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DETERMINATION OF PCDD/PCDF LEVELS

Prepared for:
Veritech
Attn: Tracy Batelli
175 Route 46 West
Fairfield, NJ 07004

This report contains 22 pages.

The results reported herein conform to the most current NELAC standards, where applicable, unless otherwise narrated in the body of the report.

Project: Chemical Analysis

Client Purchase Order Number: NA

REPORT OF LABORATORY ANALYSIS

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REPORT OF: CHEMICAL ANALYSES

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PROJECT: PCDD/PCDF ANALYSES

DATE: April 4, 2002

ISSUED TO: Veritech
Attn: Ms. Tracy Batelli
175 Route 46 West
Fairfield, NJ 07004

REPORT NO: 02-1054762

INTRODUCTION

This report presents the results from the analysis performed on one sample which was submitted by a representative of Veritech. The sample was analyzed for the presence or absence of polychlorinated dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs) using a modified version of USEPA Method 1613 as described below.

SAMPLE IDENTIFICATION

Client ID	Sample Type	Date Received	PACE ID
GW-002	Water	03/06/02	103367884

METHODOLOGY

Sample Extraction

The sample was spiked with $^{13}C_{12}$-labeled PCDD/PCDF internal standards (Table 1) and extracted with methylene chloride in a separatory funnel. The extract was quantitatively transferred to a Kuderna-Danish concentrator, concentrated, and solvent exchanged to hexane. The hexane extract was then spiked with 2,3,7,8-TCDD-$^{37}Cl_4$ enrichment efficiency standard (Table 1) and processed through the analyte enrichment procedures described below.

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PCDD/PCDF Analyte Enrichment

The extraction procedure often removes a variety of compounds, in addition to the PCDDs and PCDFs, from the sample matrix. Some of these compounds can directly interfere with the analyses while others can overload the capillary column causing degradation in chromatographic resolution or sensitivity. The analyte enrichment steps described below are used to remove interferences from the extracts.

The extract was diluted to 100 mL with hexane, transferred to a separatory funnel, and washed with 1N sodium hydroxide, concentrated sulfuric acid, and aqueous sodium chloride (5% w/v) as needed. The hexane extract was quantitatively transferred to a liquid chromatography column containing alternating layers of silica gel, 40% concentrated sulfuric acid on silica gel, and 33% 1 N sodium hydroxide on silica gel. The column was eluted with 90 mL of hexane and the entire eluate was collected and concentrated, under ambient conditions, to a volume of 1 mL.

The extract was then fractionated on a liquid chromatography column containing 4 g of activated alumina. The column was eluted with 20 mL of hexane followed by 15 mL of 60% methylene chloride/hexane. The 60% methylene chloride/hexane fraction was concentrated under a stream of dry nitrogen, spiked with recovery standards (1,2,3,4-TCDD-$^{13}C_{12}$ and 1,2,3,7,8,9-HxCDD-$^{13}C_{12}$) and taken to a final volume of 20 uL.

PCDD/PCDF Analyses

The sample extract was analyzed for the presence of PCDDs and PCDFs using combined capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS). The instrumentation consisted of a Hewlett Packard Model 6890 gas chromatograph interfaced to a Micromass Ultima high-resolution mass spectrometer. The capillary column was interfaced directly into the ion source of the mass spectrometer, thus providing the highest possible sensitivity while minimizing degradation of the chromatographic resolution.

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PCDD/PCDF Analyses (Cont.)

The mass spectrometer was operated in the electron impact ionization mode at a mass resolution of 10,000-11,000 (M/ΔM, 10 percent valley definition). This resolution is sufficient to resolve most interferences, such as PCBs, thus providing the highest level of confidence that the detected levels of PCDD/PCDF were not false positives resulting from interferences. Typical operating parameters for the HRGC/HRMS analyses are summarized in Table 2.

The data were acquired by selected-ion-recording (SIR) using groups of ion masses similar to those described in USEPA Method 1613. The five groups corresponded to the tetrachlorinated through octachlorinated congener classes. Each group contained two ion masses for the PCDDs, two ion masses for the PCDFs, the corresponding ion masses from the two isotopically labeled internal standards, and the ion mass characteristic of the polychlorinated diphenylether (PCDE) which, if present, could cause false responses in the dibenzofuran channels.

Each group of ion masses also contained a lock mass which was used by the data system to automatically correct the mass focus of the instrument. The data system determined the centroid of the lock mass during each data acquisition cycle and corrected the mass focus of the analyte and internal standard ion masses to assure that the centers of the mass peaks were being monitored.

The criteria used to judge positive responses for a PCDD/PCDF isomer included:

* Simultaneous response at both ion masses of the PCDD or PCDF
* Signal-to-noise ratio equal to or greater than 2.5:1.0 for both ion masses
* Chlorine isotope ratio within 15% of the theoretical value
* Chromatographic retention time within +/- 2 seconds of the expected retention time
* Chromatographic retention times within elution windows determined from analyses of standard mixtures
* Absence of simultaneous response in the PCDF and PCDPE ion traces

A list of the exact ion masses monitored for the determination of PCDD/PCDF isomers and the PCDE interferences is presented in Table 3. Also included are the theoretical chlorine isotope ratios for the ten congener classes.

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PROJECT: PCDD/PCDF ANALYSES **DATE:** April 4, 2002

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PCDD/PCDF Quantification and Calculations

The PCDD/PCDF isomers were quantified by comparison of their responses to the responses of the labeled internal standards. Relative response factors were calculated from analyses of standard mixtures containing representatives of each of the PCDD/PCDF congener classes at five concentration levels, and each of the internal standards at one concentration level, as shown in Table 4. The PCDD/PCDF response factors were calculated by comparing the sum of the responses from the two ion masses monitored for each chlorine congener class to the sum of the responses from the two ion masses of the corresponding isotopically labeled internal standard. The formula for the response factor calculation is:

$$Rf = \frac{An \times Qis}{Ais \times Qn}$$

where:

Rf = Response factor
An = Sum of integrated areas for native isomer
Qis = Quantity of labeled internal standard
Ais = Sum of integrated areas for labeled internal standard
Qn = Quantity of native isomer

The levels of PCDD/PCDF in the sample were quantified using the following equation:

$$C = \frac{An \times Qis}{Ais \times W \times Rf}$$

where:

C = Concentration of target isomer or congener class
An = Sum of integrated areas for the target isomer or congener class
Qis = Quantity of labeled internal standard added to the sample
Ais = Sum of integrated areas for the labeled internal standard
W = Sample amount
Rf = Response factor

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PCDD/PCDF Quantification and Calculations (Cont.)

Each pair of ion mass peaks in the selected-ion-current chromatograms was evaluated manually to determine if it met the criteria for a PCDD or PCDF isomer. Areas of all peaks exhibiting correct ion ratios, having retention times within the correct windows, and having areas corresponding to concentrations in the range covered by the initial calibration were then summed for calculations of total congener concentrations.

The Pace Reporting Limit (PRL), equivalent to the practical quantitation limit, was determined based on the weight of the sample aliquot that was extracted, the volume of the final extract, and the concentration of the lowest level standard in the calibration curve. A PRL was calculated for each isomer/isomer group using the following equation:

$$PRL = \frac{(C \times V)}{W}$$

where:

PRL = Pace Reporting Limit
C = Concentration of Lowest Level Standard
V = Volume of Final Extract
W = Initial Sample Amount

The recovery of the 2,3,7,8-TCDD-$^{37}Cl_4$ enrichment efficiency standard and each $^{13}C_{12}$-labeled internal standard, relative to either 1,2,3,4-TCDD-$^{13}C_{12}$ or 1,2,3,7,8,9-HxCDD-$^{13}C_{12}$, was calculated using the following equation:

$$\%R = \frac{Ais \times Qrs \times 100\%}{Rfr \times Ars \times Qis}$$

where:

%R = Percent recovery of labeled internal standard
Ais = Sum of integrated areas of labeled internal standard
Qrs = Quantity of recovery standard
Ars = Sum of integrated areas of recovery standard
Rfr = Response factor of the specific labeled internal standard relative to the recovery standard
Qis = Quantity of the labeled internal standard congener added to the sample

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PROJECT: PCDD/PCDF ANALYSES **DATE:** April 4, 2002

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Quality Control for PCDD/PCDF Analyses

The performance of the sample processing steps and the instrumentation are monitored on a routine basis. The procedures and criteria are summarized below.

One method blank and one laboratory spike sample are typically prepared with each ten samples of any given matrix. The recoveries of the native PCDD/PCDF analytes in the laboratory spike samples generally range from 70 to 130%. Recoveries of selected analytes outside this range do not invalidate the data but provide information which is used by the laboratory to monitor recovery trends and to assure optimization of the method.

Internal standards are spiked into each sample prior to extraction in order to monitor the level of recovery which is achieved for each individual sample. Acceptable recoveries range from 25 to 150 percent for the internal standards unless a deviation is due to variation in instrument response as a result of analytical interferences.

The resolution of the mass spectrometer is verified prior to each analysis to be 10,000 or greater. Hardcopies of the reference peaks are printed at the beginning and end of each analysis day. The resolving power of the DB-5MS chromatographic column is checked daily by analyzing a standard solution containing 2,3,7,8-TCDD and the adjacent TCDD isomers. The DB-225 column resolution is checked daily by analyzing a standard solution containing 2,3,7,8-TCDF and the adjacent TCDF isomers. Acceptable performance is achieved when 2,3,7,8-TCDD or 2,3,7,8-TCDF is resolved from the adjacent isomers by a valley of 25% or less. The group times for the selected-ion-monitoring data acquisitions are also checked daily by analyzing the column performance mix which has been modified to contain the first and last eluting isomers of each congener class. In this way one is assured of collecting data representative of the total PCDD/PCDF content and that the 2,3,7,8-substituted isomers are suitably resolved.

Initial calibrations are generated by analyzing standard solutions (see Table 4) containing target native and labeled PCDD/PCDF compounds. Response factors are calculated and averaged for each compound. These averages are used for quantification and for comparison to the daily continuing calibration. The relative standard deviation for each native compound must be 20% or less (30% or less for the labeled compounds) as specified in Method 8290. A continuing calibration standard is analyzed at the beginning of each 12-hour shift on days when initial calibrations are not performed. The initial calibration is considered to be valid when the response factors from the continuing calibration analysis agree with the averages from the initial calibration within the Method 1613 target limits.

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RESULTS

The results from the analyses are presented in the following:

Appendix A - Documentation
Appendix B - PCDD/PCDF Analysis Results

DISCUSSION

The recoveries of the isotopically-labeled PCDD/PCDF internal standards in the sample extract ranged from 68-112% and indicate a level of efficiency through the extraction and enrichment steps that is considered typical for this matrix. All of the internal standard recoveries were within the Method 1613 target ranges. Since the quantifications of the native 2,3,7,8-substituted isomers were based on isotope dilution, the data were automatically corrected for variation in recovery and accurate values were obtained.

A laboratory method blank was prepared and analyzed with the sample batch as part of our routine quality control procedures. The results, found at the beginning of Appendix B, show the blank to be free of PCDDs and PCDFs at the reported detection limits. This indicates that the sample preparation procedures did not significantly impact the results of the field sample determinations.

A laboratory spike sample was also prepared by extracting laboratory water that had been fortified with native standard materials. Recoveries of the native compounds in the spike sample ranged from 89-111%. These results indicate a high level of accuracy for these analyses.

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REMARKS

The sample extract will be retained for a period of 30 days from the date of this report and then discarded unless other arrangements are made. The raw mass spectral data will be archived on magnetic tape for a period of not less than one year. Questions regarding the data contained in this report may be directed to the authors at the numbers provided below.

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TABLE 1. Spike Levels of PCDD/PCDF Standards

Internal Standards	Spike Level (ng)
2,3,7,8-TCDF-$^{13}C_{12}$	2.0
2,3,7,8-TCDD-$^{13}C_{12}$	2.0
1,2,3,7,8-PeCDF-$^{13}C_{12}$	2.0
2,3,4,7,8-PeCDF-$^{13}C_{12}$	2.0
1,2,3,7,8-PeCDD-$^{13}C_{12}$	2.0
1,2,3,4,7,8-HxCDF-$^{13}C_{12}$	2.0
1,2,3,6,7,8-HxCDF-$^{13}C_{12}$	2.0
1,2,3,7,8,9-HxCDF-$^{13}C_{12}$	2.0
2,3,4,6,7,8-HxCDF-$^{13}C_{12}$	2.0
1,2,3,4,7,8-HxCDD-$^{13}C_{12}$	2.0
1,2,3,6,7,8-HxCDD-$^{13}C_{12}$	2.0
1,2,3,4,6,7,8-HpCDF-$^{13}C_{12}$	2.0
1,2,3,4,7,8,9-HpCDF-$^{13}C_{12}$	2.0
1,2,3,4,6,7,8-HpCDD-$^{13}C_{12}$	2.0
OCDD-$^{13}C_{12}$	4.0
Recovery Standards	
1,2,3,4-TCDD-$^{13}C_{12}$	2.0
1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	2.0
Enrichment Efficiency Standard	
2,3,7,8-TCDD-$^{37}Cl_4$	0.2

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TABLE 2. High Resolution PCDD/PCDF Analyses
HRGC/HRMS Operating Parameters

Mass Resolution	10,000-11,000 (M/ΔM, 10% valley)
Electron Energy	32 electron volts
Accelerating Voltage	8,000 volts
Source Temperature	275°C
Preamplifier Gain	10^{-6} amp/volt
Multiplier Gain	~10^5
Chromatographic Column	60 M DB-5MS
Transfer Line Temperature	260°C
Injection Mode	Splitless
Carrier Gas	Helium
Carrier Flow Velocity	~30 cm/sec

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TABLE 3. Exact Ion Masses Monitored for the Determination of PCDDs, PCDFs, and PCDEs

Compound	Accurate Mass Mass 1	Mass 2	Theoretical Ratio Mass 1/Mass 2
Tetra-CDDs	319.8965	321.8936	0.77
Tetra-CDFs	303.9016	305.8987	0.77
Hexa-CDEs	375.8364		
Penta-CDDs	355.8546	357.8517	1.54
Penta-CDFs	339.8597	341.8567	1.54
Hepta-CDEs	409.7974		
Hexa-CDDs	389.8156	391.8127	1.23
Hexa-CDFs	373.8207	375.8178	1.23
Octa-CDEs	445.7555		
Hepta-CDDs	423.7766	425.7737	1.03
Hepta-CDFs	407.7817	409.7788	1.03
Nona-CDEs	479.7165		
Octa-CDD	457.7377	459.7347	0.88
Octa-CDF	441.7428	443.7398	0.88
Deca-CDE	513.6775		

CDDs = Chlorinated Dibenzo-p-dioxins
CDFs = Chlorinated Dibenzofurans
CDEs = Chlorinated Diphenylethers

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TABLE 4. High Resolution Calibration Solutions

	Concentration (pg/uL)				
Native CDDs/CDFs	**CS1**	**CS2**	**CS3**	**CS4**	**CS5**
2,3,7,8-TCDD	0.5	2	10	40	200
2,3,7,8 TCDF	0.5	2	10	40	200
1,2,3,7,8-PeCDD	2.5	10	50	200	1000
1,2,3,7,8-PeCDF	2.5	10	50	200	1000
2,3,4,7,8-PeCDF	2.5	10	50	200	1000
1,2,3,4,7,8-HxCDD	2.5	10	50	200	1000
1,2,3,6,7,8-HxCDD	2.5	10	50	200	1000
1,2,3,7,8,9-HxCDD	2.5	10	50	200	1000
1,2,3,4,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,6,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,7,8,9-HxCDF	2.5	10	50	200	1000
2,3,4,6,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,4,6,7,8-HpCDD	2.5	10	50	200	1000
1,2,3,4,6,7,8-HpCDF	2.5	10	50	200	1000
1,2,3,4,7,8,9-HpCDF	2.5	10	50	200	1000
OCDD	5.0	20	100	400	2000
OCDF	5.0	20	100	400	2000
Internal Standards					
2,3,7,8-TCDD-$^{13}C_{12}$	100	100	100	100	100
2,3,7,8-TCDF-$^{13}C_{12}$	100	100	100	100	100
1,2,3,7,8-PeCDD-$^{13}C_{12}$	100	100	100	100	100
1,2,3,7,8-PeCDF-$^{13}C_{12}$	100	100	100	100	100
2,3,4,7,8-PeCDF-$^{13}C_{12}$	100	100	100	100	100
1,2,3,4,7,8-HxCDD-$^{13}C_{12}$	100	100	100	100	100
1,2,3,6,7,8-HxCDD-$^{13}C_{12}$	100	100	100	100	100
1,2,3,4,7,8-HxCDF-$^{13}C_{12}$	100	100	100	100	100
1,2,3,6,7,8-HxCDF-$^{13}C_{12}$	100	100	100	100	100
1,2,3,7,8,9-HxCDF-$^{13}C_{12}$	100	100	100	100	100
2,3,4,6,7,8-HxCDF-$^{13}C_{12}$	100	100	100	100	100
1,2,3,4,6,7,8-HpCDD-$^{13}C_{12}$	100	100	100	100	100
1,2,3,4,6,7,8-HpCDF-$^{13}C_{12}$	100	100	100	100	100
1,2,3,4,7,8,9-HpCDF-$^{13}C_{12}$	100	100	100	100	100
OCDD-$^{13}C_{12}$	200	200	200	200	200
Recovery Standards					
1,2,3,4-TCDD-$^{13}C_{12}$	100	100	100	100	100
1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	100	100	100	100	100
Enrichment Efficiency Standard					
2,3,7,8-TCDD-$^{37}C1_4$	0.5	2	10	40	200

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TABLE 5. 2,3,7,8-TCDD Equivalency Factors (TEFs) for the Polychlorinated Dibenzo-p-dioxins and Dibenzofurans

Number	Compound(s)	TEF
1	2,3,7,8-TCDD	1.00
2	1,2,3,7,8-PeCDD	0.50
3	1,2,3,6,7,8-HxCDD	0.1
4	1,2,3,7,8,9-HxCDD	0.1
5	1,2,3,4,7,8-HxCDD	0.1
6	1,2,3,4,6,7,8-HpCDD	0.01
7	OCDD	0.001
8	* Total - TCDD	0.0
9	* Total - PeCDD	0.0
10	* Total - HxCDD	0.0
11	* Total - HpCDD	0.0
12	2,3,7,8-TCDF	0.10
13	1,2,3,7,8-PeCDF	0.05
14	2,3,4,7,8-PeCDF	0.5
15	1,2,3,6,7,8-HxCDF	0.1
16	1,2,3,7,8,9-HxCDF	0.1
17	1,2,3,4,7,8-HxCDF	0.1
18	2,3,4,6,7,8-HxCDF	0.1
19	1,2,3,4,6,7,8-HpCDF	0.01
20	1,2,3,4,7,8,9-HpCDF	0.01
21	OCDF	0.001
22	* Total - TCDF	0.0
23	* Total - PeCDF	0.0
24	* Total - HxCDF	0.0
25	* Total - HpCDF	0.0

*Excluding the 2,3,7,8-substituted congeners.

Reference: 1989 ITEFs

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