02
Total HpCDF	2.44	7.95	8.25

Note:
1. ND/EDL = Analyte Not Detected at or above sample specific Estimated Detection Limit.

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received: October 8, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT08OC01
Customer Project Number:

MS File Number:	13DEC91LCB2101	13DEC91LCB2111	13DEC91LCB2121
Keystone/NEA Number:	91TT08OC01-03	91TT08OC01-03MS	91TT08OC01-03MSd
Customer Number:	D16	D16	D16
Sample Description:	Sediment	Sediment plus Matrix Spike	Sediment Matrix Spike Dup
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	2.49	8.97	8.36
Total PeCDD	1.64	29.20	27.9
Total HxCDD	18.9	136	131
Total HpCDD	60.5	79.3	73.2
Furans			
Total TCDF	13.9	17.9	16.7
Total PeCDF	7.07	69.7	65.5
Total HxCDF	8.16	139	121
Total HpCDF	17.0	114	71.8

Note:
1. ND/EDL = Analyte Not Detected at or above sample specific Estimated Detection Limit.

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received: October 8, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT08OC01
Customer Project Number:

MS File Number:	16DEC91LCB3011	16DEC91LCB3021	16DEC91LCB3031
Keystone/NEA Number:	91TT08OC01-MB1	91TT08OC01-04	91TT08OC01-05
Customer Number:		D18	D19
Sample Description:	Method Blank	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	0.12	0.96	2.1
Total PeCDD	ND/EDL=0.10	1.61	ND/EDL=0.08
Total HxCDD	0.37	16.0	4.84
Total HpCDD	0.60	55.5	48.5
Furans			
Total TCDF	0.13	7.78	11.9
Total PeCDF	0.15	11.80	9.37
Total HxCDF	0.84	38.7	3.71
Total HpCDF	1.82	76.5	5.27

Table 2c

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	October 8, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT08OC01
Customer Project Number:	

MS File Number:	16DEC91LCB3041	16DEC91LCB3051
Keystone/NEA Number:	91TT08OC01-06	91TT08OC01-07
Customer Number:	D20	D23
Sample Description:	Sediment	Sediment
Units	pg/g (ppt)	pg/g (ppt)
Dioxins		
Total TCDD	1.54	1.40
Total PeCDD	0.67	0.66
Total HxCDD	10.4	6.19
Total HpCDD	105	31.1
Furans		
Total TCDF	10.8	10.8
Total PeCDF	5.60	5.42
Total HxCDF	6.08	4.01
Total HpCDF	11.2	6.34

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	October 8, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT08OC01
Customer Project Number:	

MS File Number:	16DEC91LCB3071	16DEC91LCB3081	16DEC91LCB3091
Keystone/NEA Number:	91TT08OC01-MB2	91TT08OC01-01	91TT08OC01-02
Customer Number:		D14	D15
Sample Description:	Method Blank	Sediment	Sediment
Units	%	%	%
Dioxins			
13C-2378-TCDD	87	94	94
13C-12378-PeCDD	112	121	125
13C-123478-HxCDD	79	89	95
13C-123678-HxCDD	80	73	72
13C-1234678-HpCDD	101	105	105
13C-OCDD	76	84	91
Furans			
13C-2378-TCDF	100	80*	83*
13C-12378-PeCDF	89	93	94
13C-23478-PeCDF	98	101	101
13C-123478-HxCDF	76	77	73
13C-123678-HxCDF	70	67	69
13C-234678-HxCDF	64	72	74
13C-123789-HxCDF	88	92	95
13C-1234678-HpCDF	86	84	89
13C-1234789-HpCDF	95	98	103
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	91	96	97

Notes:
1. Recoveries marked with an asterisk (*) are from a DB-225 column.

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received: October 8, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT08OC01
Customer Project Number:

MS File Number:	13DEC91LCB3101	13DEC91LCB3111	13DEC91LCB3121
Keystone/NEA Number:	91TT08OC01-03	91TT08OC01-03MS	91TT08OC01-03MSd
Customer Number:	D16	D16	D16
Sample Description:	Sediment	Sediment plus Matrix Spike	Sediment Matrix Spike Dup
Units	%	%	%
Dioxins			
13C-2378-TCDD	64	94	90
13C-12378-PeCDD	82	121	113
13C-123478-HxCDD	75	102	101
13C-123678-HxCDD	41	59	55
13C-1234789-HpCDD	74	104	100
13C-OCDD	70	97	100
Furans			
13C-2378-TCDF	55*	80*	76*
13C-12378-PeCDF	63	92	87
13C-23478-PeCDF	67	98	93
13C-123478-HxCDF	61	96	81
13C-123678-HxCDF	35	42	51
13C-234678-HxCDF	45	64	46
13C-123789-HxCDF	61	90	87
13C-1234678-HpCDF	54	78	78
13C-1234789-HpCDF	53	95	93
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	71	97	100

Notes:
1. Recoveries marked with an asterisk (*) are from a DB-225 column.

Table 3b

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received: October 8, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT08OC01
Customer Project Number:

MS File Number:	16DEC91LCB3011	16DEC91LCB3021	16DEC91LCB3031
Keystone/NEA Number:	91TT08OC01-MB1	91TT08OC01-04	91TT08OC01-05
Customer Number:		D18	D19
Sample Description:	Method Blank	Sediment	Sediment
Units	%	%	%
Dioxins			
13C-2378-TCDD	94	92	88
13C-12378-PeCDD	113	114	79
13C-123478-HxCDD	80	78	84
13C-123678-HxCDD	92	78	129
13C-1234678-HpCDD	99	93	152
13C-OCDD	81	79	141
Furans			
13C-2378-TCDF	106	80*	78*
13C-12378-PeCDF	92	88	91
13C-23478-PeCDF	102	98	63
13C-123478-HxCDF	84	75	108
13C-123678-HxCDF	82	69	82
13C-234678-HxCDF	68	61	82
13C-123789-HxCDF	97	88	117
13C-1234678-HpCDF	92	82	121
13C-1234789-HpCDF	100	93	148
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	104	92	95

Notes:
1. Recoveries marked with an asterisk (*) are from a DB-225 column.

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received: October 8, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT08OC01
Customer Project Number:

MS File Number:	16DEC91LCB3041	16DEC91LCB3051	16DEC91LCB3061
Keystone/NEA Number:	91TT08OC01-06	91TT08OC01-07	91TT08OC01-PAR
Customer Number:	D20	D23	
Sample Description:	Sediment	Sediment	PAR Sample
Units	%	%	%
Dioxins			
13C-2378-TCDD	90	89	95
13C-12378-PeCDD	115	113	118
13C-123478-HxCDD	81	84	80
13C-123678-HxCDD	74	67	87
13C-1234789-HpCDD	96	91	98
13C-OCDD	84	76	82
Furans			
13C-2378-TCDF	75*	76*	103
13C-12378-PeCDF	89	87	92
13C-23478-PeCDF	98	95	102
13C-123478-HxCDF	75	74	80
13C-123678-HxCDF	68	66	75
13C-234678-HxCDF	65	49	60
13C-123789-HxCDF	89	89	93
13C-1234678-HpCDF	80	78	87
13C-1234789-HpCDF	94	92	98
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	95	97	102

Notes:
1. Recoveries marked with an asterisk (*) are from a DB-225 column.

SUMMARY OF ANALYTICAL RESULTS

Precision and Recovery Samples

Date received: October 8, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT08OC01
Customer Project Number:
Invoice Number:

MS File Number: 16DEC91LCB3061
Keystone/NEA Number: 91TT08OC01-PAR

Sample Description:	Spiked Levels	Measured Levels	Percent Recy
Units	pg	pg	%
Dioxins			
2378-TCDD	200	238	119
12378-PeCDD	1079	1003	93
123478-HxCDD	904	1193	132
123678-HxCDD	888	1135	128
123789-HxCDD	783	1024	131
1234678-HpCDD	1012	1106	109
OCDD	1909	2593	136
Furans			
2378-TCDF	188	203	108
12378-PeCDF	931	1211	130
23478-PeCDF	880	1143	130
123478-HxCDF	950	1150	121
123678-HxCDF	934	1147	123
234678-HxCDF	904	1102	122
123789-HxCDF	960	1077	112
1234678-HpCDF	897	1168	130
1234789-HpCDF	948	1156	122
OCDF	1842	2333	127

SUMMARY OF ANALYTICAL RESULTS
Matrix Spike Samples

Date received: October 8, 1991
Client name: TetraTech
Laboratory Project Number: 91TT08OC01
Customer Project Number:

MS File Number:	16DEC91LCB3101			16DEC91LCB3111		
Keystone/NEA Number:	91TT08OC01-03			91TT08OC01-03MS		
Sample Description:	Measured Levels	Spiked Levels*	Spiked Levels**	Theoretical Levels	Measured Levels	% Dev.
Units	pg/g (ppt)	pg	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)	%
Dioxins						
2378-TCDD	0.35	200	6.34	6.69	7.32	9
12378-PeCDD	0.23	1079	34.22	34.45	28.3	-18
123478-HxCDD	0.74	904	28.67	29.41	34.4	17
123678-HxCDD	1.67	888	28.16	29.83	35.3	18
123789-HxCDD	1.59	783	24.83	26.42	44.3	68
1234678-HpCDD	28.8	1012	32.10	60.90	53.8	-12
OCDD	303	1909	60.55	363.55	294	-19
Furans						
2378-TCDF	4.60	188	5.96	10.56	9.31	-12
12378-PeCDF	0.57	931	29.53	30.10	34.8	16
23478-PeCDF	0.49	880	27.91	28.40	33.4	18
123478-HxCDF	1.14	950	30.13	31.27	35.4	13
123678-HxCDF	0.37	934	29.62	29.99	33.4	11
234678-HxCDF	0.61	904	28.67	29.28	32.3	10
123789-HxCDF	0.27	960	30.45	30.72	30.1	-2
1234678-HpCDF	5.14	897	28.45	33.59	38.4	14
1234789-HpCDF	0.75	948	30.07	30.82	32.7	6
OCDF	8.61	1842	58.42	67.03	77.0	15

Notes:
1. Concentrations marked with an asterisk (*) are the absolute amount of each native analyte spiked into the sample -03MS.
2. Concentrations marked with a double asterisk (**) are the spike levels expressed as pg/g (ppt) for a sample weight of 31.53 grams.

SUMMARY OF ANALYTICAL RESULTS
Matrix Spike Samples

Date received: October 8, 1991
Client name: TetraTech
Laboratory Project Number: 91TT08OC01
Customer Project Number:

MS File Number:	16DEC91LCB3101			16DEC91LCB3121		
Keystone/NEA Number:	91TT08OC01-03			91TT08OC01-03MSd		
Sample Description:	Measured Levels	Spiked Levels*	Spiked Levels**	Theoretical Levels	Measured Levels	% Dev.
Units	pg/g (ppt)	pg	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)	%
Dioxins						
2378-TCDD	0.35	200	5.50	5.85	6.79	16
12378-PeCDD	0.23	1079	29.68	29.91	27.3	-9
123478-HxCDD	0.74	904	24.86	25.60	31.9	25
123678-HxCDD	1.67	888	24.42	26.09	31.3	20
123789-HxCDD	1.59	783	21.53	23.12	40.8	76
1234678-HpCDD	28.8	1012	27.83	56.63	49.5	-13
OCDD	303	1909	52.50	355.50	255	-28
Furans						
2378-TCDF	4.60	188	5.17	9.77	9.19	-6
12378-PeCDF	0.57	931	25.61	26.18	32.4	24
23478-PeCDF	0.49	880	24.20	24.69	30.4	23
123478-HxCDF	1.14	950	26.13	27.27	29.6	9
123678-HxCDF	0.37	934	25.69	26.06	34.5	32
234678-HxCDF	0.61	904	24.86	25.47	29.6	16
123789-HxCDF	0.27	960	26.40	26.67	27.9	5
1234678-HpCDF	5.14	897	24.67	29.81	32.5	9
1234789-HpCDF	0.75	948	26.07	26.82	30.5	14
OCDF	8.61	1842	50.66	59.27	62.0	5

Notes:
1. Concentrations marked with an asterisk (*) are the absolute amount of each native analyte spiked into the sample -03MS.
2. Concentrations marked with a double asterisk (**) are the spike levels expressed as pg/g (ppt) for a sample weight of 36.36 grams.