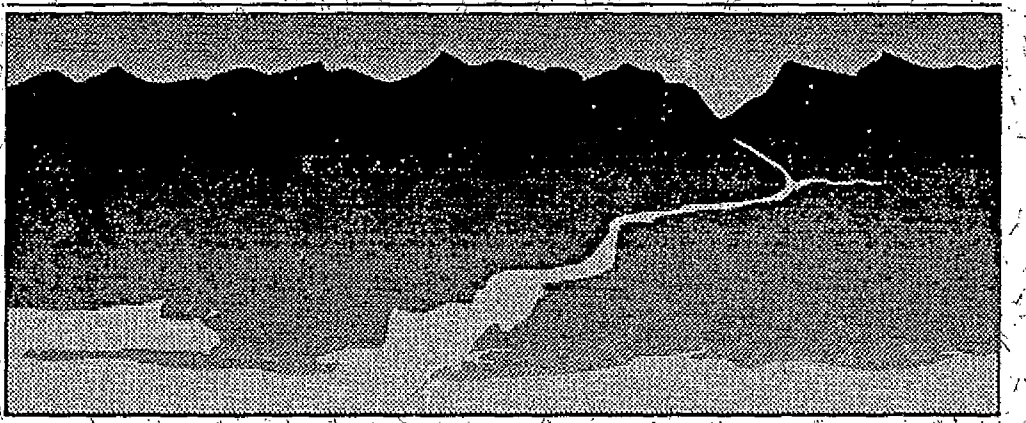
LOWER COLUMBIA RIVER



BI-STATE PROGRAM

RECONNAISSANCE SURVEY OF THE LOWER COLUMBIA RIVER

LABORATORY DATA REPORT
VOLUME 6: DIOXIN AND FURAN DATA FOR TISSUES

APRIL, 1992

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TETRA TECH

In Association With:

KEYSTONE/NEA

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VOLUME 6

DIOXIN/FURAN DATA - TISSUE

SECTION A. STURGEON

SECTION B. CRAYFISH

SECTION C. SUCKERS

SECTION D. CARP

SECTION E. PEAMOUTH CHUB

SECTION A. STURGEON

ANALYSIS OF STURGEON

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

Eight sturgeon samples were received for PCDD/PCDF analysis under Chain-of-Custody, between September 30 and October 24, 1991. The samples were in good condition upon receipt, and were stored in a freezer maintained at -21°C until analysis. The samples were thawed slightly, ground, then immediately returned to the freezer. The ground tissues were extracted in two sets on January 18, 1992. They were analyzed on a DB-5 column on February 6, 1992. Confirmation analyses were on a DB-225 column on February 13, 1992.

Two laboratory method blanks were analyzed with these sample sets. One sample, ST-4-1-D, 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 set of samples was as follows:

LAB ID NUMBER	ANALYSIS	DETECTION LIMIT
91TT30SP01	EPA Method 1613x	1 ppt (tetras)
91TT03OC01	EPA Method 1613x	1 ppt (tetras)
91TT04OC01	EPA Method 1613x	1 ppt (tetras)
91TT11OC01	EPA Method 1613x	1 ppt (tetras)
91TT22OC01	EPA Method 1613x	1 ppt (tetras)
91TT24OC01	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 and sample clean-up followed general guidelines outlined in EPA Method 8290. (Method 1613 does not cover extraction of fish tissue.) 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. The modifications are outlined below:

Clean-Up Recovery Standard Spiking Levels EPA Method 1613 calls for spiking the sample extracts with 800 pg of ^{37}Cl-2378-TCDD immediately prior to the clean-up procedure. That level has been reduced to 200 pg. The purpose of this change is to reduce the occurence of false positives due to 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 aqueous matrices.

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 for the continuing 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.)

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 20 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 20 gram sample, the LMCL and UMCL are:

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

NOTE: pg/g = ppt

D. Results

General Sturgeon tissue results are based on the initial weight of the sample (approximately 20 grams). All of the reported analyte concentrations are rounded to three significant figures. Percent lipids are rounded to 2 significant figures. Laboratory Method Blank results are also based on a theoretical sample size of 20 grams. Reported results for the 2378-TCDF are from a DB-225 column, and are highlighted with an asterisk (*). All other results are from a DB-5 column.

Sturgeon Sample Results Only one analyte was present in all eight of the sturgeon samples at levels above the LMCL: 2378-TCDF. The concentration of this analyte ranged from 1.54 ppt to 22.8 ppt. Octachlorodibenzo-*p*-dioxin (OCDD) was also present in most of the samples, but only in trace levels, and below the LMCL for that analyte. Otherwise, the eight sturgeon samples were Non-Detect for the remaining PCDD/PCDF isomers. (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 eight samples: ST-4-1-D.

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.

Both laboratory method blanks, 91TT11OC01-MB and 91TT30SP01-MB, were Non-Detect for all PCDD and PCDF isomers at or below the LMCL of 0.5 ppt (tetras), 2.5 ppt (pentas, hexas, heptas), and 5.0 ppt (octas). Many of the analytes had sample specific EDL's significantly below their respective LMCLs, ranging from 0.24 ppt to 1.77 ppt. One analyte, 234678-HxCDF, had an EDL which exceeded the LMCL of 2.5 ppt for that analyte. This was due to very low recovery of that internal standard (see discussion below).

Internal Standard Recoveries The Internal Standard recoveries for these samples are listed in Tables 3a - 3d. One analyte, ^{13}C-234678-HxCDF, had recoveries below the method guidelines in six of the twelve analyses. Since the samples were Non-Detect for that analyte, the only effect of the low recoveries was to raise the sample specific EDLs for the corresponding native furan.

The matrix spike duplicate had unusually low recoveries for several analytes due to laboratory induced interferences. All other recoveries for the 12 analyses were within method guidelines.

Matrix Spike Sample Results The results of the matrix spike and matrix spike duplicate are in Tables 4a and 4b, respectively. All of the results of the matrix spike are within method specifications (Table 4a). Four of the 17 analytes in the matrix spike duplicate are outside the method criteria of ±50% (Table 4b). Because laboratory interferences also caused many of those Internal Standard Recoveries to be out of range, the results in table 4a should be considered more reliable.

D. Quality Control Review

All of the data have been reviewed by the scientist performing the analysis, by the Director of the Center for Analytical Mass Spectrometry, and by 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 2/18/92
Laura Chambers Date
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Center for Analytical Mass Spectrometry

Peggy Meek 2/18/92
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W H Chambers 2.18.92.
William H. Chambers Date
Director
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Jeff Sprenger Date
QA Officer
Keystone/NEA

KEYSTONE/NEA
ENVIRONMENTAL RESOURCES

SAMPLE ANALYSIS SUMMARY

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 11, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT11OC01, 91TT22OC01, 91TT24OC01
Customer Project Number:	TC 8526-06

MS File Number:	06FEB92LCB2011	06FEB92LCB2021	06FEB92LCB2031
Keystone/NEA Number:	91TT11OC01-MB	91TT11OC01-01	91TT11OC01-02
Customer Number:		ST-1-2-D	ST-2-1-D
Sample Description:	Method Blank	Sturgeon	Sturgeon
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	ND/EDL=1.10	ND/EDL=1.00	ND/EDL=0.92
12378-PeCDD	ND/EDL=1.48	ND/EDL=1.02	ND/EDL=1.14
123478-HxCDD	ND/EDL=0.52	ND/EDL=0.50	ND/EDL=0.53
123678-HxCDD	ND/EDL=0.41	ND/EDL=0.36	ND/EDL=0.38
123789-HxCDD	ND/EDL=0.44	ND/EDL=0.40	ND/EDL=0.42
1234678-HpCDD	ND/EDL=1.77	ND/EDL=1.25	ND/EDL=1.09
OCDD	ND/EDL=0.81	ND/EDL=0.61	EMPC=0.98
Furans			
2378-TCDF	ND/EDL=0.44	1.54*	6.41*
12378-PeCDF	ND/EDL=0.30	ND/EDL=0.32	ND/EDL=0.25
23478-PeCDF	ND/EDL=0.29	ND/EDL=0.28	ND/EDL=0.24
123478-HxCDF	ND/EDL=1.28	ND/EDL=1.02	ND/EDL=1.15
123678-HxCDF	ND/EDL=1.16	ND/EDL=0.83	ND/EDL=0.88
234678-HxCDF	ND/EDL=5.65	ND/EDL=3.83	ND/EDL=3.09
123789-HxCDF	ND/EDL=1.34	ND/EDL=1.67	ND/EDL=1.74
1234678-HpCDF	ND/EDL=0.77	ND/EDL=0.58	ND/EDL=0.73
1234789-HpCDF	ND/EDL=1.12	ND/EDL=0.79	ND/EDL=1.00
OCDF	ND/EDL=0.93	ND/EDL=0.65	ND/EDL=0.82
Percent Lipids	0%	1.3%	4.5%

Notes:

1. ND/EDL=Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. ND/LMCL = Analyte Not Detected at or above the Lower Method Calibration Limit.
4. Concentrations marked with an asterisk (*) are from a DB-225 column.
5. Concentrations below the LMCL should be considered ESTIMATES ONLY. (See Case Narrative.)

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 22 & 24, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT11OC01, 91TT22OC01, 91TT24OC01
Customer Project Number:	TC 8526-06

MS File Number:	06FEB92LCB2041	06FEB92LCB2051	06FEB92LCB4011
Keystone/NEA Number:	91TT22OC01-04	91TT24OC01-02	91TT24OC01-03
Customer Number:	ST-2-2-D	ST-3-3-D	ST-3-1-D
Sample Description:	Sturgeon	Sturgeon	Sturgeon
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	ND/EDL=0.79	ND/EDL=0.72	1.66
12378-PeCDD	ND/EDL=0.92	ND/EDL=0.87	ND/EDL=0.90
123478-HxCDD	ND/EDL=0.40	ND/EDL=0.43	ND/EDL=0.42
123678-HxCDD	ND/EDL=0.30	ND/EDL=0.33	ND/EDL=0.31
123789-HxCDD	ND/EDL=0.33	ND/EDL=0.36	ND/EDL=0.34
1234678-HpCDD	ND/EDL=1.00	ND/EDL=0.87	ND/EDL=1.03
OCDD	EMPC=2.22	2.90	EMPC=1.48
Furans			
2378-TCDF	1.66*	22.6*	22.8*
12378-PeCDF	ND/EDL=0.27	ND/EDL=0.29	EMPC=0.73
23478-PeCDF	ND/EDL=0.24	ND/EDL=0.28	EMPC=0.49
123478-HxCDF	ND/EDL=0.72	ND/EDL=1.08	ND/EDL=1.30
123678-HxCDF	ND/EDL=0.62	ND/EDL=0.90	ND/EDL=1.10
234678-HxCDF	ND/EDL=1.95	ND/EDL=4.81	ND/EDL=3.66
123789-HxCDF	ND/EDL=1.09	ND/EDL=1.78	ND/EDL=2.04
1234678-HpCDF	ND/EDL=0.59	ND/EDL=0.47	ND/EDL=0.84
1234789-HpCDF	ND/EDL=0.78	ND/EDL=0.63	ND/EDL=0.57
OCDF	ND/EDL=0.93	ND/EDL=0.82	ND/EDL=0.72
Percent Lipids	0%	6.6%	3.9%

Notes:

1. ND/EDL=Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. ND/LMCL = Analyte Not Detected at or above the Lower Method Calibration Limit.
4. Concentrations marked with an asterisk (*) are from a DB-225 column.
5. Concentrations below the LMCL should be considered ESTIMATES ONLY. (See Case Narrative.)

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 22 & 24, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT11OC01, 91TT22OC01, 91TT24OC01
Customer Project Number:	TC 8526-06

MS File Number:	06FEB92LCB4061	06FEB92LCB4071	06FEB92LCB4081
Keystone/NEA Number:	91TT30SP01-MB	91TT30SP01-02	91TT03OC01-01
Customer Number:		ST-4-3-D	ST-1-3-D
Sample Description:	Method Blank	Sturgeon	Sturgeon
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	ND/EDL=0.88	ND/EDL=0.59	ND/EDL=1.07
12378-PeCDD	ND/EDL=0.92	ND/EDL=0.61	ND/LMCL=2.50
123478-HxCDD	ND/EDL=0.56	ND/EDL=0.47	ND/EDL=0.18
123678-HxCDD	ND/EDL=0.42	ND/EDL=0.35	ND/EDL=0.17
123789-HxCDD	ND/EDL=0.46	ND/EDL=0.39	ND/EDL=0.19
1234678-HpCDD	ND/EDL=1.45	EMPC=0.50	0.35
OCDD	ND/EDL=1.21	EMPC=3.61	0.25
Furans			
2378-TCDF	ND/EDL=0.28	13.3*	5.52*
12378-PeCDF	ND/EDL=0.21	ND/EDL=0.31	ND/LMCL=2.50
23478-PeCDF	ND/EDL=0.20	ND/EDL=0.28	ND/LMCL=2.50
123478-HxCDF	ND/EDL=0.84	ND/EDL=0.80	ND/EDL=0.31
123678-HxCDF	ND/EDL=0.77	ND/EDL=0.70	ND/EDL=0.31
234678-HxCDF	ND/EDL=1.11	ND/EDL=1.27	ND/EDL=0.35
123789-HxCDF	ND/EDL=1.54	ND/EDL=1.33	ND/EDL=0.41
1234678-HpCDF	ND/EDL=0.52	ND/EDL=0.53	ND/EDL=0.20
1234789-HpCDF	ND/EDL=0.82	ND/EDL=0.84	ND/EDL=0.26
OCDF	ND/EDL=0.90	ND/EDL=0.49	ND/EDL=0.29
Percent Lipids	0%	2.3%	6.1%

Notes:

1. ND/EDL=Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. ND/LMCL = Analyte Not Detected at or above the Lower Method Calibration Limit.
4. Concentrations marked with an asterisk (*) are from a DB-225 column.
5. Concentrations below the LMCL should be considered ESTIMATES ONLY. (See Case Narrative.)

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 22 & 24, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT11OC01, 91TT22OC01, 91TT24OC01
Customer Project Number:	TC 8526-06

MS File Number:	06FEB92LCB4091
Keystone/NEA Number:	91TT04OC01-01
Customer Number:	ST-4-1-D
Sample Description:	Sturgeon

Units	pg/g (ppt)

Dioxins	
2378-TCDD	ND/EDL=0.62
12378-PeCDD	ND/EDL=0.57
123478-HxCDD	ND/EDL=0.37
123678-HxCDD	ND/EDL=0.30
123789-HxCDD	ND/EDL=0.33
1234678-HpCDD	ND/EDL=0.63
OCDD	1.07
Furans	
2378-TCDF	3.53*
12378-PeCDF	ND/EDL=0.26
23478-PeCDF	ND/EDL=0.21
123478-HxCDF	ND/EDL=0.67
123678-HxCDF	ND/EDL=0.58
234678-HxCDF	ND/EDL=0.83
123789-HxCDF	ND/EDL=1.13
1234678-HpCDF	ND/EDL=0.50
1234789-HpCDF	ND/EDL=0.69
OCDF	ND/EDL=0.61
Percent Lipids	3.9%

Notes:

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2. EMPC = Estimated Maximum Possible Concentration.
3. ND/LMCL = Analyte Not Detected at or above the Lower Method Calibration Limit.
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5. Concentrations below the LMCL should be considered ESTIMATES ONLY. (See Case Narrative.)

SUMMARY OF ANALYTICAL RESULTS

Homologue Group Totals

Date received:	October 11, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT11OC01, 91TT22OC01, 91TT24OC01
Customer Project Number:	TC 8526-06

MS File Number:	06FEB92LCB2011	06FEB92LCB2021	06FEB92LCB2031
Keystone/NEA Number:	91TT11OC01-MB	91TT11OC01-01	91TT11OC01-02
Customer Number:		ST-1-2-D	ST-2-1-D
Sample Description:	Method Blank	Sturgeon	Sturgeon
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	ND/EDL=1.10	0.73	ND/EDL=0.92
Total PeCDD	ND/EDL=1.48	ND/EDL=1.02	ND/EDL=1.14
Total HxCDD	ND/EDL=0.41	ND/EDL=0.36	ND/EDL=0.38
Total HpCDD	ND/EDL=1.77	ND/EDL=1.25	ND/EDL=1.09
Furans			
Total TCDF	ND/EDL=0.44	1.63	6.22
Total PeCDF	ND/EDL=0.29	ND/EDL=0.28	ND/EDL=0.24
Total HxCDF	ND/EDL=1.16	ND/EDL=0.83	ND/EDL=0.88
Total HpCDF	ND/EDL=0.77	ND/EDL=0.58	ND/EDL=0.73

Notes:

1. ND/EDL=Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. ND/LMCL = Analyte Not Detected at or above the Lower Method Calibration Limit.
4. Concentrations below the LMCL should be considered ESTIMATES ONLY. (See Case Narrative.)

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	October 22 & 24, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT11OC01, 91TT22OC01, 91TT24OC01
Customer Project Number:	TC 8526-06

MS File Number:	06FEB92LCB2041	06FEB92LCB2051	06FEB92LCB4011
Keystone/NEA Number:	91TT22OC01-04	91TT24OC01-02	91TT24OC01-03
Customer Number:	ST-2-2-D	ST-3-3-D	ST-3-1-D
Sample Description:	Sturgeon	Sturgeon	Sturgeon
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	ND/EDL=0.79	ND/EDL=0.72	2.74
Total PeCDD	ND/EDL=0.92	ND/EDL=0.87	ND/EDL=0.90
Total HxCDD	ND/EDL=0.30	ND/EDL=0.33	ND/EDL=0.31
Total HpCDD	ND/EDL=1.00	ND/EDL=0.87	ND/EDL=1.03
Furans			
Total TCDF	1.68	20.0	18.8
Total PeCDF	ND/EDL=0.24	ND/EDL=0.28	ND/LMCL=2.50
Total HxCDF	ND/EDL=0.62	ND/EDL=0.90	ND/EDL=1.10
Total HpCDF	ND/EDL=0.59	ND/EDL=0.47	0.67

Notes:

1. ND/EDL=Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. ND/LMCL = Analyte Not Detected at or above the Lower Method Calibration Limit.
4. Concentrations below the LMCL should be considered ESTIMATES ONLY. (See Case Narrative.)

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	October 22 & 24, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT11OC01, 91TT22OC01, 91TT24OC01
Customer Project Number:	TC 8526-06

MS File Number:	06FEB92LCB4061	06FEB92LCB4071	06FEB92LCB4081
Keystone/NEA Number:	91TT30SP01-MB	91TT30SP01-02	91TT03OC01-01
Customer Number:		ST-4-3-D	ST-1-3-D
Sample Description:	Method Blank	Sturgeon	Sturgeon
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	ND/EDL=0.88	0.87	0.73
Total PeCDD	ND/EDL=0.92	ND/EDL=0.61	ND/LMCL=2.50
Total HxCDD	ND/EDL=0.42	ND/EDL=0.35	ND/EDL=0.17
Total HpCDD	ND/EDL=1.45	ND/EDL=0.72	0.35
Furans			
Total TCDF	ND/EDL=0.28	10.9	4.85
Total PeCDF	ND/EDL=0.20	0.56	ND/LMCL=2.50
Total HxCDF	ND/EDL=0.77	0.97	0.50
Total HpCDF	ND/EDL=0.52	ND/EDL=0.53	ND/EDL=0.20

Notes:

1. ND/EDL=Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. ND/LMCL = Analyte Not Detected at or above the Lower Method Calibration Limit.
4. Concentrations below the LMCL should be considered ESTIMATES ONLY. (See Case Narrative.)

SUMMARY OF ANALYTICAL RESULTS

Homologue Group Totals

Date received:	October 22 & 24, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT11OC01, 91TT22OC01, 91TT24OC01
Customer Project Number:	TC 8526-06

MS File Number:	06FEB92LCB4091
Keystone/NEA Number:	91TT04OC01-01
Customer Number:	ST-4-1-D
Sample Description:	Sturgeon

Units	pg/g (ppt)

Dioxins	
Total TCDD	ND/EDL=0.62
Total PeCDD	ND/EDL=0.57
Total HxCDD	ND/EDL=0.30
Total HpCDD	ND/EDL=0.63
Furans	
Total TCDF	3.58
Total PeCDF	0.64
Total HxCDF	ND/EDL=0.58
Total HpCDF	ND/EDL=0.50

Notes:

1. ND/EDL=Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. EMPC = Estimated Maximum Possible Concentration.
3. ND/LMCL = Analyte Not Detected at or above the Lower Method Calibration Limit.
4. Concentrations below the LMCL should be considered ESTIMATES ONLY. (See Case Narrative.)

SUMMARY OF ANALYTICAL RESULTS

Internal Standard Recoveries

Date received:	October 11, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT11OC01, 91TT22OC01, 91TT24OC01
Customer Project Number:	TC 8526-06

MS File Number:	06FEB92LCB2011	06FEB92LCB2021	06FEB92LCB2031
Keystone/NEA Number:	91TT11OC01-MB	91TT11OC01-01	91TT11OC01-02
Customer Number:		ST-1-2-D	ST-2-1-D
Sample Description:	Method Blank	Sturgeon	Sturgeon
Units	%	%	%
Dioxins			
13C-2378-TCDD	50	52	56
13C-12378-PeCDD	57	64	73
13C-123478-HxCDD	40	96	48
13C-123678-HxCDD	64	76	73
13C-1234678-HpCDD	45	56	55
13C-OCDD	41	55	61
Furans			
13C-2378-TCDF	47	57*	57*
13C-12378-PeCDF	50	51	55
13C-23478-PeCDF	52	62	57
13C-123478-HxCDF	44	50	50
13C-123678-HxCDF	59	66	66
13C-234678-HxCDF	12	15	21
13C-123789-HxCDF	43	54	50
13C-1234678-HpCDF	46	57	57
13C-1234789-HpCDF	44	58	51
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	71	63	72

Notes:

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

SUMMARY OF ANALYTICAL RESULTS

Internal Standard Recoveries

Date received:	October 22 & 24, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT11OC01, 91TT22OC01, 91TT24OC01
Customer Project Number:	TC 8526-06

MS File Number:	06FEB92LCB2041	06FEB92LCB2051	06FEB92LCB4011
Keystone/NEA Number:	91TT22OC01-04	91TT24OC01-02	91TT24OC01-03
Customer Number:	ST-2-2-D	ST-3-3-D	ST-3-1-D
Sample Description:	Sturgeon	Sturgeon	Sturgeon
Units	%	%	%
Dioxins			
13C-2378-TCDD	51	60	58
13C-12378-PeCDD	66	76	64
13C-123478-HxCDD	44	45	52
13C-123678-HxCDD	72	81	68
13C-1234789-HpCDD	54	65	53
13C-OCDD	49	61	52
Furans			
13C-2378-TCDF	57*	64*	61*
13C-12378-PeCDF	52	60	49
13C-23478-PeCDF	58	64	50
13C-123478-HxCDF	50	51	49
13C-123678-HxCDF	64	70	62
13C-234678-HxCDF	24	15	22
13C-123789-HxCDF	53	53	50
13C-1234678-HpCDF	52	60	54
13C-1234789-HpCDF	55	63	57
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	67	79	71

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	October 22 & 24, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT11OC01, 91TT22OC01, 91TT24OC01
Customer Project Number:	TC 8526-06

MS File Number:	06FEB92LCB4061	06FEB92LCB4071	06FEB92LCB4081
Keystone/NEA Number:	91TT30SP01-MB	91TT30SP01-02	91TT03OC01-01
Customer Number:		ST-4-3-D	ST-1-3-D
Sample Description:	Method Blank	Sturgeon	Sturgeon
Units	%	%	%
Dioxins			
13C-2378-TCDD	48	53	56
13C-12378-PeCDD	66	74	78
13C-123478-HxCDD	49	47	83
13C-123678-HxCDD	67	78	78
13C-1234789-HpCDD	47	63	85
13C-OCDD	45	68	76
Furans			
13C-2378-TCDF	44	59*	73*
13C-12378-PeCDF	47	57	37
13C-23478-PeCDF	61	62	33
13C-123478-HxCDF	51	59	63
13C-123678-HxCDF	61	63	58
13C-234678-HxCDF	47	41	61
13C-123789-HxCDF	44	55	66
13C-1234678-HpCDF	54	67	74
13C-1234789-HpCDF	45	66	78
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	68	69	75

Notes:

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

SUMMARY OF ANALYTICAL RESULTS

Internal Standard Recoveries

Date received:	October 22 & 24, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT11OC01, 91TT22OC01, 91TT24OC01
Customer Project Number:	TC 8526-06

MS File Number:	06FEB92LCB4091	06FEB92LCB4101	06FEB92LCB4111
Keystone/NEA Number:	91TT04OC01-01	91TT04OC01-01MS	91TT04OC01-01MSd
Customer Number:	ST-4-1-D	ST-4-1-D	ST-4-1-D
Sample Description:	Sturgeon	Matrix Spike	Matrix Spike Duplicate
Units	%	%	%
Dioxins			
13C-2378-TCDD	52	59	48
13C-12378-PeCDD	81	86	80
13C-123478-HxCDD	57	69	35
13C-123678-HxCDD	65	74	39
13C-1234789-HpCDD	65	68	41
13C-OCDD	65	70	18
Furans			
13C-2378-TCDF	56*	60*	48*
13C-12378-PeCDF	55	60	64
13C-23478-PeCDF	67	77	53
13C-123478-HxCDF	54	55	31
13C-123678-HxCDF	57	60	29
13C-234678-HxCDF	49	55	25
13C-123789-HxCDF	49	58	12
13C-1234678-HpCDF	64	62	13
13C-1234789-HpCDF	60	67	6
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	66	69	72

Notes:

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

SUMMARY OF ANALYTICAL RESULTS

Matrix Spike Samples

Date received: October 11, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT11OC01, 91TT22OC01, 91TT24OC01
Customer Project Number: TC 8526-06

MS File Number:	06FEB92LCB4091			06FEB92LCB4101		
Keystone/NEA Number:	91TT04OC01-01			91TT04OC01-01MS		
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	0.00	200	9.66	9.66	10.70	11
12378-PeCDD	0.00	1079	52.14	52.14	49.5	-5
123478-HxCDD	0.00	904	43.68	43.68	54.1	24
123678-HxCDD	0.00	888	42.91	42.91	53.4	24
123789-HxCDD	0.00	783	37.83	37.83	47.7	26
1234678-HpCDD	0.00	1012	48.90	48.90	57.6	18
OCDD	1.07	1909	92.24	93.31	119	28
Furans						
2378-TCDF	3.58	188	9.08	12.66	15.2	20
12378-PeCDF	0.00	931	44.98	44.98	61.6	37
23478-PeCDF	0.00	880	42.52	42.52	57.3	35
123478-HxCDF	0.00	950	45.90	45.90	57.6	25
123678-HxCDF	0.00	934	45.13	45.13	56.9	26
234678-HxCDF	0.00	904	43.68	43.68	53.4	22
123789-HxCDF	0.00	960	46.39	46.39	53.6	16
1234678-HpCDF	0.00	897	43.34	43.34	56.8	31
1234789-HpCDF	0.00	948	45.81	45.81	54.6	19
OCDF	0.00	1842	89.00	89.00	107.0	20

Notes:

1. Concentrations marked with an asterisk (*) are the absolute amount of each native analyte spiked into the sample -01MS.
2. Concentrations marked with a double asterisk (**) are the spike levels expressed as pg/g (ppt) for a sample weight of 20.696 grams.
3. The measured level of 2378-TCDF is from a DB-225 column.

SUMMARY OF ANALYTICAL RESULTS

Matrix Spike Samples

Date received: October 11, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT11OC01, 91TT22OC01, 91TT24OC01
Customer Project Number: TC 8526-06

MS File Number:	06FEB92LCB4091			06FEB92LCB4111		
Keystone/NEA Number:	91TT04OC01-01			91TT04OC01-01MSd		
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	0.00	200	9.71	9.71	11.7	21
12378-PeCDD	0.00	1,079	52.4	52.4	50.0	-5
123478-HxCDD	0.00	904	43.9	43.9	56.2	28
123678-HxCDD	0.00	888	43.1	43.1	38.2	-11
123789-HxCDD	0.00	783	38.0	38.0	50.5	33
1234678-HpCDD	0.00	1,012	49.1	49.1	82.9	69
OCDD	1.07	1,909	92.7	93.7	213	127
Furans						
2378-TCDF	3.58	188	9.13	12.7	16.6	31
12378-PeCDF	0.00	931	45.2	45.2	63.6	41
23478-PeCDF	0.00	880	42.7	42.7	62.2	46
123478-HxCDF	0.00	950	46.1	46.1	65.4	42
123678-HxCDF	0.00	934	45.3	45.3	57.9	28
234678-HxCDF	0.00	904	43.9	43.9	61.3	40
123789-HxCDF	0.00	960	46.6	46.6	56.1	20
1234678-HpCDF	0.00	897	43.5	43.5	107	146
1234789-HpCDF	0.00	948	46.0	46.0	52.3	14
OCDF	0.00	1,842	89.4	89.4	18.9	-79

Notes:

1. Concentrations marked with an asterisk (*) are the absolute amount of each native analyte spiked into the sample -01dMS.
2. Concentrations marked with a double asterisk (**) are the spike levels expressed as pg/g (ppt) for a sample weight of 20.601 grams.
3. The measured level of 2378-TCDF is from a DB-225 column.

SECTION B. CRAYFISH

ANALYSIS OF CRAYFISH

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

Eighteen crayfish samples were received under Chain-of-Custody on September 26, 27, and October 5, 1991. The samples were in good condition upon receipt, and were stored in a freezer maintained at -21°C. The samples were homogenized by grinding several times and immediately returned to the freezer where they remained until extraction. Customer sample number D19 was not processed or analyzed, as requested by the client on October 24,1991.

Three laboratory Method Blanks, and one Matrix Spike and Matrix Spike Duplicate were also analyzed with this sample set.

II. ANALYSIS REQUEST

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

LAB ID NUMBER	ANALYSIS	LMCL
91TT26SP01	EPA Method 1613x	0.5 ppt (tetras)
91TT27SP02	EPA Method 1613x	2.5 ppt (pentas, hexas, heptas)
91TT05OC01	EPA Method 1613x	5.0 ppt (octas)

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 under "Analytical Methodology", below.

The samples were extracted in three sets on January 20, 22, and 31,1991. They were analyzed on a DB-5 column on March 3 and 5, 1992. Confirmation analyses were on a DB-225 column on February 15,1992.

B. Results

General Crayfish results are based on the initial weight/volume of the sample (approximately 20 grams). All results are reported to three significant figures. Laboratory Method Blank results are also based on a theoretical sample size of 20 grams. Reported results (concentrations and recoveries) for the 2378-TCDF are from a DB-225 column, and have been highlighted with an asterisk (*). All other results are from a DB-5 column. Concentrations below the LMCL should be considered ESTIMATES ONLY.

Sample Results Only one of the seventeen 2378-substituted isomers was consistently present in the samples at levels above the LMCL. The 2378-TCDF isomer was present in all samples at concentrations ranging from 4.10 ppt (sample D35) to 12.4 ppt (sample D28).

The OCDD isomer was present in most samples with concentrations ranging from 3.12 ppt (below the Lower Method Calibration Limit, LMCL), to 79.1 ppt (samples D40 and D28 respectively).

Most of the remaining 2378-substituted isomers were either consistently present at trace levels below the LMCL or were Non-Detect. No pattern is observed in the Homologue Group totals for the eighteen samples analyzed. Internal Standard recoveries and Clean-up Standard recoveries were within method guidelines for all samples.

During the first extraction, procedural inconsistencies caused the percent lipid determinations to be inaccurate for some samples. Those procedures have been performed a second time, and percent lipids calculated for all samples. The levels ranged from approximately 2% to 4.5%.

C. Analytical Methodology

The extraction and sample clean-up followed general guidelines outlined in EPA Method 8290. (Method 1613 does not cover extraction of fish tissue.) 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 occurrence 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. 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. The purpose of this change is to reduce the occurrence of false positives due to 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 aqueous matrices.

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 for the continuing 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).

D. 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 or above the UMCL, 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) can be calculated based on a sample size of 20 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 sample size of 20 grams:

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

Note: pg/g = ppt

IV. QUALITY CONTROL

A. Project Quality Control

Quality control measures specific to this project included a Matrix Spike and Matrix Spike Duplicate on one of the crayfish tissue samples. The project quality control plan specified a Matrix Spike and Matrix Spike Duplicate (MS/MSd) for every 20 samples run. In practice, a minimum of one MS/MSd pair was run with each tissue type (eg. sturgeon, carp, crayfish, etc.).

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. Documentation of initial and continuing calibrations, and GC and MS resolution checks can be found in the "QUALITY CONTROL DOCUMENTS" section of this report.

Continuing Calibration The continuing calibration which followed the DB-225 analysis for native 2378-TCDF deviated from the initial calibration by 30%. This exceeds the method requirement of a maximum 25% deviation, and means that the reported concentrations for native 2378-TCDF may be overstated by 5%. An alternative approach would be to calculate the concentration of 2378-TCDF using the average relative response factors from continuing calibrations before and after the run.

C. Laboratory Quality Control

Laboratory Method Blank Three laboratory method blanks were analyzed with this 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.

The laboratory method blanks 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). Many of the analytes, however, had sample specific EDL's significantly lower than the LMCL, ranging from 0.07 ppt to 0.56 ppt. Five analytes were present at levels below the LMCL for their particular homologue group, and would not normally be reported under method 1613, but are included for your review.

Matrix Spike Samples Tables 5a-5c in the "SAMPLE ANALYSIS SUMMARY" section of this report lists the results for the Matrix Spike and Matrix Spike Duplicate. The Relative Percent Difference (RPD) between the duplicate analyses ranged from 3% to 18%, and all were within method requirements.

D. Quality Control Review

All of the data have 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 3/21/92
Laura Chambers Date
Sr. Scientist
Center for Analytical Mass Spectrometry

Peggy Meek 3/26/92
Peggy L. Meek Date
Wet Lab Supervisor
Center for Analytical Mass Spectrometry

William H. Chambers 3.21.92.
William H. Chambers Date
Director
Center for Analytical Mass Spectrometry

J. Sprenger 3/24/92
Jeff Sprenger Date
QA Officer
Keystone/NEA

KEYSTONE/NEA
ENVIRONMENTAL RESOURCES

SAMPLE ANALYSIS SUMMARY

SUMMARY OF ANALYTICAL RESULTS

2378-Substituted Isomers

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	03MAR92LCB4041	03MAR92LCB4021	03MAR92LCB4031
Keystone/NEA Number:	91TT26SP01-MB	91TT27SP02-MB	91TT05OC01-MB
Customer Number:			
Sample Description:	Method Blank	Method Blank	Method Blank
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	ND/EDL=0.16	ND/EDL=0.12	ND/EDL=0.17
12378-PeCDD	ND/EDL=0.28	ND/EDL=0.29	ND/EDL=0.29
123478-HxCDD	ND/EDL=0.34	ND/EDL=0.15	ND/EDL=0.08
123678-HxCDD	0.40	ND/EDL=0.14	ND/EDL=0.07
123789-HxCDD	ND/EDL=0.30	ND/EDL=0.13	ND/EDL=0.30
1234678-HpCDD	1.24	0.30	ND/EDL=0.30
OCDD	EMPC=1.65	ND/EDL=0.42	ND/EDL=0.29
Furans			
2378-TCDF	ND/EDL=0.22	ND/EDL=0.16	ND/EDL=0.16
12378-PeCDF	ND/EDL=0.27	ND/EDL=0.20	ND/EDL=0.07
23478-PeCDF	ND/EDL=0.24	ND/EDL=0.20	ND/EDL=0.08
123478-HxCDF	ND/EDL=0.33	ND/EDL=0.36	ND/EDL=0.34
123678-HxCDF	ND/EDL=0.34	ND/EDL=0.34	ND/EDL=0.32
234678-HxCDF	0.33	ND/EDL=0.47	0.19
123789-HxCDF	ND/EDL=0.50	ND/EDL=0.56	ND/EDL=0.55
1234678-HpCDF	EMPC=0.26	ND/EDL=0.10	ND/EDL=0.18
1234789-HpCDF	ND/EDL=0.30	ND/EDL=0.13	ND/EDL=0.20
OCDF	ND/EDL=0.45	ND/EDL=0.35	ND/EDL=0.44
Percent Lipids	N.A.	N.A.	N.A.

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	05MAR92LCB3171	03MAR92LCB4051	03MAR92LCB4061
Keystone/NEA Number:	91TT26SP01-02	91TT27SP02-01	91TT27SP02-03
Customer Number:	D35	D28	D38
Sample Description:	Crayfish	Crayfish	Crayfish
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	EMPC=0.40	0.86	EMPC=0.40
12378-PeCDD	0.48	ND/EDL=0.32	ND/EDL=0.27
123478-HxCDD	EMPC=0.15	EMPC=0.16	ND/EDL=0.24
123678-HxCDD	0.53	EMPC=0.32	ND/EDL=0.25
123789-HxCDD	0.59	ND/EDL=0.19	ND/EDL=0.25
1234678-HpCDD	2.07	5.21	ND/EDL=0.32
OCDD	5.72	79.10	EMPC=1.62
Furans			
2378-TCDF	4.10*	12.4*	4.83*
12378-PeCDF	0.30	EMPC=0.39	ND/EDL=0.42
23478-PeCDF	EMPC=0.48	EMPC=0.85	EMPC=0.29
123478-HxCDF	0.21	0.28	ND/EDL=0.42
123678-HxCDF	EMPC=0.18	EMPC=0.32	ND/EDL=0.40
234678-HxCDF	0.48	7.26	EMPC=0.34
123789-HxCDF	EMPC=0.13	ND/EDL=0.71	ND/EDL=0.59
1234678-HpCDF	0.29	EMPC=0.31	EMPC=0.45
1234789-HpCDF	ND/EDL=0.07	ND/EDL=0.35	ND/EDL=0.24
OCDF	EMPC=0.42	EMPC=1.24	EMPC=0.60
Percent Lipids	3.36	3.78	3.89

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	03MAR92LCB4071	03MAR92LCB4081	03MAR92LCB4091
Keystone/NEA Number:	91TT27SP02-04	91TT05OC01-01	91TT05OC01-02
Customer Number:	D40	D6	D8
Sample Description:	Crayfish	Crayfish	Crayfish
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	0.27	0.44	0.45
12378-PeCDD	ND/EDL=0.22	ND/EDL=0.19	ND/EDL=0.18
123478-HxCDD	ND/EDL=0.20	ND/EDL=0.16	ND/EDL=0.08
123678-HxCDD	ND/EDL=0.19	ND/EDL=0.16	ND/EDL=0.07
123789-HxCDD	ND/EDL=0.18	ND/EDL=0.16	ND/EDL=0.07
1234678-HpCDD	0.62	EMPC=0.42	EMPC=0.67
OCDD	3.12	2.22	4.12
Furans			
2378-TCDF	4.81*	4.66*	4.72*
12378-PeCDF	ND/EDL=0.26	0.14	EMPC=0.11
23478-PeCDF	EMPC=0.22	0.23	EMPC=0.22
123478-HxCDF	ND/EDL=0.32	ND/EDL=0.27	ND/EDL=0.24
123678-HxCDF	ND/EDL=0.31	ND/EDL=0.27	ND/EDL=0.22
234678-HxCDF	EMPC=0.27	ND/EDL=0.32	EMPC=0.21
123789-HxCDF	ND/EDL=0.50	ND/EDL=0.41	ND/EDL=0.31
1234678-HpCDF	ND/EDL=0.09	ND/EDL=0.13	ND/EDL=0.10
1234789-HpCDF	ND/EDL=0.14	ND/EDL=0.15	ND/EDL=0.13
OCDF	ND/EDL=0.24	ND/EDL=0.29	ND/EDL=0.18
Percent Lipids	3.32	4.43	2.96

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	03MAR92LCB4111	03MAR92LCB4121	03MAR92LCB4131
Keystone/NEA Number:	91TT05OC01-03	91TT05OC01-05	91TT05OC01-07
Customer Number:	D10	D15	D19
Sample Description:	Crayfish	Crayfish	Crayfish
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	0.45	EMPC=0.39	0.62
12378-PeCDD	ND/EDL=0.17	ND/EDL=0.14	ND/EDL=0.66
123478-HxCDD	ND/EDL=0.13	ND/EDL=0.08	ND/EDL=0.21
123678-HxCDD	EMPC=0.38	ND/EDL=0.07	ND/EDL=0.30
123789-HxCDD	ND/EDL=0.12	ND/EDL=0.07	ND/EDL=0.18
1234678-HpCDD	1.57	EMPC=0.53	1.18
OCDD	7.81	3.38	6.52
Furans			
2378-TCDF	4.41*	4.12*	9.52*
12378-PeCDF	ND/EDL=0.16	ND/EDL=0.19	1.02
23478-PeCDF	EMPC=0.24	EMPC=0.29	3.05
123478-HxCDF	ND/EDL=0.26	ND/EDL=0.09	0.35
123678-HxCDF	ND/EDL=0.25	ND/EDL=0.09	0.24
234678-HxCDF	0.26	0.28	0.46
123789-HxCDF	ND/EDL=0.35	ND/EDL=0.16	ND/EDL=0.05
1234678-HpCDF	0.29	EMPC=0.27	ND/EDL=0.31
1234789-HpCDF	ND/EDL=0.16	ND/EDL=0.16	ND/EDL=0.09
OCDF	ND/EDL=0.35	ND/EDL=0.52	0.56
Percent Lipids	2.15	2.05	2.54

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	03MAR92LCB4141	03MAR92LCB4151	05MAR92LCB3201
Keystone/NEA Number:	91TT05OC01-08	91TT05OC01-10	91TT05OC01-11
Customer Number:	D20	D23	D24
Sample Description:	Crayfish	Crayfish	Crayfish
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	EMPC=0.39	0.43	0.47
12378-PeCDD	ND/EDL=0.09	ND/EDL=0.32	ND/EDL=0.83
123478-HxCDD	ND/EDL=0.30	ND/EDL=0.10	0.39
123678-HxCDD	ND/EDL=0.30	0.31	0.89
123789-HxCDD	ND/EDL=0.29	EMPC=0.15	EMPC=0.76
1234678-HpCDD	0.47	0.71	4.01
OCDD	3.33	4.67	16.70
Furans			
2378-TCDF	5.64*	6.08*	6.39*
12378-PeCDF	0.17	EMPC=0.25	0.67
23478-PeCDF	0.20	EMPC=0.42	0.98
123478-HxCDF	ND/EDL=0.09	ND/EDL=0.07	0.36
123678-HxCDF	ND/EDL=0.10	ND/EDL=0.06	0.32
234678-HxCDF	0.35	0.33	0.84
123789-HxCDF	ND/EDL=0.12	ND/EDL=0.09	0.23
1234678-HpCDF	ND/EDL=0.13	EMPC=0.37	0.70
1234789-HpCDF	ND/EDL=0.17	ND/EDL=0.27	0.19
OCDF	ND/EDL=0.44	EMPC=0.49	0.63
Percent Lipids	2.84	2.57	2.92

Notes:

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

SUMMARY OF ANALYTICAL RESULTS

Homologue Group Totals

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	03MAR92LCB4041	03MAR92LCB4021	03MAR92LCB4031
Keystone/NEA Number:	91TT26SP01-MB	91TT27SP02-MB	91TT05OC01-MB
Customer Number:			
Sample Description:	Method Blank	Method Blank	Method Blank
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	3.56	0.70	3.01
Total PeCDD	3.28	ND/EDL=0.29	ND/EDL=0.29
Total HxCDD	3.90	ND/EDL=0.13	ND/EDL=0.07
Total HpCDD	1.24	0.30	ND/EDL=0.30
Furans			
Total TCDF	0.38	ND/EDL=0.16	0.85
Total PeCDF	ND/EDL=0.24	ND/EDL=0.20	ND/EDL=0.08
Total HxCDF	0.43	ND/EDL=0.34	0.26
Total HpCDF	ND/EDL=0.30	ND/EDL=0.10	ND/EDL=0.18

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. Concentrations below the LMCL or above the UMCL are ESTIMATES ONLY.

SUMMARY OF ANALYTICAL RESULTS

Homologue Group Totals

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	05MAR92LCB3171	03MAR92LCB4051	03MAR92LCB4061
Keystone/NEA Number:	91TT26SP01-02	91TT27SP02-01	91TT27SP02-03
Customer Number:	D35	D28	D38
Sample Description:	Crayfish	Crayfish	Crayfish
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	5.96	1.79	0.37
Total PeCDD	5.16	ND/EDL=0.32	ND/EDL=0.27
Total HxCDD	6.14	1.13	ND/EDL=0.24
Total HpCDD	3.80	18.6	ND/EDL=0.32
Furans			
Total TCDF	10.2	18.5	6.33
Total PeCDF	1.79	2.99	0.59
Total HxCDF	1.72	49.1	ND/EDL=0.40
Total HpCDF	ND/EDL=0.07	0.72	ND/EDL=0.24

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. Concentrations below the LMCL or above the UMCL are ESTIMATES ONLY.

SUMMARY OF ANALYTICAL RESULTS

Homologue Group Totals

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	03MAR92LCB4071	03MAR92LCB4081	03MAR92LCB4091
Keystone/NEA Number:	91TT27SP02-04	91TT05OC01-01	91TT05OC01-02
Customer Number:	D40	D6	D8
Sample Description:	Crayfish	Crayfish	Crayfish
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	1.06	1.58	0.45
Total PeCDD	ND/EDL=0.22	ND/EDL=0.19	ND/EDL=0.18
Total HxCDD	ND/EDL=0.18	ND/EDL=0.16	ND/EDL=0.07
Total HpCDD	0.62	0.58	0.58
Furans			
Total TCDF	6.84	6.74	5.35
Total PeCDF	0.26	0.23	0.26
Total HxCDF	ND/EDL=0.31	ND/EDL=0.27	ND/EDL=0.22
Total HpCDF	ND/EDL=0.09	ND/EDL=0.13	ND/EDL=0.10

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. Concentrations below the LMCL or above the UMCL are ESTIMATES ONLY.

SUMMARY OF ANALYTICAL RESULTS

Homologue Group Totals

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	03MAR92LCB4111	03MAR92LCB4121	03MAR92LCB4131
Keystone/NEA Number:	91TT05OC01-03	91TT05OC01-05	91TT05OC01-07
Customer Number:	D10	D15	D19
Sample Description:	Crayfish	Crayfish	Crayfish
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	3.05	2.38	3.25
Total PeCDD	ND/EDL=0.17	ND/EDL=0.14	ND/EDL=0.66
Total HxCDD	ND/EDL=0.12	ND/EDL=0.07	0.33
Total HpCDD	2.85	ND/EDL=0.06	2.87
Furans			
Total TCDF	7.62	5.80	16.2
Total PeCDF	0.30	0.44	12.1
Total HxCDF	1.01	0.34	2.48
Total HpCDF	0.37	ND/EDL=0.16	ND/EDL=0.09

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. Concentrations below the LMCL or above the UMCL are ESTIMATES ONLY.

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	03MAR92LCB4141	03MAR92LCB4151	05MAR92LCB3201
Keystone/NEA Number:	91TT05OC01-08	91TT05OC01-10	91TT05OC01-11
Customer Number:	D20	D23	D24
Sample Description:	Crayfish	Crayfish	Crayfish
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	0.89	0.93	6.98
Total PeCDD	ND/EDL=0.09	ND/EDL=0.20	6.26
Total HxCDD	0.23	0.30	8.14
Total HpCDD	0.47	0.71	7.62
Furans			
Total TCDF	6.62	5.66	14.2
Total PeCDF	1.07	1.12	7.45
Total HxCDF	0.37	0.78	5.11
Total HpCDF	ND/EDL=0.13	ND/EDL=0.27	1.17

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. Concentrations below the LMCL or above the UMCL are ESTIMATES ONLY.

SUMMARY OF ANALYTICAL RESULTS

Internal Standard Recoveries

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	03MAR92LCB4041	03MAR92LCB4021	03MAR92LCB4031
Keystone/NEA Number:	91TT26SP01-MB	91TT27SP02-MB	91TT05OC01-MB
Customer Number:			
Sample Description:	Method Blank	Method Blank	Method Blank
Units	%	%	%
Dioxins			
13C-2378-TCDD	57	60	63
13C-12378-PeCDD	70	68	71
13C-123478-HxCDD	56	59	67
13C-123678-HxCDD	67	87	98
13C-1234678-HpCDD	51	57	48
13C-OCDD	42	40	37
Furans			
13C-2378-TCDF	64	70	69
13C-12378-PeCDF	54	59	58
13C-23478-PeCDF	55	58	57
13C-123478-HxCDF	59	66	69
13C-123678-HxCDF	67	82	68
13C-234678-HxCDF	60	66	66
13C-123789-HxCDF	55	64	56
13C-1234678-HpCDF	44	57	49
13C-1234789-HpCDF	42	52	48
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	70	66	76

Notes:

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

SUMMARY OF ANALYTICAL RESULTS

Internal Standard Recoveries

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	05MAR92LCB3171	03MAR92LCB4051	03MAR92LCB4061
Keystone/NEA Number:	91TT26SP01-02	91TT27SP02-01	91TT27SP02-03
Customer Number:	D35	D28	D38
Sample Description:	Crayfish	Crayfish	Crayfish
Units	%	%	%
Dioxins			
13C-2378-TCDD	65	57	50
13C-12378-PeCDD	87	71	62
13C-123478-HxCDD	80	55	49
13C-123678-HxCDD	80	98	71
13C-1234789-HpCDD	80	66	58
13C-OCDD	68	71	58
Furans			
13C-2378-TCDF	84*	76*	61*
13C-12378-PeCDF	62	56	51
13C-23478-PeCDF	63	56	50
13C-123478-HxCDF	82	62	55
13C-123678-HxCDF	63	23	60
13C-234678-HxCDF	65	55	56
13C-123789-HxCDF	74	54	53
13C-1234678-HpCDF	69	62	55
13C-1234789-HpCDF	82	69	55
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	82	73	68

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	03MAR92LCB4071	03MAR92LCB4081	03MAR92LCB4091
Keystone/NEA Number:	91TT27SP02-04	91TT05OC01-01	91TT05OC01-02
Customer Number:	D40	D6	D8
Sample Description:	Crayfish	Crayfish	Crayfish
Units	%	%	%
Dioxins			
13C-2378-TCDD	53	64	62
13C-12378-PeCDD	67	81	78
13C-123478-HxCDD	61	83	60
13C-123678-HxCDD	69	98	81
13C-1234789-HpCDD	62	78	74
13C-OCDD	70	83	67
Furans			
13C-2378-TCDF	67*	79*	76*
13C-12378-PeCDF	56	64	65
13C-23478-PeCDF	54	65	64
13C-123478-HxCDF	67	71	66
13C-123678-HxCDF	59	68	65
13C-234678-HxCDF	49	68	63
13C-123789-HxCDF	57	68	62
13C-1234678-HpCDF	57	66	63
13C-1234789-HpCDF	60	71	62
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	66	80	80

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

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	03MAR92LCB4111	03MAR92LCB4121	03MAR92LCB4131
Keystone/NEA Number:	91TT05OC01-03	91TT05OC01-05	91TT05OC01-07
Customer Number:	D10	D15	D19
Sample Description:	Crayfish	Crayfish	Crayfish
Units	%	%	%
Dioxins			
13C-2378-TCDD	58	50	67
13C-12378-PeCDD	72	57	57
13C-123478-HxCDD	72	58	68
13C-123678-HxCDD	60	68	79
13C-1234789-HpCDD	56	51	69
13C-OCDD	53	43	62
Furans			
13C-2378-TCDF	66*	62*	84*
13C-12378-PeCDF	57	50	76
13C-23478-PeCDF	58	47	35
13C-123478-HxCDF	59	59	72
13C-123678-HxCDF	60	56	64
13C-234678-HxCDF	58	50	67
13C-123789-HxCDF	59	49	69
13C-1234678-HpCDF	49	45	57
13C-1234789-HpCDF	49	42	57
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	68	62	74

Notes:

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

SUMMARY OF ANALYTICAL RESULTS

Internal Standard Recoveries

Date received: September 26, 27 and October 5, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT26SP01
Customer Project Number: STA. D35

MS File Number:	03MAR92LCB4141	03MAR92LCB4151	05MAR92LCB3201
Keystone/NEA Number:	91TT05OC01-08	91TT05OC01-10	91TT05OC01-11
Customer Number:	D20	D23	D24
Sample Description:	Crayfish	Crayfish	Crayfish
Units	%	%	%
Dioxins			
13C-2378-TCDD	59	51	64
13C-12378-PeCDD	78	66	83
13C-123478-HxCDD	58	58	83
13C-123678-HxCDD	71	59	77
13C-1234789-HpCDD	54	48	80
13C-OCDD	46	47	67
Furans			
13C-2378-TCDF	75*	66*	88*
13C-12378-PeCDF	61	53	59
13C-23478-PeCDF	59	50	59
13C-123478-HxCDF	62	56	84
13C-123678-HxCDF	57	53	58
13C-234678-HxCDF	51	50	62
13C-123789-HxCDF	57	50	71
13C-1234678-HpCDF	46	40	65
13C-1234789-HpCDF	48	42	80
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	76	67	83

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

SUMMARY OF ANALYTICAL RESULTS

Internal Standard Recoveries

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	05MAR92LCB3181	05MAR92LCB3191
Keystone/NEA Number:	91TT26SP01-02MS	91TT26SP01-02MSd
Customer Number:	D35	D35
Sample Description:	Crayfish	Crayfish
Units	%	%
Dioxins		
13C-2378-TCDD	61	64
13C-12378-PeCDD	83	86
13C-123478-HxCDD	92	93
13C-123678-HxCDD	64	72
13C-1234789-HpCDD	73	82
13C-OCDD	63	71
Furans		
13C-2378-TCDF	73*	74*
13C-12378-PeCDF	58	59
13C-23478-PeCDF	58	58
13C-123478-HxCDF	78	81
13C-123678-HxCDF	54	61
13C-234678-HxCDF	59	60
13C-123789-HxCDF	67	73
13C-1234678-HpCDF	60	66
13C-1234789-HpCDF	73	82
Clean-Up Recovery Standard		
37Cl4-2378-TCDD	79	85

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
Matrix Spike Samples

Date received: September 26, 27 and October 5, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT26SP01
Customer Project Number: STA. D35

MS File Number:	05MAR92LCB3171			05MAR92LCB3181		
Keystone/NEA Number:	91TT26SP01-02			91TT26SP01-02MS		
Sample Description:	Measured Levels	Spiked Levels*	Spiked Levels**	Theoretical Levels	Measured Levels	% Recy.
Units	pg/g (ppt)	pg	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)	%
Dioxins						
2378-TCDD	0.40	200	10.19	10.59	12.9	122
12378-PeCDD	0.48	1079	55.00	55.48	55.2	99
123478-HxCDD	0.15	904	46.08	46.23	53.3	115
123678-HxCDD	0.53	888	45.26	45.79	69.9	153
123789-HxCDD	0.59	783	39.91	40.50	72.4	179
1234678-HpCDD	2.07	1012	51.58	53.65	67.9	127
OCDD	5.72	1909	97.30	103.02	135	131
Furans						
2378-TCDF	4.10	188	9.58	13.68	17.2	126
12378-PeCDF	0.30	931	47.45	47.75	69.7	146
23478-PeCDF	0.48	880	44.85	45.33	70.2	155
123478-HxCDF	0.21	950	48.42	48.63	57.2	118
123678-HxCDF	0.18	934	47.61	47.79	73.4	154
234678-HxCDF	0.48	904	46.08	46.56	64.3	138
123789-HxCDF	0.13	960	48.93	49.06	64.7	132
1234678-HpCDF	0.29	897	45.72	46.01	63.5	138
1234789-HpCDF	0.035	948	48.32	48.36	63.7	132
OCDF	0.42	1842	93.89	94.31	105	111

Notes:
1. Concentrations marked with an asterisk (*) are the absolute amount of each native analyte spiked into the sample -02MS.
2. Concentrations marked with a double asterisk (**) are the spike levels expressed as pg/g (ppt) for a sample weight of 19.619 grams.

SUMMARY OF ANALYTICAL RESULTS

Matrix Spike Samples

Date received: September 26, 27 and October 5, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT26SP01
Customer Project Number: STA. D35

MS File Number:	05MAR92LCB3171			05MAR92LCB3191		
Keystone/NEA Number:	91TT26SP01-02			91TT26SP01-02MSd		
Sample Description:	Measured Levels	Spiked Levels*	Spiked Levels**	Theoretical Levels	Measured Levels	% Recy.
Units	pg/g (ppt)	pg	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)	%
Dioxins						
2378-TCDD	0.40	200	9.71	10.11	11.6	115
12378-PeCDD	0.48	1079	52.40	52.88	51.8	98
123478-HxCDD	0.15	904	43.90	44.05	57.6	131
123678-HxCDD	0.53	888	43.12	43.65	58.6	134
123789-HxCDD	0.59	783	38.02	38.61	61.3	159
1234678-HpCDD	2.07	1012	49.14	51.21	61.2	120
OCDD	5.72	1909	92.70	98.42	123	125
Furans						
2378-TCDF	4.10	188	9.13	13.23	16.3	123
12378-PeCDF	0.30	931	45.21	45.51	65.2	143
23478-PeCDF	0.48	880	42.73	43.21	67.2	156
123478-HxCDF	0.21	950	46.13	46.34	61.2	132
123678-HxCDF	0.18	934	45.36	45.54	63.1	139
234678-HxCDF	0.48	904	43.90	44.38	61.7	139
123789-HxCDF	0.13	960	46.62	46.75	62.5	134
1234678-HpCDF	0.29	897	43.56	43.85	58.7	134
1234789-HpCDF	0.035	948	46.04	46.07	58.2	126
OCDF	0.42	1842	89.45	89.87	95.2	106

Notes:

1. Concentrations marked with an asterisk (*) are the absolute amount of each native analyte spiked into the sample -02MSd.
2. Concentrations marked with a double asterisk (**) are the spike levels expressed as pg/g (ppt) for a sample weight of 20.593 grams.

SUMMARY OF ANALYTICAL RESULTS
Matrix Spike Samples

Date received:	September 26, 27 and October 5, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT26SP01
Customer Project Number:	STA. D35

MS File Number:	05MAR92LCB3181	05MAR92LCB3191	Relative
Keystone/NEA Number:	91TT26SP01-02MS	91TT26SP01-02MSd	Percent
Sample Description:	Crayfish	Crayfish	Difference
Units	pg/g (ppt)	pg/g (ppt)	%
Dioxins			
2378-TCDD	12.9	11.6	-11
12378-PeCDD	55.2	51.8	-6
123478-HxCDD	53.3	57.6	8
123678-HxCDD	69.9	58.6	-18
123789-HxCDD	72.4	61.3	-17
1234678-HpCDD	67.9	61.2	-10
OCDD	135	123	-9
Furans			
2378-TCDF	17.2	16.3	-5
12378-PeCDF	69.7	65.2	-7
23478-PeCDF	70.2	67.2	-4
123478-HxCDF	57.2	61.2	7
123678-HxCDF	73.4	63.1	-15
234678-HxCDF	64.3	61.7	-4
123789-HxCDF	64.7	62.5	-3
1234678-HpCDF	63.5	58.7	-8
1234789-HpCDF	63.7	58.2	-9
OCDF	105	95.2	-10

SECTION C. SUCKERS

ANALYSIS OF SUCKERS

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

Twelve sucker samples were received under Chain-of-Custody between October 16 and November 20, 1991. The samples were in good condition upon receipt, and were stored in a freezer maintained at -21°C. The samples were homogenized by grinding several times, and immediately returned to the freezer where they remained until extraction.

Three laboratory Method Blanks, and one Matrix Spike and Matrix Spike Duplicate were also analyzed with this sample set.

II. ANALYSIS REQUEST

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

LAB ID NUMBER	ANALYSIS	LMCL
91TT16OC01	EPA Method 1613x	0.5 ppt (tetras)
91TT18OC01	EPA Method 1613x	2.5 ppt (pentas, hexas, heptas)
91TT22OC02	EPA Method 1613x	5.0 ppt (octas)
91TT28OC02	EPA Method 1613x	
91TT20NV01	EPA Method 1613x	

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 under "Analytical Methodology", below.

The samples were extracted in four sets on January 14, 15, 16, and February 3, 1992. The extraction set from January 15 (samples D19S, D15S, D6S, D8S, and D10S) was returned to the laboratory for re-extraction on February 9, 1992, because of severe diphenyl ether contamination. The diphenyl ether contamination also made it necessary to run all samples through carbon columns a second time for additional clean-up.

The samples were analyzed on a DB-5 column on March 11, 1992. Confirmation analyses were on a DB-225 column on February 14, 1992.

B. Results

General Sucker results are based on the initial weight of the sample (approximately 20 grams). All results are reported to three significant figures. Laboratory Method Blank results are also based on a theoretical sample size of 20 grams. Reported results (concentrations and recoveries) for the 2378-TCDF are from a DB-225 column, and have been highlighted with an asterisk (*). All other results are from a DB-5 column. Concentrations below the Lower Method Calibration Limit (LMCL) should be considered ESTIMATES ONLY.

Sample Results Only one of the 2378-substituted isomers was consistently present at levels above the Lower Method Calibration Limit (LMCL). The 2378-TCDF isomer was present at concentrations ranging from 2.46 ppt to 11.4 ppt (samples D20S and D38S respectively).

The OCDD isomer was present in several samples at concentrations above the LMCL. The levels detected ranged from 5.25 ppt to 21.3 ppt (samples D23S and D24S, respectively).

Most of the remaining 2378-substituted isomers were either consistently present at trace levels near the LMCL or were Non-Detect. No distinctive pattern is observed in the Homologue Group totals for the twelve samples analyzed.

Diphenyl ether frequently interfered with the 234678-HxCDF isomer. This occurrence has been flagged with an EMPC/D qualifier in the summary tables.

C. Analytical Methodology

The extraction and sample clean-up were done according to EPA Method 8290 guidelines. (Method 1613 does not address extraction of fish tissue.) 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 occurrence 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. The modifications are outlined below.

Clean-Up Recovery Standard Spiking Levels EPA Method 1613 calls for spiking the sample extracts with 800 pg of ^{37}Cl-2378-TCDD immediately prior to the clean-up procedure. That level has been reduced to 200 pg, as suggested by NCASI. The purpose of this change is to reduce the occurrence of false positives due to 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 aqueous matrices.

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 for the continuing 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 EPA Method 8290. (Method 1613 does not specify how these values should be calculated and/or reported, but instead reports only the Lower Method Calibration Limits (LMCL).

D. 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 or above the UMCL, 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) can be calculated based on a sample size of 20 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 sample size of 20 grams:

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

Note: pg/g = ppt

IV. QUALITY CONTROL

A. Project Quality Control

Quality control measures specific to this project included a Matrix Spike and Matrix Spike Duplicate on one of the sucker tissue samples. The project quality control plan specified a Matrix Spike and Matrix Spike Duplicate (MS/MSd) for every 20 samples run. In practice, a minimum of one MS/MSd pair was run with each tissue type (eg. sturgeon, carp, sucker, etc.).

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. 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 Three laboratory method blanks were analyzed with this 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.

The laboratory method blanks 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). Many analytes were present at levels below the LMCL for their particular homologue group. These analyte levels would not normally be reported under method 1613, but are included for your review.

Internal Standard Recoveries In several instances, the percent recovery of Internal Standard is outside the method guidelines of 25%-150% for individual labeled analytes within a sample. In all cases, however, the signal-to-noise ratio for the labeled internal standard exceeds the recommended ratio of 10/1 by a significant margin. Since most of the corresponding unlabeled analytes are either Not Detected, or are present only at levels below the LMCL, their results are not expected to be affected.

Matrix Spike Samples Tables 5a-5c in the "SAMPLE ANALYSIS SUMMARY" section of this report lists the results for the Matrix Spike and Matrix Spike Duplicate. The Relative Percent Difference (RPD) between the duplicate analyses ranged from 1% to 70%.

The Matrix Spike Duplicate sample was subject to unusual chemical contamination in all five homologue groups during the first analysis. The extract of that sample was run through an additional carbon column to remove the interferences, and analyzed a second time. The analyte concentrations and percent recoveries for that sample have been calculated, but should be considered ESTIMATES ONLY, due to the interference.

D. Quality Control Review

All of the data have 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 4/1/92
Laura Chambers Date
Sr. Scientist
Center for Analytical Mass Spectrometry

Peggy Meek 4/1/92
Peggy L. Meek Date
Wet Lab Supervisor
Center for Analytical Mass Spectrometry

4.1.92
William H. Chambers Date
Director
Center for Analytical Mass Spectrometry

J. Sprenger 4/1/92
Jeff Sprenger Date
QA Officer
Keystone/NEA

KEYSTONE/NEA
ENVIRONMENTAL RESOURCES

SAMPLE ANALYSIS SUMMARY

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR92LCB3031	11MAR92LCB3021	11MAR92LCB3041
Keystone/NEA Number:	91TT16OC01-MB2	91TT18OC01-MB2	91TT28OC02-MB1RX
Customer Number:			
Sample Description:	Method Blank	Method Blank	Method Blank
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	ND/EDL=0.08	ND/EDL=0.07	0.06
12378-PeCDD	EMPC=0.29	0.25	EMPC=0.24
123478-HxCDD	EMPC=0.16	0.17	0.12
123678-HxCDD	0.45	0.33	EMPC=0.47
123789-HxCDD	EMPC=0.28	EMPC=0.30	0.33
1234678-HpCDD	EMPC=0.85	0.66	1.16
OCDD	EMPC=1.73	2.12	1.65
Furans			
2378-TCDF	ND/EDL=0.08	ND/EDL=0.10	0.29
12378-PeCDF	EMPC=0.28	0.31	0.15
23478-PeCDF	EMPC=0.32	EMPC=0.25	0.27
123478-HxCDF	0.30	0.30	0.21
123678-HxCDF	EMPC=0.39	0.30	0.22
234678-HxCDF	0.61	EMPC=0.59	EMPC=0.49
123789-HxCDF	0.39	EMPC=0.40	EMPC=0.18
1234678-HpCDF	0.34	EMPC=0.32	EMPC=0.29
1234789-HpCDF	0.34	0.25	EMPC=0.11
OCDF	EMPC=0.62	0.77	EMPC=0.27
Percent Lipids	N.A.	N.A.	N.A.

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR92LCB3091	11MAR92LCB3101	11MAR92LCB3111
Keystone/NEA Number:	91TT16OC01-01	91TT16OC01-02	91TT16OC01-05
Customer Number:	D35S	D38S	D40S
Sample Description:	Sucker	Sucker	Sucker
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	0.62	1.38	0.72
12378-PeCDD	EMPC=0.40	EMPC=0.72	EMPC=0.48
123478-HxCDD	0.20	0.33	EMPC=0.17
123678-HxCDD	0.18	0.81	0.41
123789-HxCDD	0.11	0.38	EMPC=0.32
1234678-HpCDD	1.04	2.41	1.82
OCDD	3.79	4.12	0.79
Furans			
2378-TCDF	7.09*	11.4*	11.0*
12378-PeCDF	0.18	EMPC=0.23	EMPC=0.16
23478-PeCDF	0.31	0.72	EMPC=0.45
123478-HxCDF	0.08	EMPC=0.27	0.09
123678-HxCDF	0.16	0.36	EMPC=0.15
234678-HxCDF	EMPC/D=1.61	EMPC/D=2.69	EMPC/D=2.77
123789-HxCDF	0.11	EMPC=0.18	EMPC=0.17
1234678-HpCDF	0.90	EMPC/D=1.79	EMPC=0.30
1234789-HpCDF	0.10	0.15	EMPC=0.11
OCDF	EMPC=0.35	0.69	10.6
Percent Lipids	3.4%	9.2%	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 highlighted with an asterisk (*) are from a DB-225 column.
4. Concentrations below the LMCL or above the UMCL are ESTIMATES ONLY.

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR92LCB3051	11MAR92LCB3061	11MAR92LCB3071
Keystone/NEA Number:	91TT18OC01-05	91TT22OC02-08	91TT22OC02-10
Customer Number:	D28S	D24S	D23S
Sample Description:	Sucker	Sucker	Sucker
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	1.41	1.01	0.92
12378-PeCDD	EMPC=0.90	EMPC=0.58	EMPC=0.43
123478-HxCDD	0.35	0.22	EMPC=0.13
123678-HxCDD	1.42	0.65	0.44
123789-HxCDD	0.36	0.28	0.19
1234678-HpCDD	4.36	3.11	1.10
OCDD	20.1	21.3	5.25
Furans			
2378-TCDF	6.98*	7.24*	6.36*
12378-PeCDF	0.42	EMPC=0.28	0.16
23478-PeCDF	0.92	0.50	0.38
123478-HxCDF	0.45	EMPC=0.22	EMPC=0.13
123678-HxCDF	0.25	0.18	EMPC=0.11
234678-HxCDF	EMPC=1.50	EMPC=0.54	EMPC=0.49
123789-HxCDF	0.33	0.17	0.09
1234678-HpCDF	0.70	0.55	EMPC=0.23
1234789-HpCDF	0.30	EMPC=0.15	0.09
OCDF	3.07	1.76	0.56
Percent Lipids	8.2%	8.1%	6.7%

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR92LCB3141	11MAR92LCB3151	11MAR92LCB3161
Keystone/NEA Number:	91TT28OC02-01RX	91TT28OC02-02RX	91TT28OC02-03RX
Customer Number:	D19S	D15S	D6S
Sample Description:	Sucker	Sucker	Sucker
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	1.32	0.88	0.49
12378-PeCDD	EMPC=0.64	EMPC=0.51	EMPC=0.46
123478-HxCDD	0.23	EMPC=0.19	0.18
123678-HxCDD	0.87	0.74	EMPC=0.68
123789-HxCDD	0.48	0.42	0.43
1234678-HpCDD	2.98	2.45	2.07
OCDD	9.28	6.43	4.04
Furans			
2378-TCDF	8.79*	4.69*	5.24*
12378-PeCDF	EMPC=0.34	0.20	0.18
23478-PeCDF	0.69	0.48	0.43
123478-HxCDF	0.27	0.20	0.18
123678-HxCDF	0.22	0.22	0.23
234678-HxCDF	EMPC/D=1.41	EMPC/D=1.65	EMPC/D=1.35
123789-HxCDF	0.18	EMPC=0.12	0.13
1234678-HpCDF	EMPC/D=1.05	EMPC/D=1.03	EMPC/D=0.29
1234789-HpCDF	0.13	EMPC=0.10	0.06
OCDF	1.03	0.47	0.30
Percent Lipids	7.6%	5.4%	5.3%

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR92LCB3171	20MAR92LCB2201	11MAR92LCB3081
Keystone/NEA Number:	91TT28OC02-04RX	91TT28OC02-13RX	91TT20NV01-01
Customer Number:	D8S	D10S	D20S
Sample Description:	Sucker	Sucker	Sucker
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	0.82	1.56	0.76
12378-PeCDD	EMPC=0.65	EMPC=1.10	EMPC=0.40
123478-HxCDD	0.23	0.53	EMPC=0.13
123678-HxCDD	0.97	1.01	EMPC=0.33
123789-HxCDD	0.45	0.92	0.16
1234678-HpCDD	2.44	3.35	1.66
OCDD	4.41	6.67	13.7
Furans			
2378-TCDF	7.97*	5.45*	EMPC=2.46*
12378-PeCDF	0.23	0.49	0.14
23478-PeCDF	0.52	1.21	0.33
123478-HxCDF	0.21	0.39	0.14
123678-HxCDF	EMPC=0.21	EMPC=0.33	0.09
234678-HxCDF	EMPC=2.17	0.78	0.40
123789-HxCDF	0.14	0.60	EMPC=0.09
1234678-HpCDF	0.36	0.85	EMPC=0.33
1234789-HpCDF	EMPC=0.08	0.43	EMPC=0.12
OCDF	0.35	1.20	1.44
Percent Lipids	7.1%	9.7%	3.4%

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR92LCB3031	11MAR92LCB3021	11MAR92LCB3041
Keystone/NEA Number:	91TT16OC01-MB2	91TT18OC01-MB2	91TT28OC02-MB1RX
Customer Number:			
Sample Description:	Method Blank	Method Blank	Method Blank
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	1.80	0.89	1.84
Total PeCDD	1.72	0.60	3.38
Total HxCDD	2.19	1.05	3.8
Total HpCDD	0.61	0.66	2.08
Furans			
Total TCDF	0.76	0.15	2.07
Total PeCDF	ND/EDL=0.14	0.30	1.16
Total HxCDF	1.29	0.77	0.52
Total HpCDF	0.68	0.25	ND/EDL=0.03

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. Concentrations below the LMCL or above the UMCL are ESTIMATES ONLY.

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR92LCB3091	11MAR92LCB3101	11MAR92LCB3111
Keystone/NEA Number:	91TT16OC01-01	91TT16OC01-02	91TT16OC01-05
Customer Number:	D35S	D38S	D40S
Sample Description:	Sucker	Sucker	Sucker
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	2.15	0.30	2.35
Total PeCDD	3.15	1.65	1.98
Total HxCDD	1.12	3.61	2.78
Total HpCDD	1.83	3.28	4.38
Furans			
Total TCDF	6.41	16.8	12.9
Total PeCDF	1.13	2.51	1.18
Total HxCDF	1.17	1.89	0.95
Total HpCDF	1.53	1.94	2.52

Notes:
1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. Concentrations below the LMCL or above the UMCL are ESTIMATES ONLY.

SUMMARY OF ANALYTICAL RESULTS

Homologue Group Totals

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR92LCB3051	11MAR92LCB3061	11MAR92LCB3071
Keystone/NEA Number:	91TT18OC01-05	91TT22OC02-08	91TT22OC02-10
Customer Number:	D28S	D24S	D23S
Sample Description:	Sucker	Sucker	Sucker
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	3.5	3.36	2.69
Total PeCDD	ND/EDL=0.11	0.85	0.26
Total HxCDD	3.63	2.23	1.54
Total HpCDD	7.30	5.74	1.90
Furans			
Total TCDF	7.95	7.34	6.07
Total PeCDF	2.15	1.14	0.76
Total HxCDF	2.71	1.21	0.38
Total HpCDF	2.23	1.53	0.22

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. Concentrations below the LMCL or above the UMCL are ESTIMATES ONLY.

SUMMARY OF ANALYTICAL RESULTS

Homologue Group Totals

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR92LCB3141	11MAR92LCB3151	11MAR92LCB3161
Keystone/NEA Number:	91TT28OC02-01RX	91TT28OC02-02RX	91TT28OC02-03RX
Customer Number:	D19S	D15S	D6S
Sample Description:	Sucker	Sucker	Sucker
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	3.79	2.65	2.71
Total PeCDD	5.06	4.50	4.86
Total HxCDD	7.65	7.42	7.01
Total HpCDD	5.48	4.53	3.85
Furans			
Total TCDF	8.69	6.00	6.03
Total PeCDF	2.44	2.56	2.47
Total HxCDF	1.21	0.93	0.90
Total HpCDF	0.89	0.26	0.45

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. Concentrations below the LMCL or above the UMCL are ESTIMATES ONLY.

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR92LCB3171	20MAR92LCB2201	11MAR92LCB3081
Keystone/NEA Number:	91TT28OC02-04RX	91TT28OC02-13RX	91TT20NV01-01
Customer Number:	D8S	D10S	D20
Sample Description:	Sucker	Sucker	Sucker
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	3.24	52.6	3.11
Total PeCDD	4.90	7.22	0.18
Total HxCDD	8.03	10.2	1.36
Total HpCDD	4.33	5.76	2.66
Furans			
Total TCDF	7.97	7.82	3.51
Total PeCDF	2.69	4.22	0.47
Total HxCDF	0.36	2.25	0.61
Total HpCDF	0.92	1.23	0.41

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. Concentrations below the LMCL or above the UMCL are ESTIMATES ONLY.

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR92LCB3031	11MAR92LCB3021	11MAR92LCB3041
Keystone/NEA Number:	91TT16OC01-MB2	91TT18OC01-MB2	91TT28OC02-MB1RX
Customer Number:			
Sample Description:	Method Blank	Method Blank	Method Blank
Units	%	%	%
Dioxins			
13C-2378-TCDD	29	29	59
13C-12378-PeCDD	35	38	71
13C-123478-HxCDD	31	31	65
13C-123678-HxCDD	36	39	72
13C-1234678-HpCDD	34	34	68
13C-OCDD	29	22	53
Furans			
13C-2378-TCDF	30	31	62
13C-12378-PeCDF	27	28	54
13C-23478-PeCDF	27	28	56
13C-123478-HxCDF	32	32	62
13C-123678-HxCDF	29	31	61
13C-234678-HxCDF	24	23	53
13C-123789-HxCDF	32	30	63
13C-1234678-HpCDF	25	25	49
13C-1234789-HpCDF	33	32	66
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	41	37	84

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR92LCB3091	11MAR92LCB3101	11MAR92LCB3111
Keystone/NEA Number:	91TT16OC01-01	91TT16OC01-02	91TT16OC01-05
Customer Number:	D35S	D38S	D40S
Sample Description:	Sucker	Sucker	Sucker
Units	%	%	%
Dioxins			
13C-2378-TCDD	35	39	23
13C-12378-PeCDD	23	52	31
13C-123478-HxCDD	42	47	30
13C-123678-HxCDD	97	47	32
13C-1234789-HpCDD	46	50	29
13C-OCDD	39	43	21
Furans			
13C-2378-TCDF	38*	44*	27*
13C-12378-PeCDF	34	37	22
13C-23478-PeCDF	35	38	23
13C-123478-HxCDF	39	44	31
13C-123678-HxCDF	36	41	27
13C-234678-HxCDF	31	37	21
13C-123789-HxCDF	40	44	24
13C-1234678-HpCDF	37	39	22
13C-1234789-HpCDF	45	50	28
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	47	52	31

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

SUMMARY OF ANALYTICAL RESULTS

Internal Standard Recoveries

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR92LCB3051	11MAR92LCB3061	11MAR92LCB3071
Keystone/NEA Number:	91TT18OC01-05	91TT22OC02-08	91TT22OC02-10
Customer Number:	D28S	D24S	C23S
Sample Description:	Sucker	Sucker	Sucker
Units	%	%	%
Dioxins			
13C-2378-TCDD	26	28	27
13C-12378-PeCDD	36	37	35
13C-123478-HxCDD	30	31	30
13C-123678-HxCDD	34	35	34
13C-1234789-HpCDD	31	33	33
13C-OCDD	13	20	19
Furans			
13C-2378-TCDF	32*	31*	31*
13C-12378-PeCDF	26	27	26
13C-23478-PeCDF	27	28	27
13C-123478-HxCDF	29	29	30
13C-123678-HxCDF	28	29	27
13C-234678-HxCDF	5	13	12
13C-123789-HxCDF	28	31	30
13C-1234678-HpCDF	25	26	26
13C-1234789-HpCDF	31	34	34
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	34	43	41

Notes:

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

SUMMARY OF ANALYTICAL RESULTS

Internal Standard Recoveries

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR92LCB3141	11MAR92LCB3151	11MAR92LCB3161
Keystone/NEA Number:	91TT28OC02-01RX	91TT28OC02-02RX	91TT28OC02-03RX
Customer Number:	D19S	D15S	D6S
Sample Description:	Sucker	Sucker	Sucker
Units	%	%	%
Dioxins			
13C-2378-TCDD	64	69	66
13C-12378-PeCDD	78	87	84
13C-123478-HxCDD	67	73	75
13C-123678-HxCDD	73	75	78
13C-1234789-HpCDD	65	74	79
13C-OCDD	48	59	65
Furans			
13C-2378-TCDF	72*	76*	75*
13C-12378-PeCDF	59	64	64
13C-23478-PeCDF	61	65	65
13C-123478-HxCDF	66	70	70
13C-123678-HxCDF	61	60	61
13C-234678-HxCDF	50	51	53
13C-123789-HxCDF	64	68	68
13C-1234678-HpCDF	57	64	67
13C-1234789-HpCDF	67	77	80
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	84	86	90

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR92LCB3171	20MAR92LCB2201	11MAR92LCB3081
Keystone/NEA Number:	91TT28OC02-04RX	91TT28OC02-13RX	91TT20NV01-01
Customer Number:	D8S	D10S	D20
Sample Description:	Sucker	Sucker	Sucker
Units	%	pg/g (ppt)	%
Dioxins			
13C-2378-TCDD	61	83	28
13C-12378-PeCDD	79	85	34
13C-123478-HxCDD	70	64	32
13C-123678-HxCDD	70	63	33
13C-1234789-HpCDD	73	73	33
13C-OCDD	64	47	21
Furans			
13C-2378-TCDF	71*	60*	34*
13C-12378-PeCDF	56	89	27
13C-23478-PeCDF	57	94	27
13C-123478-HxCDF	60	68	28
13C-123678-HxCDF	59	55	28
13C-234678-HxCDF	47	54	15
13C-123789-HxCDF	62	68	29
13C-1234678-HpCDF	61	62	25
13C-1234789-HpCDF	71	86	34
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	88	79	41

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

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	October 16,18, 22, 28 and November 20, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16-28OC01, 91TT20NV01
Customer Project Number:	8526-06

MS File Number:	11MAR91LCB3121	28MAR92LCB3011
Keystone/NEA Number:	91TT16OC01-02MS	91TT16OC01-02MSd
Customer Number:	D38S	D38S
Sample Description:	Sucker	Sucker
Units	%	%
Dioxins		
13C-2378-TCDD	43	51
13C-12378-PeCDD	57	63
13C-123478-HxCDD	55	40
13C-123678-HxCDD	50	54
13C-1234789-HpCDD	55	15
13C-OCDD	45	6
Furans		
13C-2378-TCDF	48*	63*
13C-12378-PeCDF	42	39
13C-23478-PeCDF	43	33
13C-123478-HxCDF	50	25
13C-123678-HxCDF	46	31
13C-234678-HxCDF	38	23
13C-123789-HxCDF	49	6
13C-1234678-HpCDF	40	12
13C-1234789-HpCDF	55	8
Clean-Up Recovery Standard		
37Cl4-2378-TCDD	65	93

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
Matrix Spike Samples

Date received: September 26, 27 and October 5, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT26SP01
Customer Project Number: STA. D35

MS File Number:	11MAR91LCB3101			11MAR91LCB3121		
Keystone/NEA Number:	91TT16OC01-02			91TT16OC01-02MS		
Sample Description:	Measured Levels	Spiked Levels*	Spiked Levels**	Theoretical Levels	Measured Levels	% Recy.
Units	pg/g (ppt)	pg	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)	%
Dioxins						
2378-TCDD	1.38	200	9.97	11.35	13.0	114
12378-PeCDD	0.72	1079	53.81	54.53	52.5	96
123478-HxCDD	0.33	904	45.09	45.42	51.8	114
123678-HxCDD	0.81	888	44.29	45.10	66.4	147
123789-HxCDD	0.38	783	39.05	39.43	40.2	102
1234678-HpCDD	2.41	1012	50.47	52.88	61.2	116
OCDD	4.12	1909	95.21	99.33	123	124
Furans						
2378-TCDF	11.4	188	9.38	20.78	25.7	124
12378-PeCDF	0.23	931	46.43	46.66	65.2	140
23478-PeCDF	0.72	880	43.89	44.61	62.4	140
123478-HxCDF	0.27	950	47.38	47.65	76.3	160
123678-HxCDF	0.36	934	46.58	46.94	75.6	161
234678-HxCDF	2.69	904	45.09	47.78	77.5	162
123789-HxCDF	0.18	960	47.88	48.06	71.4	149
1234678-HpCDF	1.79	897	44.74	46.53	61.3	132
1234789-HpCDF	0.15	948	47.28	47.43	57.7	122
OCDF	0.69	1842	91.87	92.56	112	121

Notes:
1. Concentrations marked with an asterisk (*) are the absolute amount of each native analyte spiked into the sample -02MS.
2. Concentrations marked with a double asterisk (**) are the spike levels expressed as pg/g (ppt) for a sample weight of 20.051 grams.

SUMMARY OF ANALYTICAL RESULTS

Matrix Spike Samples

Date received: September 26, 27 and October 5, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT26SP01
Customer Project Number: STA. D35

MS File Number:	11MAR91LCB3101			31MAR92LCB2071		
Keystone/NEA Number:	91TT26SP01-02			91TT16OC01-02MSd		
Sample Description:	Measured Levels	Spiked Levels*	Spiked Levels**	Theoretical Levels (See Note #3.)	Measured Levels	% Recy.
Units	pg/g (ppt)	pg	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)	%
Dioxins						
2378-TCDD	1.38	200	9.67	11.05	19.4	176
12378-PeCDD	0.72	1079	52.18	52.90	65.7	124
123478-HxCDD	0.33	904	43.71	44.04	54.6	124
123678-HxCDD	0.81	888	42.94	43.75	74.4	170
123789-HxCDD	0.38	783	37.86	38.24	66.6	174
1234678-HpCDD	2.41	1012	48.94	51.35	82.7	161
OCDD	4.12	1909	92.31	96.43	256	265
Furans						
2378-TCDF	11.4	188	9.09	20.49	23.8	116
12378-PeCDF	0.23	931	45.02	45.25	65.5	145
23478-PeCDF	0.72	880	42.55	43.27	80.2	185
123478-HxCDF	0.27	950	45.94	46.21	75.4	163
123678-HxCDF	0.36	934	45.16	45.52	74.8	164
234678-HxCDF	2.69	904	43.71	46.40	87.7	189
123789-HxCDF	0.18	960	46.42	46.60	79.9	171
1234678-HpCDF	1.79	897	43.38	45.17	102	226
1234789-HpCDF	0.15	948	45.84	45.99	67.5	147
OCDF	0.69	1842	89.07	89.76	139	155

Notes:

1. Concentrations marked with an asterisk (*) are the absolute amount of each native analyte spiked into the sample -02MSd.
2. Concentrations marked with a double asterisk (**) are the spike levels expressed as pg/g (ppt) for a sample weight of 20.680 grams.
3. The Matrix Spike Duplicate sample was subject to unusual chemical contamination in all five groups. Reported concentrations should be considered ESTIMATES ONLY.

SUMMARY OF ANALYTICAL RESULTS
Matrix Spike Samples

Date received: September 26, 27 and October 5, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT26SP01
Customer Project Number: STA. D35

MS File Number:	11MAR91LCB3121	11MAR91LCB3131	Relative
Keystone/NEA Number:	91TT16OC01-02MS	91TT16OC01-02MSd	Percent
Sample Description:	Sucker	Sucker	Difference
Units	pg/g (ppt)	pg/g (ppt)	%
Dioxins			
2378-TCDD	13.0	19.4	40
12378-PeCDD	52.5	65.7	22
123478-HxCDD	51.8	54.6	5
123678-HxCDD	66.4	74.4	11
123789-HxCDD	40.2	66.6	49
1234678-HpCDD	61.2	82.7	30
OCDD	123	256	70
Furans			
2378-TCDF	25.7	23.8	-8
12378-PeCDF	65.2	65.5	0
23478-PeCDF	62.4	80.2	25
123478-HxCDF	76.3	75.4	-1
123678-HxCDF	75.6	74.8	-1
234678-HxCDF	77.5	87.7	12
123789-HxCDF	71.4	79.9	11
1234678-HpCDF	61.3	102	50
1234789-HpCDF	57.7	67.5	16
OCDF	112	139	22

SECTION D. CARP

ANALYSIS OF CARP

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

Five carp samples were received for PCDD/PCDF analysis under Chain-of-Custody on October 16, 18, and 22, 1991. The samples were in good condition upon receipt, and were stored in a freezer maintained at -21°C until analysis. The samples were thawed slightly, ground, then immediately returned to the freezer. The ground tissues were extracted in two sets on January 10 and February 5, 1992, and analyzed on a DB-5 column on February 27 and 29, 1992. Confirmation analyses were on a DB-225 column on February 16, 1992.

Two laboratory method blanks and a matrix spike and matrix spike duplicate were also analyzed with this sample set.

II. ANALYSIS REQUEST

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

LAB ID NUMBER	ANALYSIS	DETECTION LIMIT
91TT16OC01	EPA Method 1613x	1.0 ppt (tetras)
91TT18OC01	EPA Method 1613x	1.0 ppt (tetras)
91TT22OC02	EPA Method 1613x	1.0 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 and sample clean-up followed general guidelines outlined in EPA Method 8290. (Method 1613 does not cover extraction of fish tissue.) 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 occurrence 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. The modifications are outlined below:

Clean-Up Recovery Standard Spiking Levels EPA Method 1613 calls for spiking the sample extracts with 800 pg of ^{37}Cl-2378-TCDD immediately prior to the clean-up procedure. That level has been reduced to 200 pg, as suggested by NCASI. The purpose of this change is to reduce the occurrence of false positives due to 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.

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 or above the UMCL, 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) can be calculated based on a sample size of 20 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 sample size of 20 grams:

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

Note: pg/g = ppt and pg/L = ppq

D. Results

General Carp results are based on the initial weight of the sample (approximately 20 grams). All results are reported to three significant figures. Laboratory Method Blank results are also based on a theoretical sample size of 20 grams. Reported results (concentrations and recoveries) for the 2378-TCDF are from a DB-225 column, and have been highlighted with an asterisk (*). All other results are from a DB-5 column.

Sample Results The 2378-TCDD was present at concentrations ranging from 1.28 ppt (sample D38C), to 2.10 ppt (sample D40C). The 2378-TCDF was present at concentrations ranging from 4.37 ppt (sample D24C) to 12.2 ppt (sample D40C). Two samples contained OCDD at concentrations that differed significantly from the method blank. Samples D28C and D24C contained 30.6 ppt and 20.1 ppt OCDD, respectively.

Most of the remaining 2378-substituted isomers were either consistently present at trace levels below or near the Lower Method Calibration Limit or were Non-Detect. (See Tables 1a - 1c.)

Samples D28C and D24C contained significant levels of chlorinated diphenyl ether (DPE) contamination that could not be removed even after additional clean-up procedures. The 2378-TCDF concentrations are from a DB-225 capillary column and are not affected. The remaining 2378-substituted furans which are affected by co-eluting diphenyl ethers are coded "EMPC/D". Non-2378-substituted furans which had significant contributions from the DPEs are not included in the "Homologue Group Totals". The dioxin concentrations are not affected.

The Relative Percent Differences between laboratory duplicate analyses were within method requirements (25%) for all of the analytes tested.

The percent lipids reported for sample D38C is 1.5%, which is the percentage determination for that specific analysis. That tissue sample was also used for Matrix Spike and Matrix Spike Duplicate samples, and the average percent lipids for the three aliquots of D38C was 3.3%. The difference is likely due to inhomogeniety of the sample.

IV. QUALITY CONTROL

A. Project Quality Control

No special quality control measures specific to this project were required or requested.

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. Documentation of initial and continuing calibrations, and GC and MS resolution checks can be found in the "QUALITY CONTROL DOCUMENTS" section of this report.

Continuing Calibration The Continuing Calibration Standard (ConCal) was analyzed prior to and after this analysis set. The ConCal following this analysis set showed percent deviations above the recommended limits for the ^{13}C-Labeled Internal Standards for the TCDF, PeCDF, and PeCDD groups. These deviations would affect the accuracy of the calculated recoveries of the ^{13}C-Labeled Internal Standards in the samples.

Examination, however, of the ^{13}C-Labeled Internal Standards in sample -06RX, when calculated as a ConCal, shows these same three groups to be well within the the tolerances of the method at the time their data was acquired. The "% Deviation" for the ^{13}C-2378-TCDF, for example, is calculated to be -1%, while the ^{13}C-12378-PeCDF and ^{13}C-12378-PeCDD are calculated to be -1% and +4%, respectively. This indicates that the deviations exhibited by the following ConCal occurred as a result of drift following analysis of these samples.

An alternative approach would be to use the average of the beginning and ending ConCal RRF's to calculate the specifically affected analytes. However, because of the calculations discussed above, this approach is believed to be less accurate.

C. Laboratory Quality Control

Laboratory Method Blank Two method blanks were analyzed with this 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.

The laboratory method blanks were Non-Detect for all PCDD and PCDF isomers at the LMCL of 0.5 ppt (tetras), 1.0 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 0.12 ppt to 0.30 ppt. Nine analytes were present in one or both method blanks at levels below the LMCL, and would not normally be reported under Method 1613, but are included for your review.

D. Quality Control Review

All of the data have 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 3/10/92

Laura Chambers Date
Sr. Scientist
Center for Analytical Mass Spectrometry

For Peggy L. Meek,

3-10-92

Peggy L. Meek Date
Wet Lab Supervisor
Center for Analytical Mass Spectrometry

3.10.92

William H. Chambers Date
Director
Center for Analytical Mass Spectrometry

3/10/92

Jeff Sprenger Date
QA Officer
Keystone/NEA

KEYSTONE/NEA
ENVIRONMENTAL RESOURCES

SAMPLE ANALYSIS SUMMARY

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 16, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16OC02, 91TT18OC01 and 91TT22OC01
Customer Project Number:	8526-06

MS File Number:	27FEB92LCB9051	29FEB92LCB3011	29FEB92LCB3021
Keystone/NEA Number:	91TT16OC01-MB1	91TT16OC01-03	91TT16OC01-04
Customer Number:	8526-06	D35C	D40C
Sample Description:	Method Blank	Carp	Carp
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	ND/EDL=0.14	1.32	2.10
12378-PeCDD	0.19	EMPC=1.11	EMPC=1.68
123478-HxCDD	ND/EDL=0.23	EMPC=0.62	EMPC=0.40
123678-HxCDD	ND/EDL=0.19	EMPC=1.53	1.93
123789-HxCDD	ND/EDL=0.19	EMPC=0.21	EMPC=0.27
1234678-HpCDD	0.78	3.42	4.39
OCDD	10.2	12.3	7.54
Furans			
2378-TCDF	ND/EDL=0.14	9.53*	12.2*
12378-PeCDF	ND/EDL=0.17	0.29	0.39
23478-PeCDF	ND/EDL=0.18	EMPC=0.73	0.96
123478-HxCDF	EMPC=0.14	EMPC=0.23	EMPC=0.19
123678-HxCDF	0.14	0.18	0.16
234678-HxCDF	0.28	EMPC=0.33	EMPC=0.40
123789-HxCDF	ND/EDL=0.28	ND/EDL=0.21	ND/EDL=0.12
1234678-HpCDF	0.21	0.40	EMPC=0.27
1234789-HpCDF	EMPC=0.14	0.12	ND/EDL=0.16
OCDF	1.13	0.84	ND/EDL=0.52
Percent Lipids		3.9	6.9

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 16 and 18, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16OC02, 91TT18OC01 and 91TT22OC01
Customer Project Number:	8526-06

MS File Number:	29FEB92LCB3031	27FEB92LCB9061	29FEB92LCB3061
Keystone/NEA Number:	91TT16OC01-07	91TT18OC01-MBRX	91TT18OC01-06RX
Customer Number:	D38C		D28C
Sample Description:	Carp	Method Blank	Carp
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	1.28	ND/EDL=0.12	1.64
12378-PeCDD	EMPC=0.84	ND/EDL=0.30	EMPC=1.77
123478-HxCDD	0.26	EMPC=0.15	1.18
123678-HxCDD	0.73	0.28	3.73
123789-HxCDD	EMPC=0.12	0.27	ND/EDL=0.36
1234678-HpCDD	1.59	0.60	9.50
OCDD	2.71	1.29	30.6
Furans			
2378-TCDF	7.60*	ND/EDL=0.12	4.89*
12378-PeCDF	0.21	ND/EDL=0.22	EMPC=0.57
23478-PeCDF	0.46	ND/EDL=0.25	1.37
123478-HxCDF	0.12	ND/EDL=0.18	0.52
123678-HxCDF	EMPC=0.09	ND/EDL=0.18	EMPC=0.42
234678-HxCDF	0.26	EMPC=0.36	EMPC/D=3.50
123789-HxCDF	EMPC=0.05	ND/EDL=0.30	ND/EDL=0.34
1234678-HpCDF	EMPC=0.18	EMPC=0.17	1.31
1234789-HpCDF	ND/EDL=0.56	ND/EDL=0.12	ND/EDL=0.18
OCDF	ND/EDL=0.29	ND/EDL=0.30	2.45
Percent Lipids	1.5		2.9

Notes:

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

SUMMARY OF ANALYTICAL RESULTS

2378-Substituted Isomers

Date received:	October 22, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16OC02, 91TT18OC01 and 91TT22OC01
Customer Project Number:	8526-06

MS File Number:	29FEB92LCB3071
Keystone/NEA Number:	91TT22OC01-07RX
Customer Number:	D24C
Sample Description:	Carp

Units	pg/g (ppt)
Dioxins	
2378-TCDD	1.57
12378-PeCDD	EMPC=1.89
123478-HxCDD	EMPC=1.45
123678-HxCDD	4.82
123789-HxCDD	0.50
1234678-HpCDD	9.81
OCDD	20.1
Furans	
2378-TCDF	4.37*
12378-PeCDF	0.76
23478-PeCDF	1.37
123478-HxCDF	0.66
123678-HxCDF	0.57
234678-HxCDF	EMPC/D=5.70
123789-HxCDF	ND/EDL=0.30
1234678-HpCDF	0.75
1234789-HpCDF	ND/EDL=0.11
OCDF	EMPC=0.86
Percent Lipids	6.2

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	October 16, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16OC02, 91TT18OC01 and 91TT22OC01
Customer Project Number:	8526-06

MS File Number:	27FEB92LCB9051	29FEB92LCB3011	29FEB92LCB3021
Keystone/NEA Number:	91TT16OC01-MB1	91TT16OC01-03	91TT16OC01-04
Customer Number:	8526-06	D35C	D40C
Sample Description:	Method Blank	Carp	Carp
Units	pg/g	pg/g	pg/g
Dioxins			
Total TCDD	1.74	2.28	3.76
Total PeCDD	0.19	ND/EDL=0.16	ND/EDL=0.24
Total HxCDD	ND/EDL=0.19	ND/EDL=0.48	1.91
Total HpCDD	1.61	4.31	4.39
Furans			
Total TCDF	0.43	9.90	12.9
Total PeCDF	ND/EDL=0.17	0.67	1.34
Total HxCDF	0.43	0.72	0.43
Total HpCDF	0.43	1.00	0.32

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

SUMMARY OF ANALYTICAL RESULTS
Homologue Group Totals

Date received:	October 16 and 18, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16OC02, 91TT18OC01 and 91TT22OC01
Customer Project Number:	8526-06

MS File Number:	29FEB92LCB3031	27FEB92LCB9061	29FEB92LCB3061
Keystone/NEA Number:	91TT16OC01-07	91TT18OC01-MBRX	91TT18OC01-06RX
Customer Number:	D38C		D28C
Sample Description:	Carp	Method Blank	Carp
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	2.91	1.63	2.75
Total PeCDD	ND/EDL=0.19	2.50	1.59
Total HxCDD	1.00	1.47	6.41
Total HpCDD	1.59	1.06	11.8
Furans			
Total TCDF	8.55	0.50	4.57
Total PeCDF	0.77	ND/EDL=0.22	1.69
Total HxCDF	0.66	ND/EDL=0.18	2.97
Total HpCDF	ND/EDL=0.56	ND/EDL=0.12	3.57

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

SUMMARY OF ANALYTICAL RESULTS

Homologue Group Totals

Date received:	October 22, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16OC02, 91TT18OC01 and 91TT22OC01
Customer Project Number:	8526-06

MS File Number:	29FEB92LCB3071
Keystone/NEA Number:	91TT22OC01-07RX
Customer Number:	D24C
Sample Description:	Carp

Units	pg/g (ppt)

Dioxins	
Total TCDD	3.36
Total PeCDD	1.91
Total HxCDD	6.87
Total HpCDD	11.6
Furans	
Total TCDF	8.49
Total PeCDF	2.59
Total HxCDF	1.60
Total HpCDF	0.95

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	October 16, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16OC02, 91TT18OC01 and 91TT22OC01
Customer Project Number:	8526-06

MS File Number:	27FEB92LCB9051	29FEB92LCB3011	29FEB92LCB3021
Keystone/NEA Number:	91TT16OC01-MB1	91TT16OC01-03	91TT16OC01-04
Customer Number:	8526-06	D35C	D40C
Sample Description:	Method Blank	Carp	Carp
Units	%	%	%
Dioxins			
13C-2378-TCDD	54	61	61
13C-12378-PeCDD	75	84	82
13C-123478-HxCDD	59	66	68
13C-123678-HxCDD	66	82	84
13C-1234678-HpCDD	51	67	61
13C-OCDD	31	42	32
Furans			
13C-2378-TCDF	68	91*	87*
13C-12378-PeCDF	74	70	70
13C-23478-PeCDF	69	71	69
13C-123478-HxCDF	69	69	69
13C-123678-HxCDF	73	72	74
13C-234678-HxCDF	45	35	41
13C-123789-HxCDF	70	67	65
13C-1234678-HpCDF	61	62	56
13C-1234789-HpCDF	70	68	57
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	72	82	83

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received: October 16, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT16OC02, 91TT18OC01 and 91TT22OC01
Customer Project Number: 8526-06

MS File Number:	29FEB92LCB3031	29FEB92LCB3041	29FEB92LCB3051
Keystone/NEA Number:	91TT16OC01-07	91TT16OC01-07MS	91TT16OC01-07MSd
Customer Number:	D38C	D38C	D38C
Sample Description:	Carp	Carp	Carp
Units	%	%	%
Dioxins			
13C-2378-TCDD	59	56	59
13C-12378-PeCDD	93	89	86
13C-123478-HxCDD	70	73	74
13C-123678-HxCDD	85	76	73
13C-1234789-HpCDD	69	68	68
13C-OCDD	47	45	45
Furans			
13C-2378-TCDF	85*	79*	81*
13C-12378-PeCDF	78	76	76
13C-23478-PeCDF	77	73	75
13C-123478-HxCDF	68	69	68
13C-123678-HxCDF	66	67	65
13C-234678-HxCDF	47	44	42
13C-123789-HxCDF	63	65	64
13C-1234678-HpCDF	63	59	61
13C-1234789-HpCDF	71	67	71
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	81	79	77

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

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	October 18 and 22, 1991
Client name:	Tetra Tech
Laboratory Project Number:	91TT16OC02, 91TT18OC01 and 91TT22OC01
Customer Project Number:	8526-06

MS File Number:	27FEB92LCB9061	29FEB92LCB3061	29FEB92LCB3071
Keystone/NEA Number:	91TT18OC01-MBRX	91TT18OC01-06RX	91TT22OC01-07RX
Customer Number:		D28C	D24C
Sample Description:	Method Blank	Carp	Carp
Units	%	%	%
Dioxins			
13C-2378-TCDD	59	74	63
13C-12378-PeCDD	65	104	89
13C-123478-HxCDD	58	71	72
13C-123678-HxCDD	63	86	83
13C-1234789-HpCDD	65	77	75
13C-OCDD	44	60	58
Furans			
13C-2378-TCDF	74	89*	92*
13C-12378-PeCDF	79	100	85
13C-23478-PeCDF	75	97	81
13C-123478-HxCDF	67	78	77
13C-123678-HxCDF	66	75	74
13C-234678-HxCDF	42	50	41
13C-123789-HxCDF	64	71	73
13C-1234678-HpCDF	66	70	69
13C-1234789-HpCDF	73	72	69
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	83	99	83

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

SUMMARY OF ANALYTICAL RESULTS

Matrix Spike Samples

Date received: October 16, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT16OC01
Customer Project Number: 8526-06

MS File Number:	29FEB92LCB3031			29FEB92LCB3041		
Keystone/NEA Number:	91TT16OC01-07			91TT16OC01-07MS		
Sample Description:	Measured Levels	Spiked Levels*	Spiked Levels**	Theoretical Levels	Measured Levels	% Recy
Units	pg/g (ppt)	pg	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)	%
Dioxins						
2378-TCDD	1.28	200	9.94	11.2	17.3	154
12378-PeCDD	0.84	1079	53.6	54.4	63.3	116
123478-HxCDD	0.26	904	44.9	45.2	54.9	122
123678-HxCDD	0.73	888	44.1	44.8	71.0	158
123789-HxCDD	0.12	783	38.9	39.0	52.2	134
1234678-HpCDD	1.59	1012	50.3	51.9	59.8	115
OCDD	2.71	1909	94.8	97.5	123	126
Furans						
2378-TCDF	7.60	188	9.34	16.9	19.1	113
12378-PeCDF	0.21	931	46.3	46.5	69.8	150
23478-PeCDF	0.46	880	43.7	44.2	68.8	156
123478-HxCDF	0.12	950	47.2	47.3	59.8	126
123678-HxCDF	0.09	934	46.4	46.5	65.5	141
234678-HxCDF	0.26	904	44.9	45.2	59.5	132
123789-HxCDF	0.05	960	47.7	47.7	59.8	125
1234678-HpCDF	0.18	897	44.6	44.7	57.3	128
1234789-HpCDF	0	948	47.1	47.1	60.8	129
OCDF	0	1842	91.5	91.5	120	131

Notes:

1. Concentrations marked with an asterisk (*) are the absolute amount of each native analyte spiked into the sample -07MS.
2. Concentrations marked with a double asterisk (**) are the spike levels expressed as pg/g (ppt) for a sample weight of 20.129 grams.
3. The measured level of 2378-TCDF is from a DB-225 column.

SUMMARY OF ANALYTICAL RESULTS
Matrix Spike Samples

Date received: October 16, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT16OC01
Customer Project Number: 8526-06

MS File Number:	29FEB92LCB3031			29FEB92LCB3051		
Keystone/NEA Number:	91TT16OC01-07			91TT16OC01-07MSd		
Sample Description:	Measured Levels	Spiked Levels*	Spiked Levels**	Theoretical Levels	Measured Levels	% Recy
Units	pg/g (ppt)	pg	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)	%
Dioxins						
2378-TCDD	1.28	200	9.19	10.5	15.2	145
12378-PeCDD	0.84	1079	49.6	50.4	60.3	120
123478-HxCDD	0.26	904	41.5	41.8	52.9	127
123678-HxCDD	0.73	888	40.8	41.5	64.6	156
123789-HxCDD	0.12	783	36.0	36.1	49.3	137
1234678-HpCDD	1.59	1012	46.5	48.1	57.6	120
OCDD	2.71	1909	87.7	90.4	115	127
Furans						
2378-TCDF	7.60	188	8.64	16.2	18.7	115
12378-PeCDF	0.21	931	42.8	43.0	65.1	151
23478-PeCDF	0.46	880	40.4	40.9	61.3	150
123478-HxCDF	0.12	950	43.7	43.8	51.9	119
123678-HxCDF	0.09	934	42.9	43.0	59.3	138
234678-HxCDF	0.26	904	41.5	41.8	56.2	134
123789-HxCDF	0.05	960	44.1	44.2	53.8	122
1234678-HpCDF	0.18	897	41.2	41.4	51.5	124
1234789-HpCDF	0.00	948	43.6	43.6	53.1	122
OCDF	0.00	1842	84.6	84.6	113	134

Notes:
1. Concentrations marked with an asterisk (*) are the absolute amount of each native analyte spiked into the sample -07MSd.
2. Concentrations marked with a double asterisk (**) are the spike levels expressed as pg/g (ppt) for a sample weight of 21.764 grams.
3. The measured level of 2378-TCDF is from a DB-225 column.

SUMMARY OF ANALYTICAL RESULTS
Matrix Spike Samples

Date received: October 16, 1991
Client name: Tetra Tech
Laboratory Project Number: 91TT16OC01
Customer Project Number: 8526-06

MS File Number:	29FEB92LCB3041	29FEB92LCB3051	
Keystone/NEA Number:	91TT16OC01-07MS	91TT16OC01-07MSd	
Sample Description:	Matrix Spike	Matrix Spike Duplicate	RPD
Units	pg/g (ppt)	pg/g (ppt)	%
Dioxins			
2378-TCDD	17.3	15.2	-13
12378-PeCDD	63.3	60.3	-5
123478-HxCDD	54.9	52.9	-4
123678-HxCDD	71.0	64.6	-9
123789-HxCDD	52.2	49.3	-6
1234678-HpCDD	59.8	57.6	-4
OCDD	123	115	-7
Furans			
2378-TCDF	19.1	18.7	-2
12378-PeCDF	69.8	65.1	-7
23478-PeCDF	68.8	61.3	-12
123478-HxCDF	59.8	51.9	-14
123678-HxCDF	65.5	59.3	-10
234678-HxCDF	59.5	56.2	-6
123789-HxCDF	59.8	53.8	-11
1234678-HpCDF	57.3	51.5	-11
1234789-HpCDF	60.8	53.1	-14
OCDF	120	113	-6

Notes:
1. The measured level of 2378-TCDF is from a DB-225 column.

SECTION E. PEAMOUTH CHUB

ANALYSIS OF CHUB

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 chub samples were received for PCDD/PCDF analysis under Chain-of-Custody, between October 18 and October 28, 1991. The samples were in good condition upon receipt, and were stored in a freezer maintained at -21°C until analysis. The samples were thawed slightly, ground, then immediately returned to the freezer. The ground tissues were extracted in two sets on January 16, 1992. They were analyzed on a DB-5 column on February 19 and February 26, 1992. Confirmation analyses were on a DB-225 column on February 15, 1992.

Two laboratory method blanks were analyzed with these sample sets. One sample, D24, 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 set of samples was as follows:

LAB ID NUMBER	ANALYSIS	DETECTION LIMIT
91TT18OC01	EPA Method 1613x	1 ppt (tetras)
91TT22OC02	EPA Method 1613x	1 ppt (tetras)
91TT28OC02	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 and sample clean-up followed general guidelines outlined in EPA Method 8290. (Method 1613 does not cover extraction of fish tissue.) 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 occurrence 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. The modifications are outlined below:

Clean-Up Recovery Standard Spiking Levels EPA Method 1613 calls for spiking the sample extracts with 800 pg of ^{37}Cl-2378-TCDD immediately prior to the clean-up procedure. That level has been reduced to 200 pg. The purpose of this change is to reduce the occurrence of false positives due to 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 aqueous matrices.

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 for the continuing 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.)

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 20 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 20 gram sample, the LMCL and UMCL are:

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

NOTE: pg/g = ppt

D. Results

General Chub tissue results are based on the initial weight of the sample (approximately 20 grams). All of the reported analyte concentrations are rounded to three significant figures. Percent lipids are rounded to 2 significant figures. Laboratory Method Blank results are also based on a theoretical sample size of 20 grams. Reported results for the 2378-TCDF are from a DB-225 column, and are highlighted with an asterisk (*). All other results are from a DB-5 column.

Chub Sample Results Only two analytes were consistently present at levels above the LMCL. The 2378-TCDF ranged from 22.2 ppt (sample #D15) to 58.8 ppt (sample D24), and 2378-TCDD ranged from 1.44 ppt (sample D15) to 4.41 ppt (sample #D24). (See Tables 1a - 1d.) The other fifteen analytes were present only at trace levels below the LMCL.

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, D24.

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.

Both laboratory method blanks, 91TT18OC01-MB3 and 91TT22OC02-MB, were Non-Detect for all PCDD and PCDF isomers at or below the LMCL of 0.5 ppt (tetras), 2.5 ppt (pentas, hexas, heptas), and 5.0 ppt (octas). Many of the analytes had sample specific EDL's significantly below their respective LMCLs, ranging from 0.06 ppt to 0.58 ppt.

Internal Standard Recoveries The Internal Standard recoveries for these samples are listed in Tables 3a - 3d. One analyte, ^{13}C-234678-HxCDF, had recoveries below the method guidelines in the second method blank, 91TT22OC02-MB. Since the corresponding analyte was Non-Detect in that sample, the only effect is to raise the sample specific Estimated Detection Limit for that analyte.

Matrix Spike Sample Results The results of the matrix spike and matrix spike duplicate are listed in Tables 4a and 4b, respectively. The percent recoveries were within method requirements for the two analytes which were present in the samples above the LMCL: 2378-TCDD (129-133%) and 2378-TCDF (77-79%).

D. Quality Control Review

All of the data have been reviewed by the scientist performing the analysis, by the Director of the Center for Analytical Mass Spectrometry, and by 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 3/5/92
Laura Chambers Date
Sr. Scientist
Center for Analytical Mass Spectrometry

Peggy Meek 3/5/92
Peggy L. Meek Date
Wet lab Supervisor
Center for Analytical Mass Spectrometry

3.5.92
William H. Chambers Date
Director
Center for Analytical Mass Spectrometry

J. Sprenger 3/4/92
Jeff Sprenger Date
QA Officer
Keystone/NEA

KEYSTONE/NEA
ENVIRONMENTAL RESOURCES

SAMPLE ANALYSIS SUMMARY

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received: October 18 through October 28, 1991
Client name: TetraTech
Laboratory Project Number: 91TT18OC01, 91TT22OC02, and 91TT28OC02
Customer Project Number:

MS File Number:	19FEB92LCB3011	19FEB92LCB3031	19FEB92LCB3041
Keystone/NEA Number:	91TT18OC01-MB3	91TT18OC01-04	91TT28OC02-08
Customer Number:		D28	D10
Sample Description:	Method Blank	Chub	Chub
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	ND/EDL=0.22	2.00	2.32
12378-PeCDD	ND/EDL=0.19	0.66	0.50
123478-HxCDD	ND/EDL=0.27	EMPC=0.20	EMPC=0.11
123678-HxCDD	ND/EDL=0.19	0.59	0.31
123789-HxCDD	ND/EDL=0.20	0.22	0.14
1234678-HpCDD	EMPC=0.31	EMPC=1.83	0.65
OCDD	1.40	8.40	3.62
Furans			
2378-TCDF	EMPC=0.12	32.5*	40.0*
12378-PeCDF	ND/EDL=0.29	EMPC=0.38	0.31
23478-PeCDF	ND/EDL=0.23	0.82	0.59
123478-HxCDF	ND/EDL=0.24	0.24	ND/EDL=0.11
123678-HxCDF	ND/EDL=0.22	0.13	ND/EDL=0.10
234678-HxCDF	ND/EDL=0.44	0.32	EMPC=0.26
123789-HxCDF	ND/EDL=0.54	ND/EDL=0.26	ND/EDL=0.15
1234678-HpCDF	EMPC=0.13	0.43	0.21
1234789-HpCDF	ND/EDL=0.33	EMPC=0.18	EMPC=0.06
OCDF	ND/EDL=1.49	EMPC=1.01	0.31
Percent Lipids	N.A.	11.2%	17.0%

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

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 18 through October 28, 1991
Client name:	TetraTech
Laboratory Project Number:	91TT18OC01, 91TT22OC02, and 91TT28OC02
Customer Project Number:	

MS File Number:	19FEB92LCB3051	19FEB92LCB3061
Keystone/NEA Number:	91TT28OC02-10	91TT28OC02-11
Customer Number:	D19	D15
Sample Description:	Chub	Chub

Units	pg/g (ppt)	pg/g (ppt)
Dioxins		
2378-TCDD	3.29	1.44
12378-PeCDD	0.70	0.31
123478-HxCDD	0.14	0.11
123678-HxCDD	0.51	0.39
123789-HxCDD	0.15	EMPC=0.12
1234678-HpCDD	0.73	0.74
OCDD	4.47	5.67
Furans		
2378-TCDF	52.1*	22.2*
12378-PeCDF	EMPC=0.58	0.24
23478-PeCDF	0.94	0.55
123478-HxCDF	0.13	0.12
123678-HxCDF	EMPC=0.07	0.05
234678-HxCDF	EMPC=0.23	0.25
123789-HxCDF	ND/EDL=0.11	ND/EDL=0.08
1234678-HpCDF	0.20	EMPC=0.16
1234789-HpCDF	ND/EDL=0.08	0.04
OCDF	0.53	0.38
Percent Lipids	12.9%	11.4%

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 18 through October 28, 1991
Client name:	TetraTech
Laboratory Project Number:	91TT18OC01, 91TT22OC02, and 91TT28OC02
Customer Project Number:	

MS File Number:	19FEB92LCB3021	19FEB92LCB3071	26FEB92LCB3011
Keystone/NEA Number:	91TT22OC02-MB	91TT22OC02-01	91TT22OC02-04
Customer Number:		D21	D24
Sample Description:	Method Blank	Chub	Chub
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
2378-TCDD	ND/EDL=0.19	2.77	4.41
12378-PeCDD	ND/EDL=0.15	0.76	EMPC=2.04
123478-HxCDD	ND/EDL=0.12	EMPC=0.21	EMPC=0.87
123678-HxCDD	ND/EDL=0.09	0.63	1.16
123789-HxCDD	ND/EDL=0.09	0.18	ND/EDL=0.47
1234678-HpCDD	EMPC=0.02	1.09	2.81
OCDD	1.43	4.21	18.1
Furans			
2378-TCDF	ND/EDL=0.10	41.2*	58.8*
12378-PeCDF	ND/EDL=0.07	0.56	0.86
23478-PeCDF	ND/EDL=0.06	0.90	2.46
123478-HxCDF	ND/EDL=0.16	0.16	EMPC=0.56
123678-HxCDF	ND/EDL=0.15	EMPC=0.06	EMPC=0.44
234678-HxCDF	ND/EDL=0.58	0.29	ND/EDL=1.61
123789-HxCDF	ND/EDL=0.26	ND/EDL=0.14	ND/EDL=1.38
1234678-HpCDF	ND/EDL=0.16	0.18	0.74
1234789-HpCDF	ND/EDL=0.19	EMPC=0.07	ND/EDL=0.50
OCDF	0.39	EMPC=0.41	2.03
Percent Lipids	N.A.	14.5%	12.0%

Notes:

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

SUMMARY OF ANALYTICAL RESULTS
2378-Substituted Isomers

Date received:	October 18 through October 28, 1991
Client name:	TetraTech
Laboratory Project Number:	91TT18OC01, 91TT22OC02, and 91TT28OC02
Customer Project Number:	

MS File Number:	26FEB92LCB3021
Keystone/NEA Number:	91TT22OC02-06
Customer Number:	D23
Sample Description:	Chub

Units	pg/g (ppt)

Dioxins	
2378-TCDD	3.10
12378-PeCDD	0.83
123478-HxCDD	EMPC=0.39
123678-HxCDD	EMPC=0.62
123789-HxCDD	EMPC=0.29
1234678-HpCDD	0.24
OCDD	3.91
Furans	
2378-TCDF	42.5*
12378-PeCDF	EMPC=0.65
23478-PeCDF	EMPC=0.95
123478-HxCDF	ND/EDL=0.71
123678-HxCDF	ND/EDL=0.64
234678-HxCDF	ND/EDL=1.38
123789-HxCDF	ND/EDL=1.09
1234678-HpCDF	ND/EDL=0.17
1234789-HpCDF	ND/EDL=0.18
OCDF	ND/EDL=1.18
Percent Lipids	13.2%

Notes:

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

SUMMARY OF ANALYTICAL RESULTS

Homologue Group Totals

Date received:	October 18 through October 28, 1991
Client name:	TetraTech
Laboratory Project Number:	91TT18OC01, 91TT22OC02, and 91TT28OC02
Customer Project Number:	

MS File Number:	19FEB92LCB3011	19FEB92LCB3031	19FEB92LCB3041
Keystone/NEA Number:	91TT18OC01-MB3	91TT18OC01-04	91TT28OC02-08
Customer Number:		D28	D10
Sample Description:	Method Blank	Chub	Chub

Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	0.77	4.14	3.96
Total PeCDD	ND/EDL=0.19	1.17	0.5
Total HxCDD	ND/EDL=0.19	1.90	0.77
Total HpCDD	ND/EDL=0.27	1.51	1.35
Furans			
Total TCDF	0.45	25.1	30.1
Total PeCDF	ND/EDL=0.23	1.85	2.01
Total HxCDF	ND/EDL=0.22	2.41	0.31
Total HpCDF	ND/EDL=0.33	0.90	0.31

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. ND/LMCL = Analyte Not Detected at or above the Lower Method Calibration Limit.

SUMMARY OF ANALYTICAL RESULTS

Homologue Group Totals

Date received:	October 18 through October 28, 1991
Client name:	TetraTech
Laboratory Project Number:	91TT18OC01, 91TT22OC02, and 91TT28OC02
Customer Project Number:	

MS File Number:	19FEB92LCB3051	19FEB92LCB3061
Keystone/NEA Number:	91TT28OC02-10	91TT28OC02-11
Customer Number:	D19	D15
Sample Description:	Chub	Chub

Units	pg/g (ppt)	pg/g (ppt)
Dioxins		
Total TCDD	5.12	3.67
Total PeCDD	0.87	0.38
Total HxCDD	1.11	0.86
Total HpCDD	1.37	0.74
Furans		
Total TCDF	39.0	17.8
Total PeCDF	1.93	1.86
Total HxCDF	1.10	0.78
Total HpCDF	0.19	0.25

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. ND/LMCL = Analyte Not Detected at or above the Lower Method Calibration Limit.

SUMMARY OF ANALYTICAL RESULTS

Homologue Group Totals

Date received: October 18 through October 28, 1991
Client name: TetraTech
Laboratory Project Number: 91TT18OC01, 91TT22OC02, and 91TT28OC02
Customer Project Number:

MS File Number:	19FEB92LCB3021	19FEB92LCB3071	26FEB92LCB3011
Keystone/NEA Number:	91TT22OC02-MB	91TT22OC02-01	91TT22OC02-04
Customer Number:		D21	D24
Sample Description:	Method Blank	Chub	Chub
Units	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)
Dioxins			
Total TCDD	5.21	7.45	10.1
Total PeCDD	ND/EDL=0.15	1.58	ND/EDL=1.05
Total HxCDD	ND/EDL=0.09	1.92	2.51
Total HpCDD	ND/LMCL=2.50	1.89	5.34
Furans			
Total TCDF	1.09	35.6	42.6
Total PeCDF	ND/EDL=0.06	3.08	5.89
Total HxCDF	ND/EDL=0.15	1.00	1.88
Total HpCDF	ND/EDL=0.16	0.14	0.86

Notes:
1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. ND/LMCL = Analyte Not Detected at or above the Lower Method Calibration Limit.

SUMMARY OF ANALYTICAL RESULTS

Homologue Group Totals

Date received:	October 18 through October 28, 1991
Client name:	TetraTech
Laboratory Project Number:	91TT18OC01, 91TT22OC02, and 91TT28OC02
Customer Project Number:	

MS File Number:	26FEB92LCB3021
Keystone/NEA Number:	91TT22OC02-06
Customer Number:	D23
Sample Description:	Chub

Units	pg/g (ppt)

Dioxins	
Total TCDD	8.29
Total PeCDD	1.47
Total HxCDD	ND/EDL=0.16
Total HpCDD	2.77
Furans	
Total TCDF	35.8
Total PeCDF	2.21
Total HxCDF	1.34
Total HpCDF	ND/EDL=0.17

Notes:

1. ND/EDL = Analyte Not Detected at or above the sample specific Estimated Detection Limit.
2. ND/LMCL = Analyte Not Detected at or above the Lower Method Calibration Limit.

SUMMARY OF ANALYTICAL RESULTS

Internal Standard Recoveries

Date received: October 18 through October 28, 1991
Client name: TetraTech
Laboratory Project Number: 91TT18OC01, 91TT22OC02, and 91TT28OC02
Customer Project Number:

MS File Number:	19FEB92LCB3011	19FEB92LCB3031	19FEB92LCB3041
Keystone/NEA Number:	91TT18OC01-MB3	91TT18OC01-04	91TT28OC02-08
Customer Number:		D28	D10
Sample Description:	Method Blank	Chub	Chub
Units	%	%	%
Dioxins			
13C-2378-TCDD	44	54	61
13C-12378-PeCDD	53	67	76
13C-123478-HxCDD	46	59	62
13C-123678-HxCDD	72	81	83
13C-1234678-HpCDD	32	60	62
13C-OCDD	12	36	40
Furans			
13C-2378-TCDF	59	75*	90*
13C-12378-PeCDF	46	59	67
13C-23478-PeCDF	52	63	70
13C-123478-HxCDF	60	67	67
13C-123678-HxCDF	70	72	72
13C-234678-HxCDF	33	39	36
13C-123789-HxCDF	36	52	63
13C-1234678-HpCDF	39	60	59
13C-1234789-HpCDF	35	71	49
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	59	74	77

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

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received:	October 18 through October 28, 1991
Client name:	TetraTech
Laboratory Project Number:	91TT18OC01, 91TT22OC02, and 91TT28OC02
Customer Project Number:	

MS File Number:	19FEB92LCB3051	19FEB92LCB3061
Keystone/NEA Number:	91TT28OC02-10	91TT28OC02-11
Customer Number:	D19	D15
Sample Description:	Chub	Chub
Units	%	%
Dioxins		
13C-2378-TCDD	69	62
13C-12378-PeCDD	93	93
13C-123478-HxCDD	72	68
13C-123678-HxCDD	90	77
13C-1234789-HpCDD	73	67
13C-OCDD	47	42
Furans		
13C-2378-TCDF	99*	89*
13C-12378-PeCDF	76	70
13C-23478-PeCDF	84	33
13C-123478-HxCDF	78	70
13C-123678-HxCDF	77	69
13C-234678-HxCDF	51	39
13C-123789-HxCDF	72	66
13C-1234678-HpCDF	60	59
13C-1234789-HpCDF	75	50
Clean-Up Recovery Standard		
37Cl4-2378-TCDD	97	88

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

SUMMARY OF ANALYTICAL RESULTS
Internal Standard Recoveries

Date received: October 18 through October 28, 1991
Client name: TetraTech
Laboratory Project Number: 91TT18OC01, 91TT22OC02, and 91TT28OC02
Customer Project Number:

MS File Number:	19FEB92LCB3021	19FEB92LCB3071	26FEB92LCB3011
Keystone/NEA Number:	91TT22OC02-MB	91TT22OC02-01	91TT22OC02-04
Customer Number:		D21	D24
Sample Description:	Method Blank	Chub	Chub
Units	%	%	%
Dioxins			
13C-2378-TCDD	44	57	57
13C-12378-PeCDD	53	73	61
13C-123478-HxCDD	43	63	55
13C-123678-HxCDD	65	75	87
13C-1234789-HpCDD	38	58	59
13C-OCDD	20	36	39
Furans			
13C-2378-TCDF	55	81*	85*
13C-12378-PeCDF	45	60	63
13C-23478-PeCDF	51	70	67
13C-123478-HxCDF	54	64	70
13C-123678-HxCDF	59	64	83
13C-234678-HxCDF	16	32	42
13C-123789-HxCDF	46	59	58
13C-1234678-HpCDF	38	39	61
13C-1234789-HpCDF	42	59	64
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	58	79	78

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

SUMMARY OF ANALYTICAL RESULTS

Internal Standard Recoveries

Date received: October 18 through October 28, 1991
Client name: TetraTech
Laboratory Project Number: 91TT18OC01, 91TT22OC02, and 91TT28OC02
Customer Project Number:

MS File Number:	26FEB92LCB3021	26FEB92LCB3031	26FEB92LCB3041
Keystone/NEA Number:	91TT22OC02-06	91TT22OC02-04MS	91TT22OC02-04MSd
Customer Number:	D23	D24 + Matrix Spike	D24 + Matrix Spike
Sample Description:	Chub	Chub	Chub
Units	%	%	%
Dioxins			
13C-2378-TCDD	58	59	54
13C-12378-PeCDD	66	66	63
13C-123478-HxCDD	61	65	64
13C-123678-HxCDD	83	87	80
13C-1234789-HpCDD	56	62	57
13C-OCDD	33	36	32
Furans			
13C-2378-TCDF	82*	85*	74*
13C-12378-PeCDF	68	67	63
13C-23478-PeCDF	70	69	64
13C-123478-HxCDF	76	77	76
13C-123678-HxCDF	81	82	76
13C-234678-HxCDF	39	47	47
13C-123789-HxCDF	62	79	63
13C-1234678-HpCDF	58	62	59
13C-1234789-HpCDF	70	76	68
Clean-Up Recovery Standard			
37Cl4-2378-TCDD	83	81	77

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

SUMMARY OF ANALYTICAL RESULTS

Matrix Spike Samples

Date received: October 18 through October 28, 1991
Client name: TetraTech
Laboratory Project Number: 91TT18OC01, 91TT22OC02, and 91TT28OC02
Customer Project Number:

MS File Number:	26FEB92LCB3011			26FEB92LCB3031		
Keystone/NEA Number:	92TT22OC02-04			92TT22OC02-04MS		
Sample Description:	Measured Levels	Spiked Levels*	Spiked Levels**	Theoretical Levels	Measured Levels	% Recy
Units	pg/g (ppt)	pg	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)	%
Dioxins						
2378-TCDD	4.41	200	8.92	13.33	17.2	129
12378-PeCDD	2.04	1079	48.10	50.14	55.6	111
123478-HxCDD	0.87	904	40.30	41.17	60.1	146
123678-HxCDD	1.16	888	39.59	40.75	53.8	132
123789-HxCDD	0.00	783	34.91	34.91	39.5	113
1234678-HpCDD	2.81	1012	45.12	47.93	58.6	122
OCDD	18.10	1909	85.11	103.21	122	118
Furans						
2378-TCDF	58.80	188	8.38	67.18	51.5	77
12378-PeCDF	0.86	931	41.51	42.37	64.2	152
23478-PeCDF	2.46	880	39.23	41.69	63.3	152
123478-HxCDF	0.56	950	42.35	42.91	56.3	131
123678-HxCDF	0.44	934	41.64	42.08	59.5	141
234678-HxCDF	0.00	904	40.30	40.30	53.6	133
123789-HxCDF	0.00	960	42.80	42.80	54.9	128
1234678-HpCDF	0.74	897	39.99	40.73	50.9	125
1234789-HpCDF	0.00	948	42.26	42.26	51.4	122
OCDF	2.03	1842	82.12	84.15	135	160

Notes:

1. Concentrations highlighted with an asterisk (*) are the absolute amount of each native analyte spiked into the sample -04MS.
2. Concentrations highlighted with a double asterisk (**) are the spike levels expressed as pg/g (ppt) for a sample weight of 22.431 grams.
3. Reported concentrations for the 2378-TCDF are from a DB-225 column.

SUMMARY OF ANALYTICAL RESULTS
Matrix Spike Samples

Date received: October 18 through October 28, 1991
Client name: TetraTech
Laboratory Project Number: 91TT18OC01, 91TT22OC02, and 91TT28OC02
Customer Project Number:

MS File Number:				26FEB92LCB3041		
Keystone/NEA Number:	92TT22OC02-04			92TT22OC02-04MSd		
Sample Description:	Measured Levels	Spiked Levels*	Spiked Levels**	Theoretical Levels	Measured Levels	% Recy
Units	pg/g (ppt)	pg	pg/g (ppt)	pg/g (ppt)	pg/g (ppt)	%
Dioxins						
2378-TCDD	4.41	200	10.05	14.46	19.3	133
12378-PeCDD	2.04	1079	54.20	56.24	61.6	110
123478-HxCDD	0.87	904	45.41	46.28	70.4	152
123678-HxCDD	1.16	888	44.61	45.77	60.8	133
123789-HxCDD	0.00	783	39.33	39.33	44.9	114
1234678-HpCDD	2.81	1012	50.84	53.65	68.3	127
OCDD	18.10	1909	95.90	114.00	141	124
Furans						
2378-TCDF	58.80	188	9.44	68.24	53.9	79
12378-PeCDF	0.86	931	46.77	47.63	74.4	156
23478-PeCDF	2.46	880	44.21	46.67	72.8	156
123478-HxCDF	0.56	950	47.72	48.28	63.0	130
123678-HxCDF	0.44	934	46.92	47.36	66.4	140
234678-HxCDF	0.00	904	45.41	45.41	61.2	135
123789-HxCDF	0.00	960	48.23	48.23	64.4	134
1234678-HpCDF	0.74	897	45.06	45.80	62.7	137
1234789-HpCDF	0.00	948	47.62	47.62	59.4	125
OCDF	2.03	1842	92.53	94.56	153	162

Notes:
1. Concentrations marked with an asterisk (*) are the absolute amount of each native analyte spiked into the sample -04MSd.
2. Concentrations marked with a double asterisk (**) are the spike levels expressed as pg/g (ppt) for a sample weight of 19.906 grams.
3. Reported concentrations for the 2378-TCDF are from a DB-225 column.

SUMMARY OF ANALYTICAL RESULTS
Matrix Spike Samples

Date received: October 18 through October 28, 1991
Client name: TetraTech
Laboratory Project Number: 91TT18OC01, 91TT22OC02, and 91TT28OC02
Customer Project Number:

	MS File Number:	Keystone/NEA Number:	Sample Description:	Units	26FEB92LCB3031 92TT22OC02-04MS Matrix Spike pg/g (ppt)	26FEB92LCB3041 92TT22OC02-04MSd Matrix Spike Duplicate pg/g (ppt)	RPD %
Dioxins							
2378-TCDD					17.2	19.3	12
12378-PeCDD					55.6	61.6	10
123478-HxCDD					60.1	70.4	16
123678-HxCDD					53.8	60.8	12
123789-HxCDD					39.5	44.9	13
1234678-HpCDD					58.6	68.3	15
OCDD					122	141	14
Furans							
2378-TCDF					51.5	53.9	5
12378-PeCDF					64.2	74.4	15
23478-PeCDF					63.3	72.8	14
123478-HxCDF					56.3	63.0	11
123678-HxCDF					59.5	66.4	11
234678-HxCDF					53.6	61.2	13
123789-HxCDF					54.9	64.4	16
1234678-HpCDF					50.9	62.7	21
1234789-HpCDF					51.4	59.4	14
OCDF					135	153	13

Notes:
1. Concentrations marked with an asterisk (*) are the absolute amount of each native analyte spiked into the sample -04MSd.
2. Concentrations marked with a double asterisk (**) are the spike levels expressed as pg/g (ppt) for a sample weight of 19.906 grams.
3. Reported concentrations for the 2378-TCDF are from a DB-225 column.