

ATTACHMENT
LABORATORY ANALYSIS REPORT
PCDDs & PCDFs

CITY_21MC100_005390292



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DETERMINATION OF PCDD/PCDF LEVELS

Prepared for:
ATC Associates
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This report contains 24 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 Project Number: 19125-0297

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REPORT OF: CHEMICAL ANALYSES

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PROJECT: PCDD/PCDF ANALYSES**DATE:** April 5, 2002

ISSUED TO: ATC Associates
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REPORT NO: 02-1055016**INTRODUCTION**

This report presents the results from the analyses performed on three samples that were submitted by a representative of ATC Associates. The samples were analyzed for the presence or absence of polychlorinated dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs) using a modified version of USEPA Method 8290 as described below.

SAMPLE IDENTIFICATION

<u>Client ID</u>	<u>Sample Type</u>	<u>Date Received</u>	<u>Pace ID</u>
ATC-0308-89M-DX1	Wipe	03/12/02	103384756
ATC-0308-89M-DX2	Wipe	03/12/02	103384764
ATC-0308-89M-DX3	Wipe	03/12/02	103384772

METHODOLOGY**Sample Extraction**

Each sample was spiked with $^{13}\text{C}_{12}$ -labeled PCDD/PCDF internal standards (Table 1) and extracted with toluene in a Soxhlet extractor. 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- $^{37}\text{Cl}_4$ 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.

Each 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.

Each 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 to 1 mL under a stream of dry nitrogen and applied to the top of a chromatography column containing 1 g of 5% AX-21 activated carbon in silica gel. The column was eluted with two 2-mL portions of hexane, 2 mL of cyclohexane/methylene chloride (50:50 v/v) and cyclohexane/methanol/toluene (75:20:5 v/v) in the forward direction, and then with toluene in the reverse direction. The toluene fraction was collected, concentrated, spiked with recovery standards (1,2,3,4-TCDD-¹³C₁₂ and 1,2,3,7,8,9-HxCDD-¹³C₁₂) and taken to a final volume of 20 μ L.

PCDD/PCDF Analyses

Each 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 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 ($M/\Delta M$, 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 8290. 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 PCDE 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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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:

$$Rf = \frac{A_n \times Q_{is}}{A_{is} \times Q_n}$$

where:

Rf = Response factor
 A_n = Sum of integrated areas for native isomer
 Q_{is} = Quantity of labeled internal standard
 A_{is} = Sum of integrated areas for labeled internal standard
 Q_n = Quantity of native isomer

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

$$C = \frac{A_n \times Q_{is}}{A_{is} \times W \times Rf}$$

where:

C = Concentration of target isomer or congener class
 A_n = Sum of integrated areas for the target isomer or congener class
 Q_{is} = Quantity of labeled internal standard added to the sample
 A_{is} = 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 toxic equivalence of each sample was calculated using the factors listed in Table 5.

A limit of detection (LOD) based on producing a signal that is 2.5 times the noise level, was calculated for each undetected 2,3,7,8-substituted isomer of any tetra through octa chlorinated congener class. The noise heights used to calculate the detection limits were measured at the retention time of the specific isomer. The formula used for calculating the LOD is:

$$\text{LOD} = \frac{H_n \times Q_{is} \times 2.5}{H_{is} \times W \times R_f}$$

where:

LOD = Single isomer limit of detection
 H_n = Sum of noise heights at native isomer retention time
 Q_{is} = Quantity of labeled internal standard
 H_{is} = Sum of peak heights for labeled internal standard
 W = Sample amount
 R_f = Response factor

The recovery of the 2,3,7,8-TCDD-³⁷Cl₄ enrichment efficiency standard and each ¹³C₁₂-labeled internal standard, relative to either 1,2,3,4-TCDD-¹³C₁₂ or 1,2,3,7,8,9-HxCDD-¹³C₁₂, was calculated using the following equation:

$$\%R = \frac{A_{is} \times Q_{rs} \times 100\%}{R_{fr} \times A_{rs} \times Q_{is}}$$

where:

%R = Percent recovery of labeled internal standard
 A_{is} = Sum of integrated areas of labeled internal standard
 Q_{rs} = Quantity of recovery standard
 A_{rs} = Sum of integrated areas of recovery standard
 R_{fr} = Response factor of the specific labeled internal standard relative to the recovery standard
 Q_{is} = Quantity of the labeled internal standard congener added to the sample

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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. 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 40 to 135 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 using a standard solution containing 2,3,7,8-TCDD and the adjacent TCDD isomers. The DB-225 column resolution is checked daily using 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 and end 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 fall to within the ranges specified in Method 8290.

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PROJECT: PCDD/PCDF ANALYSES**DATE: April 5, 2002****PAGE: 7****REPORT NO: 02-1055016****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 extracts ranged from 50-120%. All of the internal standard recoveries for field samples were within the target limits for this method. Also, 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 each sample batch as part of our routine quality control procedures. The results, found at the beginning of Appendix B, show Blank 1523 to contain a trace level of 2,3,7,8-TCDF. This level was below the calibration range of the method. The samples were found not to contain this isomer at the reporting limits. This indicates that the sample processing procedures did not significantly impact the results of the analyses.

Laboratory spike samples were also prepared with each sample batch by extracting clean wipes that had been fortified with native standard materials. The results, found at the end of Appendix B, show that the spiked native compounds were recovered at 68-121%. These results indicate a generally high degree of accuracy for these samples. It should be noted that one isomer in spike 1537 was recovered slightly below the target range for this method. The affected point is flagged "P" on the data summary sheet.

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REMARKS

The sample extracts 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
<u>Recovery Standards</u>	
1,2,3,4-TCDD- ¹³ C ₁₂	2.0
1,2,3,7,8,9-HxCDD- ¹³ C ₁₂	2.0
<u>Enrichment Efficiency Standard</u>	
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/ Δ M, 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**

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

CDDs = Chlorinated Dibenzo-p-dioxins

CDFs = Chlorinated Dibenzofurans

CDPEs = Chlorinated Diphenylethers

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TABLE 4. High Resolution Calibration Solutions

Native CDDs/CDFs	CS1	Concentration (pg/uL)			
		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 C ₁₄	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

REPORT OF LABORATORY ANALYSIS

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