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Polychiorinated Dibenzofurans (PCDF) and Polychlorinated Dibenzo-p-Dioxins (PCDD) in Utility Transformers and Capacitors

Volume 3

Prepared by
Radian Corporation
Austin, Texas

MONS 218113

REPORT SUMMARY

SUBJECTS: Hazardous/toxic substances / Transmission: Substations / Distribution: Substations

TOPICS: PCB; Capacitors; Chemical analysis; PCDF-PCDD; Transformers; Insulating oil

AUDIENCE: Environmental managers / Distribution engineers

Polychlorinated Dibenzofurans (PCDF) and Polychlorinated Dibenzo-p-Dioxins (PCDD) in Utility Transformers and Capacitors

Volumes 1–3

Many health effects originally attributed to PCBs appear to result from their pyrolysis and combustion by-products, PCDF and PCDD. This study developed improved techniques for characterizing these products and showed that PCDF and PCDD do not form during catastrophic arcing failure in transformers and capacitors.

BACKGROUND

Partial oxidation of askarels—mixtures of polychlorinated biphenyls (PCB) and tri- and tetrachlorobenzenes—produces polychlorinatec dibenzofurans (PCDF) and polychlorinated dibenzo-p-dioxins (PCDD). However, analytic techniques for separating the active components in these mixtures are not yet mature. Information is alao sparse on the quantity and types of PCDF and PCDD in as-manufactured PCB and on their formation during normal operation or arcing failure in utility equipment. Because of these uncertainties, utilities and regulators assume the worst-case PCDF-PCDD formation when there is a PCB-related accident and thereby overstate potential toxicity.

OBJECTIVE

To improve techniques for measuring PCDF-PCDD in the presence of PCB and in utility transformers and capacitors.

APPROACH

Researchers developed a rationale for selecting transformers and capacitors to be sampled for PCDF-PCDD content. They also simulated catastrophic transformer failure through arcing in askarel and contaminated mineral oil to investigate PCDF-PCDD formation. Several laboratories then performed round-robin gas chromatography-mass spectrometry analyses of PCDF-PCDD samples using specially prepared carbon-13 (^{13}C) PCDF spiking compounds. After the first set of analyses, each laborstory modified its techniques snd analyzed a larger group of samples from utility equipment. Another organization statistically analyzed all laboratory results.

RESULTS

These three volumes are the first release in a six-volume report of PCDF-PCDD analytic findings. Results from all participating laboratories correlated remarkaoly well. Some of those results follow.

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- Making use of the ^{13}C compounds, researchers successfully separated the components in several closely eluting pairs of physiologically active PCDF and less-active components.
- The researchers found no PCDF or PCDD in the arcing of PCB, tri- and tetrachlorobenzene, or contaminated mineral oil.
- Analyses of samples from utility equipment suggested that the PCDF-PCDD present did not result from high-load, high-temperature operation.

Volume 1 presents researchers' sampling recommendations for investigating PCDF-PCDD in utility equipment. It also describes arcing simulations and results. Volumes 2 and 3 describe several analytic techniques, with volume 3 including a report on the synthesis of new PCDF spiking compounds. Later volumes will complete the descriptions of laboratory techniques, present comprehensive statistical analyses, and provide an executive summary.

EPRI PERSPECTIVE

This project appears to be a landmark in PCDF-PCDD analysis. One important result is the synthesis of new PCDFs for use as spiking compounds in gas chromatogrephy-mass spectrometry analysis. The ability to separate many of the active PCDF compounds from more-innocuous materials is also of great value. The fact that laboratory results correlated so well indicates that researchers can have great flexibility in selecting techniques and developing facilities.

Utilities benefit from this work in several ways. The study provides data showing that little or no PCDF-PCDD forms in electrical equipment either during normal operation or during arcing failure. Moreover, improved analytic techniques make it clear that past predictions of toxicity have been unnecessarily high.

PROJECTS

RP2028-5, RP2028-8, RP2028-9
EPRI Project Managers: Gil Addis; Jacques Guertin
Electrical Systems Division; Energy Analysis and Environment Division
Contractors: Radian Corporation; General Electric Company; Illinois Institute of Technology Research Institute

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Radian Corporation
Austin, Texas

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ABSTRACT

Radian Corporation is one of several participants in an interlaboratory study designed to develop and validate methodology for the analysis of polychlorodibenzo-p-dioxins (PCDD) and polychlorodibenzofurans (PCDF) in electrical equipment dielectric fluids. Radian's participation in this program focused on two parts:

- Task 1: Preparation of Samples, and
- Task 3: Gas Chromatography/Mass Spectrometry (GC/MS) Analysis.

As part of Task 1 a number of native and carbon-13 labeled PCDFs were synthesized for use as standards, spiking solutions and internal surrogate standards. Synthetic dielectric fluid samples (Baseline Liquids) consisting of unused Aroclors, a tri/tetrachlorobenzene sample and an aged mineral oil were prepared and spiked with known levels of carbon-13 standards and native PCDD/PCDFs. Several samples were spiked with "blind" levels of a variety of PCDD/PCDFs including some coeluters. These samples and standards were distributed to the contracting laboratories.

Task 3 began with a GC/MS crosscheck of the Baseline Liquids. Evaluation of the results and modification of the methodology enabled a detailed, statistically meaningful study in the second part of Task 3: the Round Robin Analysis. This part of the study evaluated the methodology in several areas: chromatographic resolution, linearity of response over a wide concentration range and the accuracy and precision of the analysis.

Results of this study indicated that PCDD/PCDFs could be accurately measured in a wide variety of dielectric fluid samples. In a few cases interferences made measurement of some analytes difficult.

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PROJECT SUMMARY

Radian Corporation was one of five participants in an interlaboratory study sponsored by the Electric Power Research Institute under EPRI Contract RP2028-5. The study was designed to develop and validate analytical methodologies and to determine the levels of polychlorodibenzo-p-dioxins (PCDD) and polychlorodibenzofurans (PCDF) in transformer and capacitor insulating fluids. Radian's participation in this program focused on two parts; preparation of samples, and gas chromatography/mass spectrometry (GC/MS) analysis.

As part of the preparation of samples for analysis, Radian synthesized 1,2,3,7,8-pentachlorodibenzofuran (PnCDF), 1,2,3,4,7,8-hexachlorodibenzofuran (HxCDF), 1,2,3,4,6,7,8-heptachlorodibenzofuran (HpCDF), 2,3,7,8-tetrachlorodibenzofuran-$^{13}C_{12}$ (TCDF-^{13}C), 1,2,3,7,8-PnCDF-^{13}C, and 1,2,3,4,7,8-HxCDF-^{13}C. Additional compounds already available at Radian, 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD), 2,3,7,8-TCDD-^{13}C, 2,3,7,8-TCDF, octachlorodibenzo-p-dioxin $^{13}C_{12}$ (OCDD-^{13}C), 2,3,4,8-TCDF, 2,3,4,7,8-PnCDF, 1,2,3,6,7,8-HxCDF, 2,3,4,6,7,8-HxCDF and octachlorodibenzofuran (OCDF) were used also in spiking samples for analysis, calibration standards, and internal surrogate standards.

Synthetic dielectric fluids (baseline liquids: Aroclor 1016, Aroclor 1242, and Aroclor 1260, a tri/tetrachlorobenzene mixture, and aged mineral oil) were prepared and spiked with known levels of carbon-13 and native (carbon-12) PCDDs and PCDFs. Additional samples of the baseline liquids were prepared with a variety of PCDDs and PCDFs spiked at levels unknown to the analytical participants.

The interlaboratory study began with the GC/MS analysis of the baseline liquids. Evaluation of the results and modification of the methodology enabled a detailed, statistically meaningful interlaboratory study then to be conducted. This second part of the GC/MS interlaboratory study tested the analytical methodology in several areas: chromatographic resolution, linearity of response over a wide concentration range, and the accuracy and precision of the analysis. Three baseline liquids and seven dielectric fluids obtained from in-service equipment were analyzed.

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Chromatographic resolution was tested by analyzing a standard mixture containing the more toxic PCDD/PCDF isomers and co-eluting components. The test mixture contained 18 PCDD and PCDF isomers. This test mixture was analyzed prior to the analysis of the samples and daily during the analysis to demonstrate and document the integrity of the analytical system.

Linearity of response for the GC/MS was determined at five points from 40 ng/mL to 7500 ng/mL by triplicate determinations of 2,3,7,8-TCDD, 2,3,7,8-TCDF, 1,2,3,7,8-PnCDF, 2,3,4,7,8-PnCDF, 1,2,3,4,7,8-HxCDF, 2,3,4,6,7,8-HxCDF, 1,2,3,4,6,7,8-HpCDF and OCDF at each concentration level. Response was determined relative to the carbon-13 analogs. The two PnCDF isomers were calibrated relative to 1,2,3,7,8-PnCDF-^{13}C. The two HxCDF isomers and the HpCDF were compared to 1,2,3,4,7,8-HxCDF-^{13}C. The OCDF response was calibrated relative to OCDD-^{13}C. Four complete calibration curves were generated; statistical analyses were performed on each curve. The curves were very linear throughout the range tested and exhibited correlation coefficients greater than 0.999 in almost all cases.

The analysis of three baseline fluids and seven in-service fluids was structured to provide data to evaluate extraction and clean-up precision and analytical precision. Three aliquots of each sample were extracted and prepared for analysis on separate days. Each extract was analyzed in triplicate to yield a total of nine determinations for each of the ten samples. Accuracy of the analysis was evaluated by comparison of the baseline liquid analytical results with the "blind" spiked levels. For the aged mineral oil, experimentally determined concentrations varied from the spiked values by 0.6% to 8.5%. The spiked Aroclor 1016 experimental results varied from the spiked values by 1.9% to 15.1%. For the Aroclor 1260, experimentally determined values did not agree with spiked values due to high levels of PCDFs in the fluid prior to spiking. The Aroclor 1260 was analyzed subsequently to quantitate the inherent PCDF levels. Overall precision for the twenty-seven discrete baseline fluids determinations was 4.0%. The overall precision for nine separate extractions of the baseline fluids was 4.2%.

The results of the analysis of the in-service samples could be evaluated for precision. Precision of the nine determinations for each in-service sample ranged from 7.6% to 12.8%. Precision of the three extractions of each in-service sample ranged from 6.3% to 16.4%. Surrogate recovery using 2,3,7,8-TCDD-^{13}C, ranged from 80% to 200%. The in-service fluids included transformer oils from

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varied types of service, including an arc furnace transformer, an ESP transformer, and a capacitor oil. The in-service fluids were selected to provide wide ranges of operational service and of dielectric fluid composition. Consequently, levels of some PCDD/PCDF isomers in those fluids ranged from less than three parts-per-billion to parts-per-million.

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Section 1

INTRODUCTION

Radian Corporation is one of five laboratories participating in the Electric Power Research Institute (EPRI) program RP2028-5, "Formation of PCDD and PCDF in Askarel and Contaminated Mineral Oil Equipment." The objectives of this program are:

- To develop and validate reliable analytical techniques to determine levels of polychlorinated dibenzodioxins (PCDD) and polychlorinated dibenzofurans (PCDF) in the dielectric fluids of operating electrical equipment; and
- To attempt to correlate these levels to operating conditions of the equipment.

This report describes Radian's completion of Task 1: Preparation of Samples, parts a, Standard Materials, and b, Baseline Samples, and Task 3: Gas Chromatography/Mass Spectroscopy (GC/MS) Analysis, parts a, b, and c, GC/MS Crosscheck, Assessment of Methodology and Round Robin Analysis.

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Section 2

SCOPE OF WORK: TASK 1, PREPARATION OF SAMPLES

INTRODUCTION

This section describes the scope of Radian's portion of Task 1: Preparation of Samples. The purpose of this task was to provide both ^{13}C-labeled and unlabeled native (^{12}C) PCDD and PCDF congeners with which samples of insulating fluids were spiked. Carbon-13 containing congeners are not present in significant quantities in naturally occuring materials and are clearly distinguishable from native congeners by mass spectrometry. Spiking samples with labeled congeners provides unique internal standards for accurate analyses.

STANDARD MATERIALS

Task 1a required that Radian obtain either by purchase or preparation a variety of native and ^{13}C-labeled PCDD and PCDF congeners. These materials were used to prepare spiking solutions and analytical standards for use in the analysis of Baseline and In-Service samples. QC analyses were conducted on all solutions to assure and document accuracy.

BASELINE SAMPLES

Task 1b required that Radian obtain samples of five liquids and spike them with measured quantities of PCDD and PCDF congeners. Approximately one gram samples of these dielectric fluids were shipped to the perticipating analytical laboratories. Four of the liquids approximated as closely as possible unused liquids formerly employed in the manufacture of electrical equipment. The fifth liquid (mineral oil) was obtained from an aged transformer and spiked with Aroclor 1260 and tri/tetrachlorobenzene (T/TCB) approximating the condition of a used contaminated oil.

Section 3

RESULTS SUMMARY: TASK 1, PREPARATION OF SAMPLES

INTRODUCTION

This section describes the results of Radian's completion of Task 1a, preparation of standard materials, and Task 1b, preparation of baseline samples.

STANDARD MATERIALS

In order to obtain the PCDF congeners required for this study Radian synthesized the following materials:

1,2,3,7,8-Pentachlorodibenzofuran (PnCDF)
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)
2,3,7,8-Tetrachlorodibenzofuran-$^{13}C_{12}$ (TCDF-^{13}C)
1,2,3,7,8-Pentachlorodibenzofuran-$^{13}C_{12}$ (PnCDF-^{13}C)
1,2,3,4,7,8-Hexachlorodibenzofuran-$^{13}C_{12}$ (HxCDF-^{13}C)

Several congeners in Radian's PCDD/PCDF Standards Inventory were donated to the program. These congeners were:

2,3,7,8-Tetrachlorodibenzo-p-dioxin
2,3,7,8-Tetrachlorodibenzo-p-dioxin-$^{13}C_{12}$
2,3,7,8-Tetrachlorodibenzofuran
Octachlorodibenzo-p-dioxin-$^{13}C_{12}$
2,3,4,8-Tetrachlorodibenzofuran
2,3,4,7,8-Pentachlorodibenzofuran
1,2,3,6,7,8-Hexachlorodibenzofuran
2,3,4,6,7,8-Hexachlorodibenzofuran
Octachlorodibenzofuran

A mixture of congeners was obtained from Professor Christoffer Rappe of the University of Umea, Sweden. This semi-quantitative mixture, the "toxic cocktail", contained the following isomers:

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1,3,4-Trichlorodibanzofuran
2,3,4,8-Tatrachlorodibanzofuran
2,3,4,7,8-Pentachlorodibenzofuran
1,2,3,4,8-Pentachlorodibenzofuran
1,2,3,4,7,9-Hexachlorodibenzofuran
1,2,3,4,6,7,8-Heptachlorodibenzofuran
Octachlorodibenzofuran

All the compounds prepared or obtained by Radian were made up as stock spiking solutions at either 50 ug/mL or 100 ug/mL. These solutions were further dilutad to produce standard solutions for distribution to the participating analytical laboratories. Tabla 3-1 lists the standards provided by Radian. One ampule of each isomer and an ampule of each mixture was provided to each participating laboratory. Additional ampules were provided on request.

BASELINE SAMPLES

Five dielectric fluids were selected to be spiked with the PCDD/PCDF congeners for the baseline analyses. These fluids were:

- Arocior 1016,
- Arocior 1242,
- Arocior 1260,
- Tri/Tetrachiorobenzene, and
- Aged Mineral Oil.

Arocior 1016, 1242 and 1260 were purchased from Foxboro/Analabs. The tri/tetrachlorobenzena mixture (T/TCB) was prepared at Raolan with reagents purchased from Aldrich Chemical Company. Its composition, compared to a "typical" T/TCB mixture, as per Dr. T. O. Rouse (General Electric), is as follows:

Isomer	Typical T/TCB	Radian T/TCB
1,2,4-Trichlorobenzene	49.5%	51.2%
1,2,3-Trichlorobenzene	13.0	13.4
1,2,4,5-Tetrachlorobenzena	2.6	2.6
1,2,3,4-Tetrachlorobenzene	27.4	28.4
Pentachlorobenzene	4.2	4.4
	96.7%	100.0%

Table 3-1

STANDARD SOLUTIONS

I.D. No.	Description	Concentration
E729-14-02	2,3,7,8-Tetrachlorodibenzofuran (TCDF)-^{13}C	100 ng/mL
E729-06-04	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)-^{13}C	100 ng/mL
E729-15-02	1,2,3,7,8-Pentachlorodibenzofuran (PnCDF)-^{13}C	100 ng/mL
E729-16-02	Octachlorodibenzo-p-dioxin (OCDD)-^{13}C	40 ng/mL
E729-05-04	2,3,7,8-Tetrachlorodibenzofuran	100 ng/mL
E729-06-02	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100 ng/mL
E729-05-01	1,2,3,7,8-Pentachlorodibenzofuran	100 ng/mL
E729-05-02	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	100 ng/mL
E729-05-03	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	100 ng/mL
E729-06-01	Radian PCDF/TCDD Mixture	
	2,3,7,8-TCDF	100 ng/mL
	2,3,7,8-TCDD	100 ng/mL
	1,2,3,7,8-PnCDF	100 ng/mL
	1,2,3,4,7,8-HxCDF	100 ng/mL
	1,2,3,4,6,7,8-HpCDF	100 ng/mL
E729-07-02	Professor Reppe's "Toxic Cocktail"	
	1,3,4-TrCDF	~ 116 ng/mL
	2,3,4,8-TCDF	~ 100 ng/mL
	2,3,4,7,8-PnCDF	~ 100 ng/mL
	1,2,3,4,8-PnCDF	~ 100 ng/mL
	1,2,3,4,7,9-HxCDF	~ 100 ng/mL
	1,2,3,4,6,7,8-HpCDF	~ 100 ng/mL
	OCDF	~ 160 ng/mL

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Table 3-1

STANDARD SOLUTIONS (continued)

I.D. No.	Description	Concentration
E729-55-01	Calibration Mixture I	
	Native Congeners	
	2,3,7,8-TCDD	7.5 ug/mL
	2,3,7,8-TCDF	7.5 ug/mL
	1,2,3,7,8-PnCDF	7.5 ug/mL
	2,3,4,7,8-PnCDF	7.5 ug/mL
	1,2,3,4,7,8-HxCDF	7.5 ug/mL
	2,3,4,6,7,8-HxCDF	7.5 ug/mL
	1,2,3,4,6,7,8-HpCDF	7.5 ug/mL
	OCDF	7.5 ug/mL
	Labeled Congeners	
	2,3,7,8-TCDD-$^{13}C_{12}$	500 ng/mL
	2,3,7,8-TCDF-$^{13}C_{12}$	500 ng/mL
	1,2,3,7,8-PnCDF-$^{13}C_{12}$	500 ng/mL
	1,2,3,4,7,8-HxCDF-$^{13}C_{12}$	500 ng/mL
	OCDD-$^{13}C_{12}$	200 ng/mL
E729-35-03	Calibration Mixture II	
	Native Congeners	
	2,3,7,8-TCDD	2.5 ug/mL
	2,3,7,8-TCDF	2.5 ug/mL
	1,2,3,7,8-PnCDF	2.5 ug/mL
	2,3,4,7,8-PnCDF	2.5 ug/mL
	1,2,3,4,7,8-HxCDF	2.5 ug/mL
	2,3,4,6,7,8-HxCDF	2.5 ug/mL
	1,2,3,4,6,7,8-HpCDF	2.5 ug/mL
	OCDF	2.5 ug/mL

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Table 3-1

STANDARD SOLUTIONS (continued)

I.D. No.	Description	Concentration
	Labeled Congeners	
	2,3,7,8-TCDD-$^{13}C_{12}$	500 ng/mL
	2,3,7,8-TCDF-$^{13}C_{12}$	500 ng/mL
	1,2,3,7,8-PnCDF-$^{13}C_{12}$	500 ng/mL
	1,2,3,4,7,8-HxCDF-$^{13}C_{12}$	500 ng/mL
	OCDD-$^{13}C_{12}$	200 ng/mL
E729-55-04	Calibration Mixture III	
	Native Congeners	
	2,3,7,8-TCDD	500 ng/mL
	2,3,7,8-TCDF	500 ng/mL
	1,2,3,7,8-PnCDF	500 ng/mL
	2,3,4,7,8-PnCDF	500 ng/mL
	1,2,3,4,7,8-HxCDF	500 ng/mL
	2,3,4,6,7,8-HxCDF	500 ng/mL
	1,2,3,4,6,7,8-HpCDF	500 ng/mL
	OCDF	500 ng/mL
	Labeled Congeners	
	2,3,7,8-TCDD-$^{13}C_{12}$	500 ng/mL
	2,3,7,8-TCDF-$^{13}C_{12}$	500 ng/mL
	1,2,3,7,8-PnCDF-$^{13}C_{12}$	500 ng/mL
	1,2,3,4,7,8-HxCDF-$^{13}C_{12}$	500 ng/mL
	OCDD-$^{13}C_{12}$	200 ng/mL
E729-55-05	Calibration Mixture IV	
	Native Congeners	
	2,3,7,8-TCDD	150 ng/mL
	2,3,7,8-TCDF	150 ng/mL
	1,2,3,7,8-PnCDF	150 ng/mL
	2,3,4,7,8-PnCDF	150 ng/mL
	1,2,3,4,7,8-HxCDF	150 ng/mL

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Table 3-1

STANDARD SOLUTIONS (continued)

I.D. No.	Description	Concentration
	2,3,4,6,7,8-HxCDF	150 ng/mL
	1,2,3,4,6,7,8-HpCDF	150 ng/mL
	OCDF	150 ng/mL
	Labeled Congeners	
	2,3,7,8-TCDD-$^{13}C_{12}$	500 ng/mL
	2,3,7,8-TCDF-$^{13}C_{12}$	500 ng/mL
	1,2,3,7,8-PnCDF-$^{13}C_{12}$	500 ng/mL
	1,2,3,4,7,8-HxCDF-$^{13}C_{12}$	500 ng/mL
	OCDD-$^{13}C_{12}$	200 ng/mL
E729-55-06	Calibration Mixture V	
	Native Congeners	
	2,3,7,8-TCDD	40 ng/mL
	2,3,7,8-TCDF	40 ng/mL
	1,2,3,7,8-PnCDF	40 ng/mL
	2,3,4,7,8-PnCDF	40 ng/mL
	1,2,3,4,7,8-HxCDF	40 ng/mL
	2,3,4,6,7,8-HxCDF	40 ng/mL
	1,2,3,4,6,7,8-HpCDF	40 ng/mL
	OCDF	40 ng/mL
	Labeled Congeners	
	2,3,7,8-TCDD-$^{13}C_{12}$	500 ng/mL
	2,3,7,8-TCDF-$^{13}C_{12}$	500 ng/mL
	1,2,3,7,8-PnCDF-$^{13}C_{12}$	500 ng/mL
	1,2,3,4,7,8-HxCDF-$^{13}C_{12}$	500 ng/mL
	OCDD-$^{13}C_{12}$	200 ng/mL

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Table 3-1

STANDARD SOLUTIONS (continued)

I.D. No.	Description	Concentration
E729-56-01	Calibration Mixture VI	
	Native Congeners	
	2,3,7,8-TCDD	10 ng/mL
	2,3,7,8-TCDF	10 ng/mL
	1,2,3,7,8-PnCDF	10 ng/mL
	2,3,4,7,8-PnCDF	10 ng/mL
	1,2,3,4,7,8-HxCDF	10 ng/mL
	2,3,4,6,7,8-HxCDF	10 ng/mL
	1,2,3,4,6,7,8-HpCDF	10 ng/mL
	OCDF	10 ng/mL
	Labeled Congeners	
	2,3,7,8-TCDD-$^{13}C_{12}$	500 ng/mL
	2,3,7,8-TCDF-$^{13}C_{12}$	500 ng/mL
	1,2,3,7,8-PnCDF-$^{13}C_{12}$	500 ng/mL
	1,2,3,4,7,8-HxCDF-$^{13}C_{12}$	500 ng/mL
	OCDD-$^{13}C_{12}$	200 ng/mL
E729-56-02	Calibration Mixture VII	
	Native Congeners	
	2,3,7,8-TCDD	2.5 ng/mL
	2,3,7,8-TCDF	2.5 ng/mL
	1,2,3,7,8-PnCDF	2.5 ng/mL
	2,3,4,7,8-PnCDF	2.5 ng/mL
	1,2,3,4,7,8-HxCDF	2.5 ng/mL
	2,3,4,6,7,8-HxCDF	2.5 ng/mL
	1,2,3,4,6,7,8-HpCDF	2.5 ng/mL
	OCDF	2.5 ng/mL
	Labeled Congeners	
	2,3,7,8-TCDD-$^{13}C_{12}$	500 ng/mL
	2,3,7,8-TCDF-$^{13}C_{12}$	500 ng/mL

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Table 3-1

STANDARD SOLUTIONS (continued)

I.D. No.	Description	Concentration
	1,2,3,7,8-PnCDF-$^{13}C_{12}$	500 ng/mL
	1,2,3,4,7,8-HxCDF-$^{13}C_{12}$	500 ng/mL
	OCDD-$^{13}C_{12}$	200 ng/mL

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The "aged mineral oil" sample was obtained from Radian's Transformer Oil Analysis Laboratory. This oil was analyzed at Radian and found to be free of PCBs. It was analyzed also by Dr. T. O. Rouse at General Electric and determined to be Exxon Univolt 60/62.

BASELINE LIQUIDS - GC/MS CROSSCHECK

All of the Baseline liquids were dissolved in hexane at a level of 0.25 g/mL in order to reduce viscosity. Each was spiked with measured levels of native and ^{13}C-labeled PCDD/PCDF congeners. In addition, the aged mineral oil was spiked with approximately 500 ppm each, Aroclor 1260 and T/TCB and "blind" spikes of several native congeners. Table 3-2 lists each sample with spiking details. Approximate one gram samples were sealed in glass ampules and shipped to the participating laboratories. Adequate amounts were retained as archival samples and as a supply of additional standards for the participating laboratories.

BASELINE LIQUIDS - ROUND ROBIN ANALYSIS

To determine the accuracy and precision of PCDD/PCDF measurements, a selection of Baseline Liquids were re-analyzed in the Round Robin Analysis portion of the program. The Baseline Liquids were dissolved in hexane at levels of 0.18 - 0.20 grams/mL and spiked with measured levels of ^{13}C-labeled congeners. In this instance, however, a selection of native congeners were spiked "blind" (at levels known only to the person performing the spiking). Additionally, a set of samples were prepared with no native congener spikes in order to determine background PCDD/PCDF levels. Table 3-3 describes these samples and reveals the spiking levels. The seven "In-Service" Liquids were spiked with ^{13}C-labeled congeners at Battelle Memorial Institute, Columbus, Ohio, using stock solutions provided by Radian Corporation.

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Table 3-2

BASELINE FLUID SAMPLES - GC/MS CROSSCHECK

I.D. No.	Description	Spike	Spiking Level
E729-09-01	Aroclor 1016	2,3,7,8-TCDF-^{13}C	100 ng/g
		1,2,3,7,8-PnCDF-^{13}C	100 ng/g
		2,3,7,8-TCDD-^{13}C	100 ng/g
		OCDD-^{13}C	40 ng/g
		1,2,3,4,7,8-HxCDF	100 ng/g
		1,2,3,4,6,7,8-HpCDF	100 ng/g
E729-10-01	Aroclor 1242	2,3,7,8-TCDF-^{13}C	100 ng/g
		1,2,3,7,8-PnCDF-^{13}C	100 ng/g
		2,3,7,8-TCDD-^{13}C	100 ng/g
		OCDD-^{13}C	40 ng/g
		1,2,3,4,7,8-HxCDF	100 ng/g
		1,2,3,4,6,7,8-HpCDF	100 ng/g
E729-11-01	Aroclor 1260	2,3,7,8-TCDF-^{13}C	100 ng/g
		1,2,3,7,8-PnCDF-^{13}C	100 ng/g
		2,3,7,8-TCDD-^{13}C	100 ng/g
		OCDD-^{13}C	40 ng/g
		1,2,3,4,7,8-HxCDF	100 ng/g
		1,2,3,4,6,7,8-HpCDF	100 ng/g

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Table 3-2

BASELINE FLUID SAMPLES - GC/MS CROSSCHECK (continued)

I.D. No.	Description	Spike	Spiking Level
E729-12-01	Tri/Tetrachlorobenzenes	2,3,7,8-TCDF-^{13}C	100 ng/g
		1,2,3,7,8-PnCDF-^{13}C	100 ng/g
		2,3,7,8-TCDD-^{13}C	100 ng/g
		OCDD-^{13}C	40 ng/g
		1,2,3,4,7,8-HxCDF	100 ng/g
		1,2,3,4,6,7,8-HpCDF	100 ng/g
E729-08-01	Aged Mineral Oil	2,3,7,8-TCDF-^{13}C	100 ng/g
		1,2,3,7,8-PnCDF-^{13}C	100 ng/g
		2,3,7,8-TCDD-^{13}C	100 ng/g
		OCDD-^{13}C	40 ng/g
		2,3,7,8-TCDD	40 ng/g
		2,3,7,8-TCDF	20 ng/g
		1,2,3,7,8-PnCDF	40 ng/g
		1,2,3,4,7,8-HxCDF	120 ng/g
		1,2,3,4,6,7,8-HpCDF	0 ng/g

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Table 3-2

BASELINE FLUID SAMPLES - GC/MS CROSSCHECK (continued)

I.D. No.	Description	Spike	Spiking Level
E729-08-01	Aged Mineral Oil	Professor Rappe's "Toxic Cocktail"	
		OCDF	- 160 ng/g
		1,2,3,4,7,9-HxCDF	- 100 ng/g
		2,3,4,7,8-PnCDF	- 100 ng/g
		1,2,3,4,8-PnCDF	- 100 ng/g
		2,3,4,8-TCDF	- 100 ng/g
		1,3,4-TrCDF	- 116 ng/g
		Aroclor 1260	510 ug/g
		Tri/Tetrachlorobenzenes	530 ug/g

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Table 3-3

BASELINE FLUID SAMPLES - ROUND ROBIN ANALYSIS

I.D. No.	Description	Spike	Spiking Level
E729-53-01	Mineral Oil	Aroclor 1260	640 ug/g
		Tri/Tetrachlorobenzenes	1250 ug/g
		2,3,7,8-TCDD-^{13}C	100 ng/g
		2,3,7,8-TCDF-^{13}C	100 ng/g
		1,2,3,7,8-PnCDF-^{13}C	100 ng/g
		1,2,3,4,7,8-HxCDF-^{13}C	100 ng/g
		OCDD-^{13}C	40 ng/g
		2,3,7,8-TCDD	25 ng/g
		2,3,7,8-TCDF	100 ng/g
		2,3,4,8-TCDF	100 ng/g
		1,2,3,7,8-PnCDF	150 ng/g
		2,3,4,7,8-PnCDF	150 ng/g
		1,2,3,4,7,8-HxCDF	250 ng/g
		1,2,3,6,7,8-HxCDF	50 ng/g
		2,3,4,6,7,8-HxCDF	175 ng/g
		1,2,3,4,6,7,8-HpCDF	400 ng/g
		OCDF	0 ng/g

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Table 3-3

BASELINE FLUID SAMPLES - ROUND ROBIN ANALYSIS (continued)

I.D. No.	Description	Spike	Spiking Level
E729-53-02	Aroclor 1016	2,3,7,8-TCDD-^{13}C	100 ng/g
		2,3,7,8-TCDF-^{13}C	100 ng/g
		1,2,3,7,8-PnCDF-^{13}C	100 ng/g
		1,2,3,4,7,8-HxCDF-^{13}C	100 ng/g
		OCDD-^{13}C	40 ng/g
		2,3,7,8-TCDD	53 ng/g
		2,3,7,8-TCDF	53 ng/g
		2,3,4,8-TCDF	0 ng/g
		1,2,3,7,8-PnCDF	158 ng/g
		2,3,4,7,8-PnCDF	158 ng/g
		1,2,3,4,7,8-HxCDF	0 ng/g
		1,2,3,6,7,8-HxCDF	53 ng/g
		2,3,4,6,7,8-HxCDF	132 ng/g
		1,2,3,4,6,7,8-HpCDF	526 ng/g
		OCDF	132 ng/g
E729-53-05	Aroclor 1260	2,3,7,8-TCDD-^{13}C	100 ng/g
		2,3,7,8-TCDF-^{13}C	100 ng/g
		1,2,3,7,8-PnCDF-^{13}C	100 ng/g
		1,2,3,4,7,8-HxCDF-^{13}C	100 ng/g
		OCDD-^{13}C	40 ng/g

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Table 3-3

BASELINE FLUID SAMPLES - ROUND ROBIN ANALYSIS (continued)

I.D. No.	Description	Spike	Spiking Level
		2,3,7,8-TCDD	111 ng/g
		2,3,7,8-TCDF	833 ng/g
		2,3,4,8-TCDF	194 ng/g
		1,2,3,7,8-PnCDF	111 ng/g
		2,3,4,7,8-PnCDF	111 ng/g
		1,2,3,4,7,8-HxCDF	500 ng/g
		1,2,3,6,7,8-HxCDF	278 ng/g
		2,3,4,6,7,8-HxCDF	56 ng/g
		1,2,3,4,6,7,8-HpCDF	111 ng/g
		OCDF	222 ng/g
		Aroclor 1260	680 ug/g
		Tri/Tetrachlorobenzenes	1320 ug/g
E729-54-02	Mineral Oil	2,3,7,8-TCDD-^{13}C	100 ng/g
		2,3,7,8-TCDF-^{13}C	100 ng/g
		1,2,3,7,8-PnCDF-^{13}C	100 ng/g
		1,2,3,4,7,8-HxCDF-^{13}C	100 ng/g
		OCDD-^{13}C	40 ng/g

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Table 3-3

BASELINE FLUID SAMPLES - ROUND ROBIN ANALYSIS (continued)

I.D. No.	Description	Spike	Spiking Level
E729-54-03	Aroclor 1260	2,3,7,8-TCDD-^{13}C	100 ng/g
		2,3,7,8-TCDF-^{13}C	100 ng/g
		1,2,3,7,8-PnCDF-^{13}C	100 ng/g
		1,2,3,4,7,8-HxCDF-^{13}C	100 ng/g
		OCDD-^{13}C	40 ng/g
E729-54-04	Aroclor 1016	2,3,7,8-TCDD-^{13}C	100 ng/g
		2,3,7,8-TCDF-^{13}C	100 ng/g
		1,2,3,7,8-PnCDF-^{13}C	100 ng/g
		1,2,3,4,7,8-HxCDF-^{13}C	100 ng/g
		OCDD-^{13}C	40 ng/g

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Section 4

EXPERIMENTAL

EXPERIMENTAL

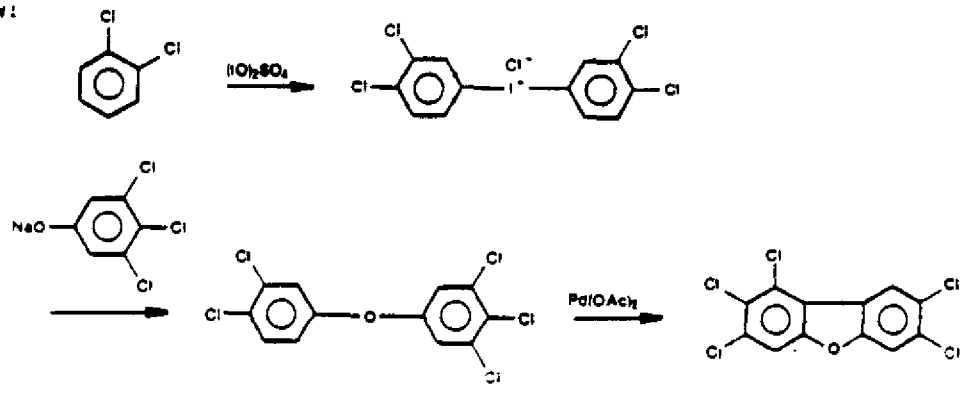
The following sections discuss experimental details of the synthetic procedures and the quality control data. Chromatograms of substances synthesized in this program are included in Appendix A.

SYNTHESIS

Several PCDF congeners were synthesized by Radian using the method of Norstrom, Anderson and Rappe (1). Native congeners were synthesized from basic starting materials at Radian while carbon-13 labeled congeners were prepared from the labeled precursors (polychlorinated diphenylethers) synthesized by Cambridge Isotope Laboratories, Inc. The native congener preparations served as models for the labeled congener preparation as well as for use in the spiking schemes.

1,2,3,7,8-PENTACHLORODIBENZOFURAN (PnCDF)

1,2,3,7,8-PnCDF was prepared by treating o-dichlorobenzene with iodyl sulfate to produce 3,4,3',4'-tetrachlorodiphenyliodonium chloride. This material then was allowed to react with the sodium salt of 3,4,5-trichlorophenol. The resulting 3,4,5,3',4'-pentachlorodiphenyl ether was purified by repeated recrystallization from n-hexane. Reaction with palladium acetate ($Pd(OAc)_2$) produced the desired PnCDF in crude form. Repeated recrystallization yielded approximately 10 mg (34%) of 1,2,3,7,8-PnCDF at a purity level of >98%. This sequence is illustrated below:



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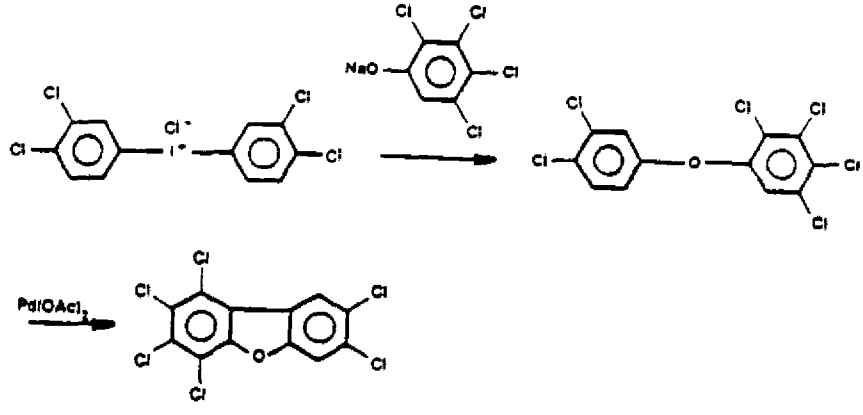
1,2,3,4,7,8-HEXACHLORODIBENZOFURAN (HxCDF)

1,2,3,4,7,8-HxCDF was prepared in the following manner. A portion of the 3,4,3',4'-tetrachlorodiphenyliodonium chloride previously prepared for the synthesis of 1,2,3,7,8-PnCDF was allowed to react with the sodium salt of 2,3,4,5-tetrachlorophenol to give 2,3,4,5,3',4'-hexachlorodiphenyl ether. Purification was accomplished by repeated recrystallization from n-hexane. Reaction with palladium acetate produced the desired HxCDF in crude form. Multiple recrystallizations yielded approximately 10 mg (39%) of 1,2,3,4,7,8-HxCDF at a purity level of >98%. This sequence is illustrated below:



1,2,3,4,6,7,8-HEPTACHLORODIBENZOFURAN (HpCDF)

1,2,3,4,6,7,8-HpCDF was prepared by allowing 1,2,3-trichlorobenzene to react with iodyl sulfate to produce 2,3,4,2',3',4'-hexachlorodiphenyliodonium chloride. Reaction of this material with the sodium salt of 2,3,4,5-tetrachlorophenol gave 2,3,4,5,2',3',4'-heptachlorodiphenyl ether which was purified by repeated recrystallization from n-hexane. Reaction with palladium acetate gave the desired HpCDF in crude form. Multiple recrystallizations yielded approximately 10 mg (46%) of 1,2,3,4,6,7,8-HpCDF at a purity level of >98%. This sequence is illustrated below:

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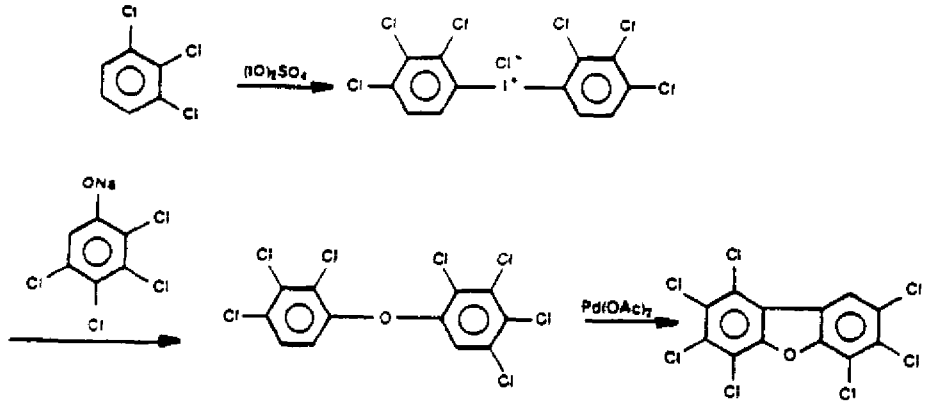
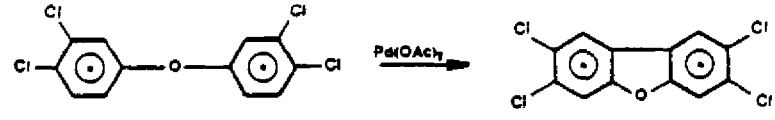


2,3,7,8-TETRACHLORODIBENZOFURAN (TCDF) - $^{13}C_{12}$

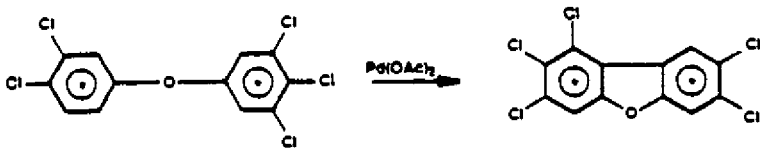
2,3,7,8-TCDF-$^{13}C_{12}$ was prepared by the reaction of 3,4,3',4'-tetrachlorodiphenyl ether-$^{13}C_{12}$ with palladium acetate. Multiple recrystallizations of the product yielded approximately 0.5 mg (14%) of 2,3,7,8-TCDF$^{13}C_{12}$ at a purity level of >96%. This sequence is illustrated below:



* = Carbon - 13 labeled

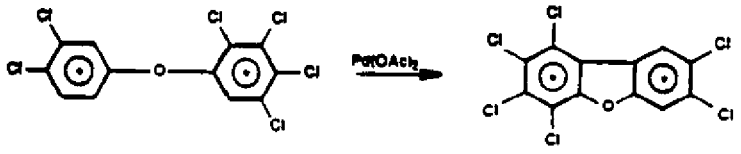
1,2,3,7,8-PENTACHLORODIBENZOFURAN (PnCDF) - $^{13}C_{12}$

1,2,3,7,8-PnCDF-$^{13}C_{12}$ was prepared by the reaction of 3,4,5,3',4'-pentachlorodiphenyl ether-$^{13}C_{12}$ with palladium acetate. Multiple recrystallizations of the product yielded approximately 0.5 mg (27%) of 1,2,3,7,8-PnCDF-$^{13}C_{12}$ at a purity level of >98%. This sequence is illustrated below:



1,2,3,4,7,8-HEXACHLORODIBENZOFURAN (HxCDF) - $^{13}C_{12}$

1,2,3,4,7,8-HxCDF-$^{13}C_{12}$ was prepared by the reaction of 2,3,4,5,3',4'-hexachlorodiphenyl ether-$^{13}C_{12}$ with palladium acetate. Multiple recrystallizations of the product yielded approximately 0.5 mg (41%) of 1,2,3,4,7,8-HxCDF-$^{13}C_{12}$ at a purity of >99%. This sequence is illustrated below:



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QA/QC ANALYSES

Stock solutions of all PCDD/PCDF congeners synthesized for this study were prepared in n-nonane for use in spiking. Dilutions were prepared for use as standards. Separate, identical solutions were prepared on a different day for use as QA/QC samples. Additionally, as the baseline samples were spiked for the GC/MS Crosscheck Study, an identically spiked solution was prepared as a QA/QC check on spiking technique. The samples were analyzed on a gas chromatograph equipped with an electron capture detector (ECD). The chromatographic conditions were:

Column:	OV-101, 2 m x 2 mm I.D.
Oven Temperature:	200°C (250°C for higher congeners)
Detector Temperature:	300°C
Injector Temperature:	250°C
Carrier Gas:	N_2 @ 30 mL/min

Each sample was analyzed three times; the results were averaged and compared with triplicate analyses of the QA/QC samples. These results are tabulated in Tables 4-1 and 4-2.

REFERENCES

1. A. Norstrum, K. Anderson and C. Rappe, Chemosphere, No. 6, 1976, pp. 419-423.

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Table 4-1

STOCK SOLUTIONS QUALITY CONTROL ANALYTICAL DATA

Standard Solution		QC Solution		% Deviation of QC Solution from Standard Solution
I.D. Number, Compound	Area Counts*	I.D. Number, Compound	Area Counts*	
E729-05-04, TCDF	32952	E729-05-09, TCDF	31858	-3.3
E729-05-01, PnCDF	70508	E729-05-05, PnCDF	69580	-1.3
E729-05-02, HxCDF	79715	E729-05-06, HxCDF	81985	2.8
E729-06-04, HpCDF	81585	E729-05-07, HpCDF	82390	0.99
E729-06-04, ^{13}C-TCDD	14215	E729-06-02, TCDD	14205	-0.07
E729-14-02, ^{13}C-TCDF	44963	E729-05-04, TCDF	48534	7.9
E729-15-02, ^{13}C-PnCDF	86402	E729-05-01, PnCDF	94149	9.0
E729-16-02, ^{13}C-OCDD	119676	E729-17-02, OCDD	115145	-3.8
E729-34-01, ^{13}C-HxCDF	2119966	E729-05-02, HxCDF	2130933	0.52

*Average of three determinations.

Table 4-2

BASELINE LIQUID "SPIKE" QC CHECK ANALYTICAL DATA

Solution	Analyte	Area Counts*	% Deviation of Reference Mixture from QC Check
E729-13-01 "Spike" QC Check	^{13}C-TCDF/^{13}C-TCDD	56083	12.6
	^{13}C-PnCDF	95499	17.6
	HxCDF	125140	1.7
	HpCDF	148430	-4.9
	^{13}C-OCDD	112317	2.5
E729-06-01 QC Reference Mixture	TCDF/TCDD	63138	
	PnCDF	112490	
	HxCDF	127280	
	HpCDF	141110	
E729-17-02 OCDD		115145	

*Average of three determinations.

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Section 5

SCOPE OF WORK: TASK 3, GAS CHROMATOGRAPHY/MASS SPECTROMETRY ANALYSIS

INTRODUCTION

This section describes the scope of Radian's portion of Task 3: GC/MS Crosscheck, Assessment of Methodology, and Round Robin Analysis. The purpose of this task was to evaluate the applicability of various analytical methods for determining PCDD/PCDF levels in dielectric fluids, determine their usefulness through a GC/MS crosscheck study and then conduct a Round Robin Analytical Program designed to establish a statistical basis for accuracy and precision.

GC/MS CROSSCHECK AND ASSESSMENT OF METHODOLOGY

Tasks 3a and 3b required that Radian propose methodology to measure PCDD/PCDF in dielectric fluids and assess its usefulness relative to methodologies utilized by other laboratories. Analyses were performed on five baseline liquids composed of various unused Aroclors, a used mineral oil and a tri/tetrachlorobenzene mixture. These samples were spiked with known levels of ^{13}C-labeled surrogates and native PCDFs. The data obtained in this phase of the study was used to identify deficiencies in the methodology, suggest areas of refinement, provide a common basis for the Round Robin Analysis and to assist in detailed planning of the Round Robin Analysis.

ROUND ROBIN ANALYSIS

Task 3c of this study was designed to apply analytical methodology developed in Task 3a for the identification and quantitation of polychlorodibenzo-p-dioxins and polychlorodibenzofurans (PCDD/PCDFs) in dielectric fluids. The first part of this study involved evaluation of selected high resolution capillary chromatography columns for separation of the PCDD/PCDF isomers of interest. The second part of this study was to demonstrate linearity of GC/MS response over a wide range of concentrations. For the third part of this study, three blind-spiked baseline liquids were provided and the accuracy of extraction and GC/MS analysis was assessed. In the fourth part of this study, seven in-service dielectric

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fluids from various electrical equipment were analyzed. For both baseline and in-service liquid analyses, triplicate extractions were performed and triplicate GC/MS determinations were made on each extract. Measurements of multiple extraction and multiple GC/MS determination variability were obtained to evaluate the analytical methodology.

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Section 6

RESULTS SUMMARY: TASK 3, GAS CHROMATOGRAPHY/MASS SPECTROMETRY ANALYSIS

INTRODUCTION

This section describes the results of Radian's completion of Task 3, GC/MS Crosscheck, Assessment of Methodology, and Round Robin Analysis.

SAMPLE EXTRACTION: GC/MS CROSSCHECK

The sample extraction technique proposed and utilized by Radian in this phase of the program was a method that has been applied successfully to the analysis of PCDDs and PCDFs in biota and other environmental samples.

A 1 mL aliquot of the sample was mixed with 50 mL acetonitrile in a 2 L separatory funnel. Deionized water, 900 mL, was added and mixed thoroughly. This mixture was extracted in the separatory funnel three times with 75 mL aliquots of n-hexane. The hexane extracts were combined and filtered through anhydrous sodium sulfate. The dried extract was concentrated to 5 mL using a rotary evaporator and transferred to a centrifuge tube, rinsing the original flask with four 5 mL aliquots of hexane. The extract was then concentrated to 10 mL under a nitrogen stream at 50°C.

The concentrate was transferred with three 5 mL aliquots of hexane to a 1 x 10 cm chromatography column containing 1.0 g silica gel (Type 60, 70-230 mesh) and 6.0 g of a 40% sulfuric acid-silica gel mixture. The extract was eluted with 75 mL of hexane directly onto a second column containing 12.0 g acid alumina (AG 4) and a 1 cm layer of anhydrous sodium sulfate. The silica gel was discarded. The alumina was washed with another 25 mL of hexane. The eluted hexane was discarded. The alumina was eluted with 100 mL of 20% methylene chloride in hexane. This fraction was collected and concentrated using a rotary evaporator. The extract was transferred with three 2 mL aliquots of hexane to a conical vial and concentrated to 100 uL under a nitrogen stream.

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ANALYSIS: GC/MS CROSSCHECK

GC/MS analysis of the sample extracts was performed on a Hewlett-Packard (H-P) 5987 GC/MS system. GC separation was accomplished with a 60 m DB-5 capillary column. Instrumental conditions are detailed in Table 6-1. Quantitation of PCDD/PCDF congeners was accomplished by the isotope dilution method using ^{13}C-labeled internal standards added before extraction. Concentrations were determined from $^{12}C/^{13}C$ ratios of sample analyte to internal standard relative to ^{12}C standards provided for the study. Standard solutions utilized and spiking levels of ^{13}C internal standards and ^{12}C congeners were detailed previously in Tables 3-1 and 3-2. Results of the analyses of the five baseline liquids are summarized in Table 6-2. Reported values are the average of two determinations. Values for 2,3,7,8-TCDF and 2,3,4,8-TCDF are reported as 2,3,7,8-TCDF since resolution was not accomplished on the DB-5 capillary column.

Summary: Aged Mineral Oil

Background interference from contaminants was very high in the aged mineral oil. Interference made quantitation impossible at Cl_3-dibenzofuran and difficult at Cl_4-dibenzofuran. Quantitation of 2,3,7,8-TCDD was not possible due to heavy interference. Cl_5-dibenzofurans through Cl_8-dibenzofurans were recovered and analyzed with little or no difficulty. Despite heavy interferences, the ^{13}C-labeled internal standards were recovered well. Analysis of this sample resulted in significant GC/MS system degradation.

Summary: Tri/Tetrachlorobenzenes

This matrix gave a relatively clean extract. Some interference was noted in the lower congener classes (Cl_2 - Cl_5-dibenzofurans), but this was not considered significant. Recovery of ^{13}C-labeled internal standards was good.

Summary: Aroclor 1260

The sample clean-up method failed to remove significant amounts of PCB contamination resulting in many interferences. These interferences would make determination of low level PCDD/PCDFs virtually impossible. Interestingly, large quantities of Cl_5-, Cl_6-, Cl_7- and Cl_8-dibenzofurans were detected. These levels were much higher than the original spikes and some compounds not spiked were detected. This indicates the presence of PCDFs in the original Aroclor sample. Recovery of the ^{13}C-labeled surrogates was acceptable.

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Table 6-1

GC/MS CROSSCHECK INTRUMENTAL CONDITIONS

Mass Spectrometry

Instrument:	Hewlett Packard 5987
Electron Energy:	70 eV EI
Tuning Calibrant:	PFTBA (perfluorotributylamine)
Measured Resolution (M/ΔM, 10% valley):	> 500 @ 500 m/z
Source Temperature:	250°C
Transfer Line Temperature:	275°C

Gas Chromatography

Column:	
Stationary Phase:	DB-5, fused silica
Length:	60 meter
Diameter:	0.32 mm I.D.
Film Thickness:	1.0 um
Conditions:	
Injection Type:	On-column
Injection Volume:	1.5 uL, hexane
Oven Conditions:	35°C for 1 minute, 30°C/min for 5.5 minutes, 4°C/min 5.5 to 28 minutes, 20°C/min to 310°C, hold 22 minutes
Carrier Gas:	Hydrogen
Flow Rate:	45 cm/second

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Table 6-2

RESULTS OF GC/MS CROSSCHECK*

Matrix: / Sample Number:	Aroclor 1016 (E729-09-01)	Aroclor 1242 (E729-10-01)	Aroclor 1260 (E729-11-01)	Tri/Tetrachlorobenzene (E729-12-01)	Mineral Oil (E729-08-01)
Furans					
Total Tri-CDF					
Total Tetra-CDF					125
Total Penta-CDF			477		226
Total Hexa-CDF	97	60	672	161	178
Total Hepta-CDF	80	46	807	170	36
Octa-CDF		24	1786		126
Dioxins					
Total Tetra-CDD					
Octa-CDD				386	
Congeners					
2,3,7,8-TCDD					
OCDD				386	
2,3,8-TrCDF					
2,3,7,8-TCDF					125
2,3,4,8-TCDF					
1,2,3,7,8-PnCDF			477		48
2,3,4,7,8-PnCDF					66
1,2,3,4,8-PnCDF					110
1,2,3,4,7,8-HxCDF	97	60	672	161	65
1,2,3,4,7,9-HxCDF					112
1,2,3,4,6,7,8-HpCDF	80	46	807	170	36
OCDF		24	1786		126

*All data in units of ppb (ng/g).

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Summary: Aroclor 1242

The sample clean-up method failed to remove significant quantities of PCB contamination from this sample, although it was not as heavily contaminated as the Aroclor 1260 sample. Reliable recovery and quantitation of the Cl_6- and Cl_7-dibenzofuran spikes was possible. Recovery of the ^{13}C-labeled internal standards was good. Except for a small amount of OCDF, additional PCDFs were not detected in this sample.

Summary: Aroclor 1016

Again, significant amounts of PCB contamination were not removed from this sample. However, interference with spiked analytes was not significant. The Cl_6- and Cl_7-dibenzofuran spikes were measured easily and ^{13}C-labeled internal standard recovery was good.

ASSESSMENT OF METHODOLOGY

The clean-up method gave good recovery of ^{13}C-labeled internal standards and ^{12}C spikes. However, the amount of PCB interference varied greatly among the samples. It must be noted that these samples were produced in the laboratory and not necessarily representative of actual dielectric fluids which are likely to be more complex mixtures due to the harsh environment of day-to-day operations. Since problems were encountered in the analysis of several of these synthetic samples, it is doubtful that this clean-up method can be applied successfully to a wide variety of samples. Major modifications of this method are necessary for general use.

SAMPLE EXTRACTION: ROUND ROBIN ANALYSIS

The extraction technique used by Radian for the Round Robin analysis phase of this study was a modified Smith-Stallings/EPA Region VII clean-up. This method has been shown to be effective for a wide variety of samples and was a significant improvement over the method used in the GC/MS crosscheck phase of this study. Figure 6-1 describes the clean-up procedure used for these EPRI dielectric samples.

An aliquot of the sample (approximately 0.25 g) was transferred to a column consisting of 2.5 cm sodium sulfate (on bottom), 5 g silica gel type 60, (70-230 mesh), 6 g basic silica gel (35% potassium hydroxide), and 10 g acid silica gel (40% sulfuric acid). This column was eluted with 150 mL 1:1 methylene chlo-

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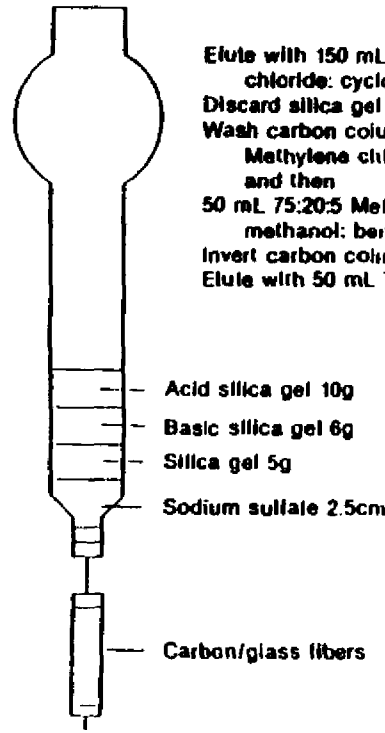
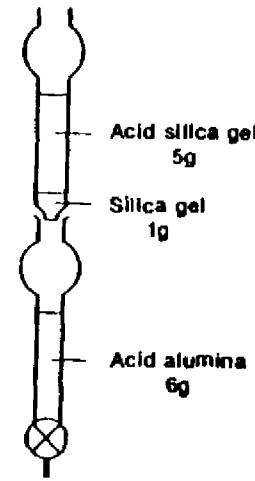
Part 1
Modified Smith-Stallings Clean-Up



Elute with 150 mL 1:1 Methylene chloride: cyclohexane
Discard silica gel
Wash carbon column with 25 mL 1:1 Methylene chloride: cyclohexane and then
50 mL 75:20:5 Methylene chloride: methanol: benzene
Invert carbon column
Elute with 50 mL toluene (collect)

Part 2
"Region 7" Clean-Up



Solvent exchange the toluene to hexane
Transfer hexane extract to silica gel column
Elute with 60 mL hexane onto alumina column
Discard silica gel
Wash alumina with 20 mL hexane, discard hexane
Elute alumina with 20 mL 20% methylene chloride: hexane (collect)
Concentrate

Figure 6-1. Sample Clean-Up Procedure: Round Robin Analysis

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ride:cyclohexane directly onto a 5 cm column of 1-9 um carbon (PX-2) on glass fibers. The silica gel was discarded and the carbon column washed with 25 mL 1:1 methylene chloride:cyclohexane and 50 mL 75:20:5 methylene chloride: methanol: benzene. The eluted solvents were discarded. The PCDDs and PCDFs were eluted from the inverted carbon column with 50 mL toluene. The toluene was evaporated to dryness on a rotary evaporator. The dried extract was redissolved in hexane and transferred to a column of 5 g acid-impregnated silica gel and 1 g silica gel (lower layer). This column was eluted with 60 mL hexane directly onto a 6 g acid alumina (AG 4, 100-200 mesh) column. The silica gel was discarded and the acid alumina was washed with another 20 mL hexane. The eluted hexane was discarded. The PCDDs and PCDFs were eluted off the alumina with 20 mL 20% methylene chloride/hexane. The volume of this eluate was reduced to 1-2 mL with a stream of nitrogen. The extract was transferred to a 1 mL conical vial and taken to dryness.

GC/MS ANALYSIS: ROUND ROBIN ANALYSIS

GC/MS analysis of the sample extracts was performed on a Hewlett-Packard 5985/87 in the selected ion monitoring mode for quantitation and identification of PCDD/PCDFs. Each sample was extracted in triplicate and each extract was analyzed in triplicate yielding nine determinations on each sample. Appendix B shows Radian log-in codes, sample numbers, and other sample control information. The first series of extracts were run on a Hewlett-Packard 50 m, Ultra-2 column (a methylphenyl silicone coating equivalent to to DB-5). On the Ultra-2 column, an initial temperature of 80°C was used. The second and third determinations were performed using a J&W 60 m, 0.32 mm I.D., 1 um film, DB-5 column. The GC/MS conditions are shown on Table 6-3. Dried sample extracts were dissolved in 50 uL of hexane and a 1.0 uL volume was injected onto a cool SGE on-column injector.

Each of the three extracts was analyzed a fourth time using a Supelco SP-2331, 60 m, 0.32 mm I.D., 0.2 um film capillary column to saparate 2,3,7,8-TCDF and 2,3,4,8-TCDF. For this separation, samples were evaporated to dryness and then reconstituted with 40 uL of nonane. A two-stage temperature program of 170°-200° at 20°/min followed by 200°-275° at 4°/min was used. Nonane solutions were necessary with the SP-2331 column to prevent "thermal shock", i.e., puddling of the column's non-bonded phase from a wide temperature program range.

Table 6-3

HRGC-MS ANALYTICAL CONDITIONS

Mass Spectrometer:	Hewlett-Packard 5985 with 5987 Upgrade
Data System:	HP1000 RTE/6
Source Temperature:	250°C
Internal Source Pressure:	3×10^{-5} Torr
Mode:	Electron Impact (EI)
Electron Energy:	70 eV
Electron Multiplier Voltage:	2650-2800 volts
Resolution:	Unit
Interface Temperature:	275°C
Scan Mode:	Selected Ion Monitoring
Cycle Time:	0.5 sec
Column:	60 meters x 0.32 mm I.D., J&W DB-5 fused silica capillary column, 1.0 um film
Carrier Gas:	Hydrogen at 3.45×10^{4}Pa (5 psi)
Carrier Linear Velocity:	35 cm/sec
Injector:	Cool On-column (65°C)
Interface to MS:	SGE (Scientific Glass Engineering) Source Coupling at 275°C
GC Program:	Initial hold of 0.5 min at 65°C then 65°-200° at 30°/min, then 200°-310° at 4°/min
Injection Volume	1 uL
Tailing Factor:	0.7-2.0

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Chromatographic Test Mixture

Part of this project involved separating the more toxic PCDD/PCDF isomers from coeluters. The chromatographic test mixture prepared at Battelle Memorial Institute contained the following analytes at 200 ng/L each:

1,3,6,8-TCDD	1,2,3,4,8-PnCDF
1,2,8,9-TCDD	1,2,4,6,8-PnCDF
1,2,3,4-TCDD	2,3,4,6,7-PnCDF
2,3,7,8-TCDD	1,2,3,7,8-PnCDF
1,3,7,8-TCDD	2,3,4,7,8-PnCDF
3,4,6,7-TCDF	1,2,4,6,7,8-HxCDF
2,3,4,8-TCDF	2,3,4,6,7,8-HxCDF
1,2,3,8-TCDF	1,2,3,4,7,8-HxCDF
2,3,7,8-TCDF	1,2,3,6,7,8-HxCDF

This test mixture was run daily to evaluate the chromatography. Chromatograms of all the analytes in the test mixture are shown on Figures 6-2 through 6-5 for DB-5 and Figures 6-6 through 6-9 for SP-2331. One analyte, 1,2,4,6,8-PnCDF, could not be found in the chromatographic test mixture. On DB-5, 2,3,7,8-TCDF and 2,3,4,8-TCDF were not resolved, but on SP-2331 this pair could be separated with a 65% valley. On SP-2331, 1,2,3,4,8-PnCDF and 1,2,3,7,8-PnCDF could not be resolved, but on DB-5 this pair could be separated with a 55% valley.

Actual samples were more difficult to resolve than the chromatographic test mixture. The analytes 1,2,3,4,8-PnCDF and 1,2,3,7,8-PnCDF could not be resolved in many cases, particularly when the 1,2,3,4,8-PnCDF concentration was much higher than the 1,2,3,7,8-PnCDF concentration. In one sample (4115911-14), the 2,3,7,8-TCDF and 2,3,4,8-TCDF pair could not be resolved accurately due to a coeluting interference. Several of the in-service liquid extracts were very complex and required the removal of 30 to 50 cm of the front of the column after every injection to give adequate resolution for subsequent samples. Column degradation for DB-5 and SP-2331 could be observed from the daily test mixture chromatograms over the course of this work.

Calibration

Four $^{12}C/^{13}C$ area ratio calibration curves were generated during this study, the first on Ultra-2, the second and third on DB-5 and the fourth on SP-2331. Concentrations of 40, 150, 500, 2500, and 7500 ng/mL were calibrated for the

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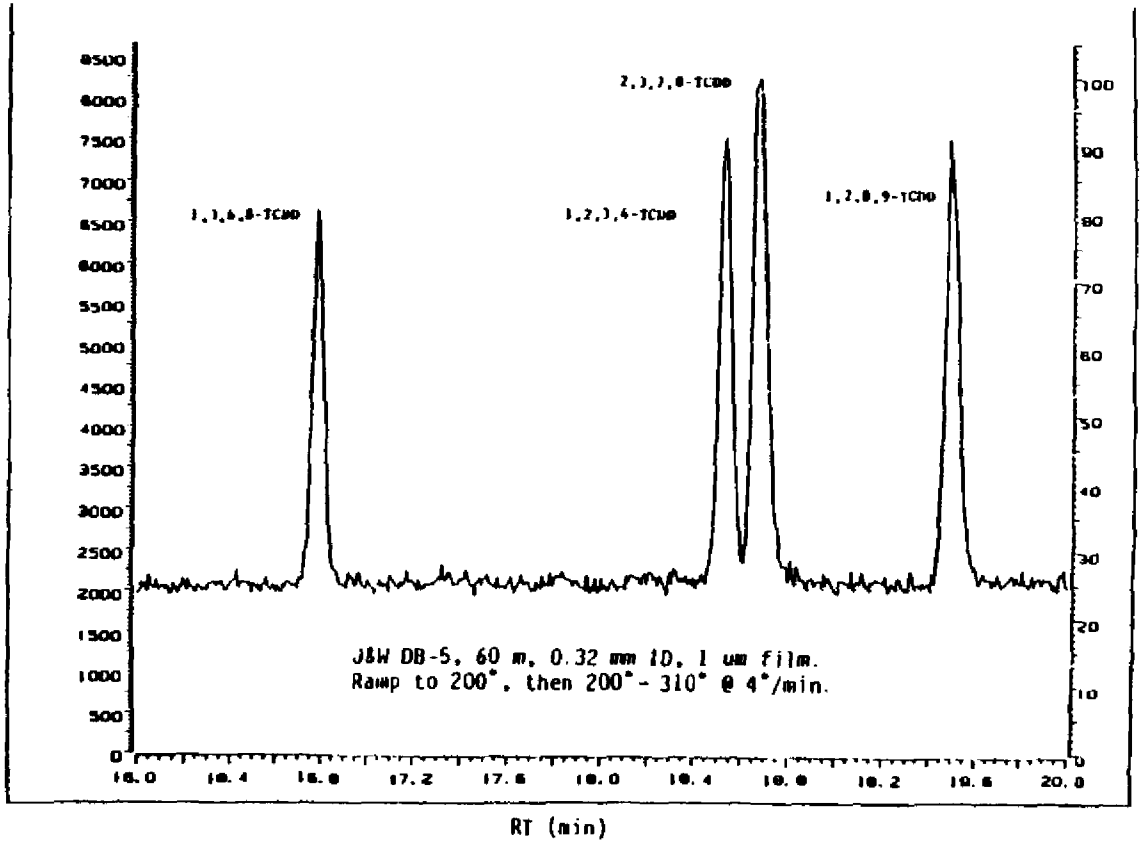

Figure 6-2. TCDD Test Mixture Chromatogram on DB-5

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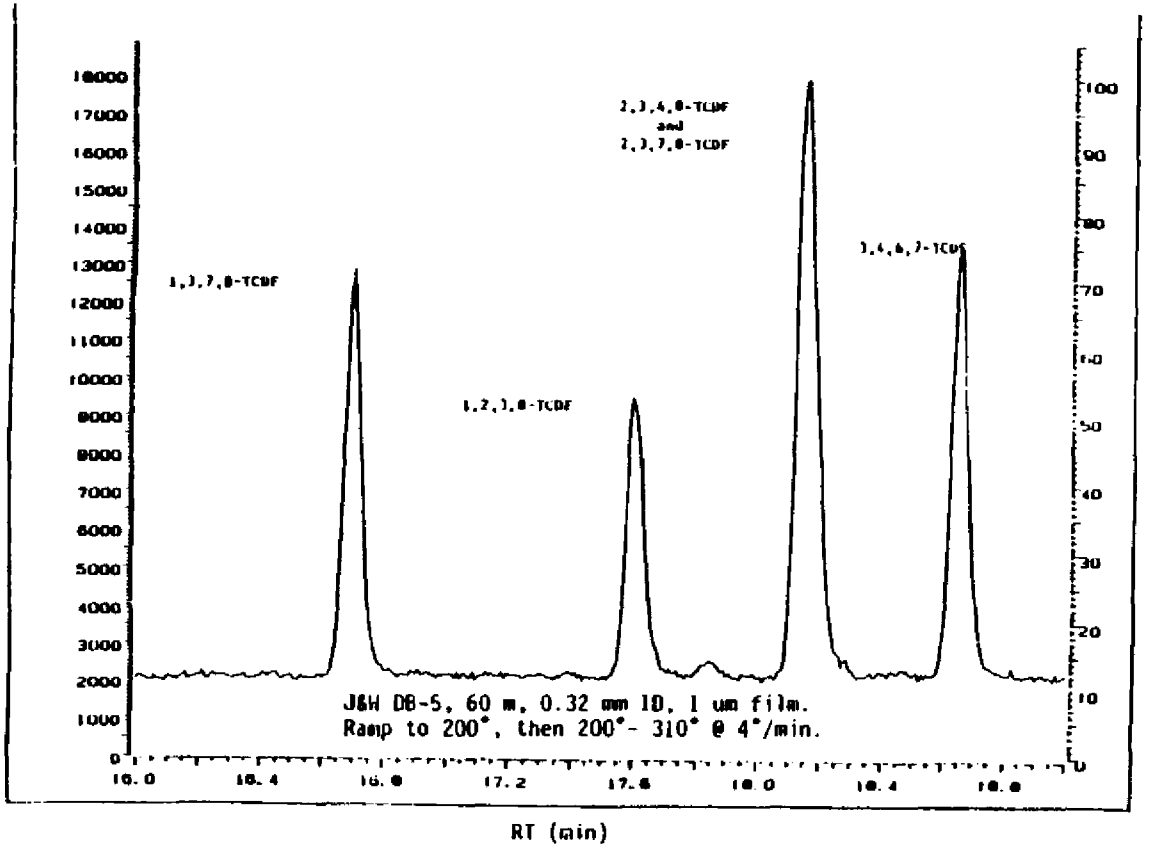

Figure 6-3. TCDF Test Mixture Chromatogram on DB-5

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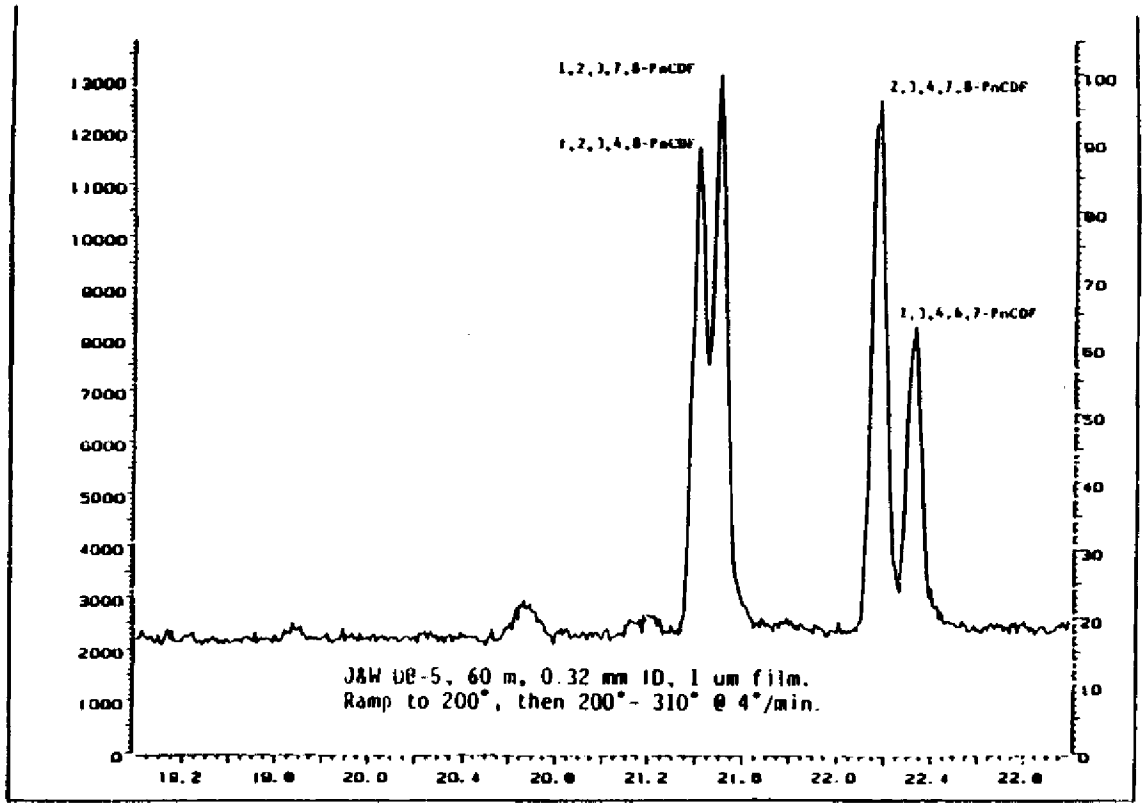

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Figure 6-4. PnCDF Test Mixture Chromatogram on DB-5

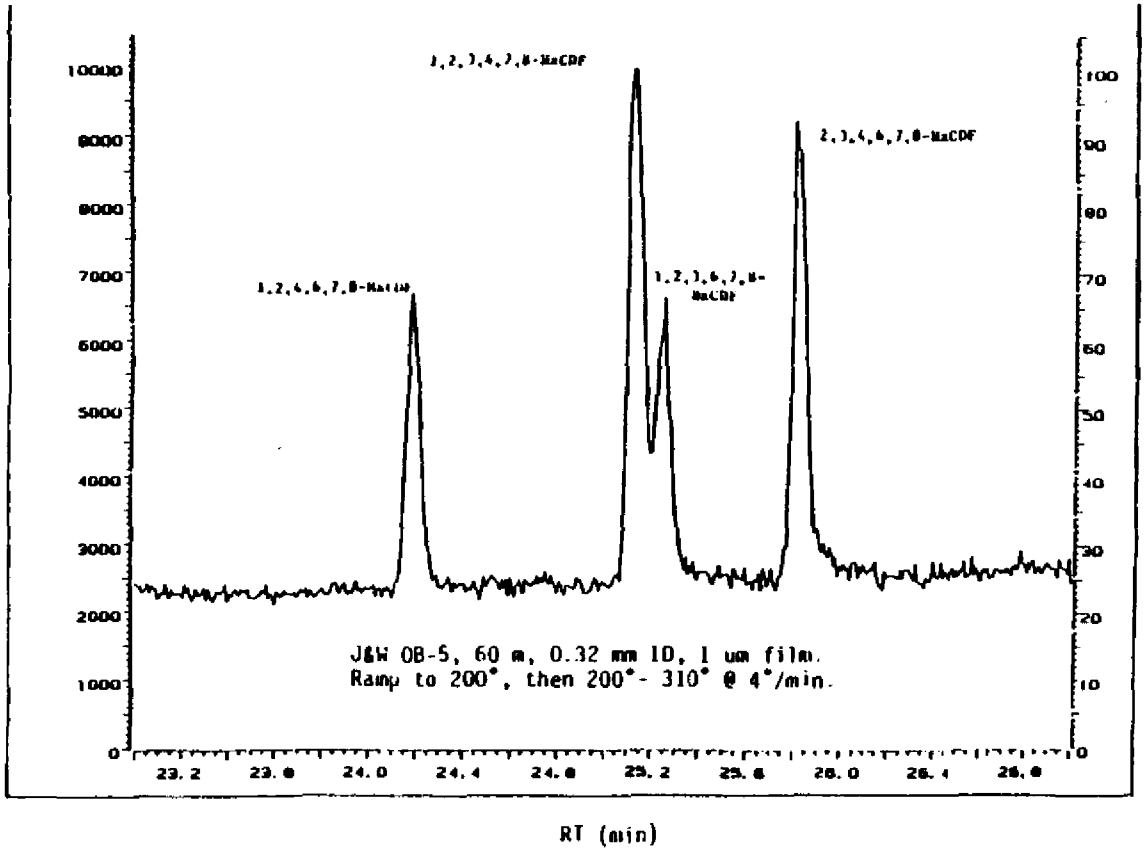

Figure 6-5. HxCDF Test Mixture Chromatogram on DB-5

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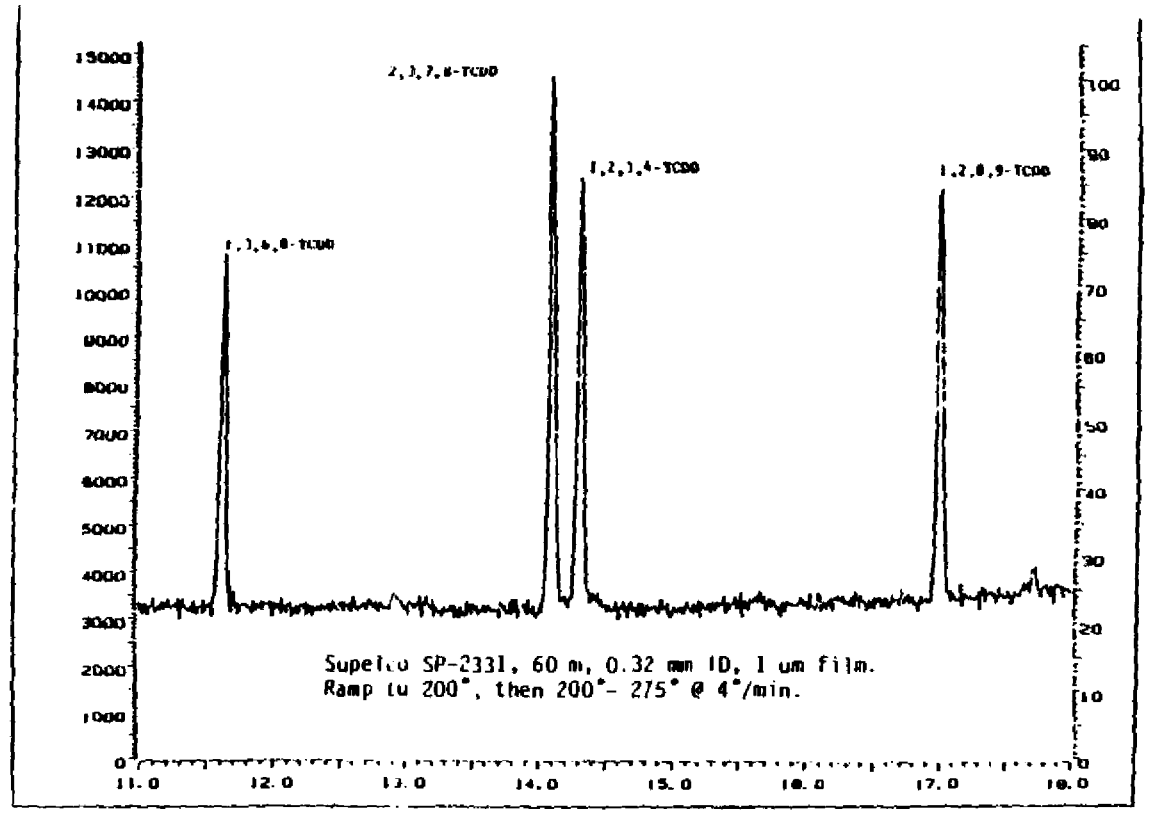

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Figure 6-6. TCDD Test Mixture Chromatogram on SP-2331

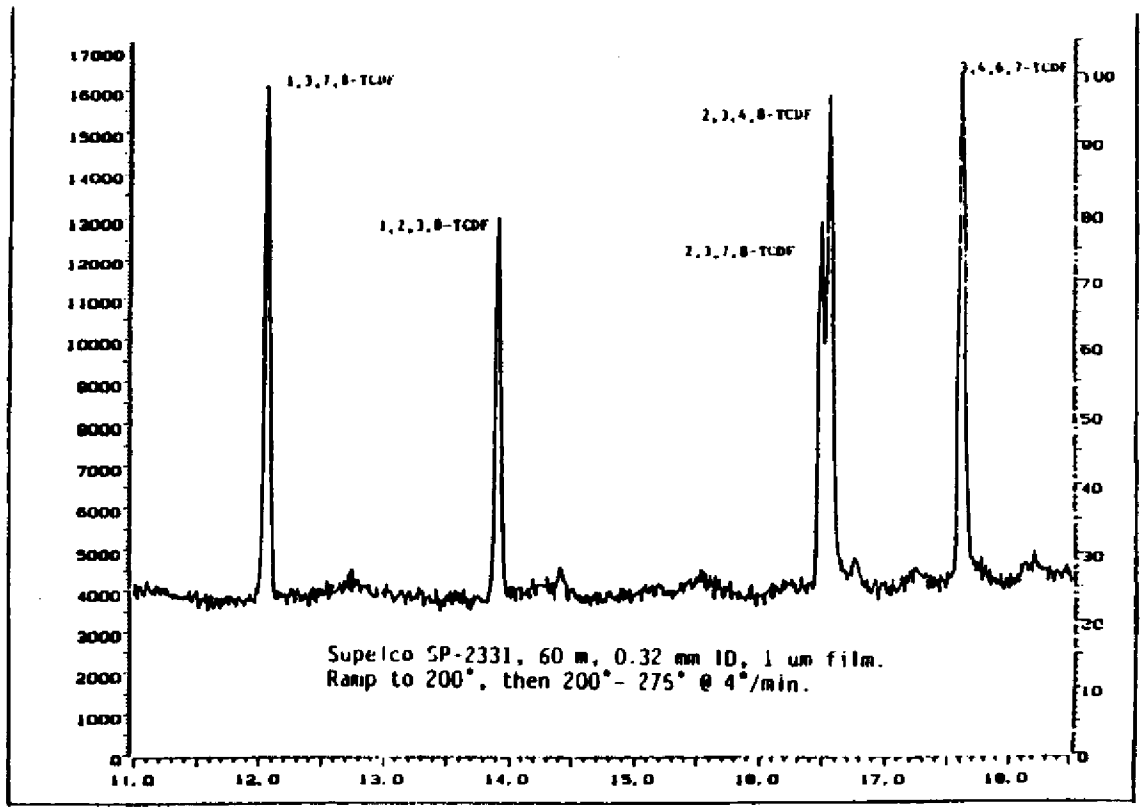

Figure 6-7. TCDF Test Mixture Chromatogram on SP-2331

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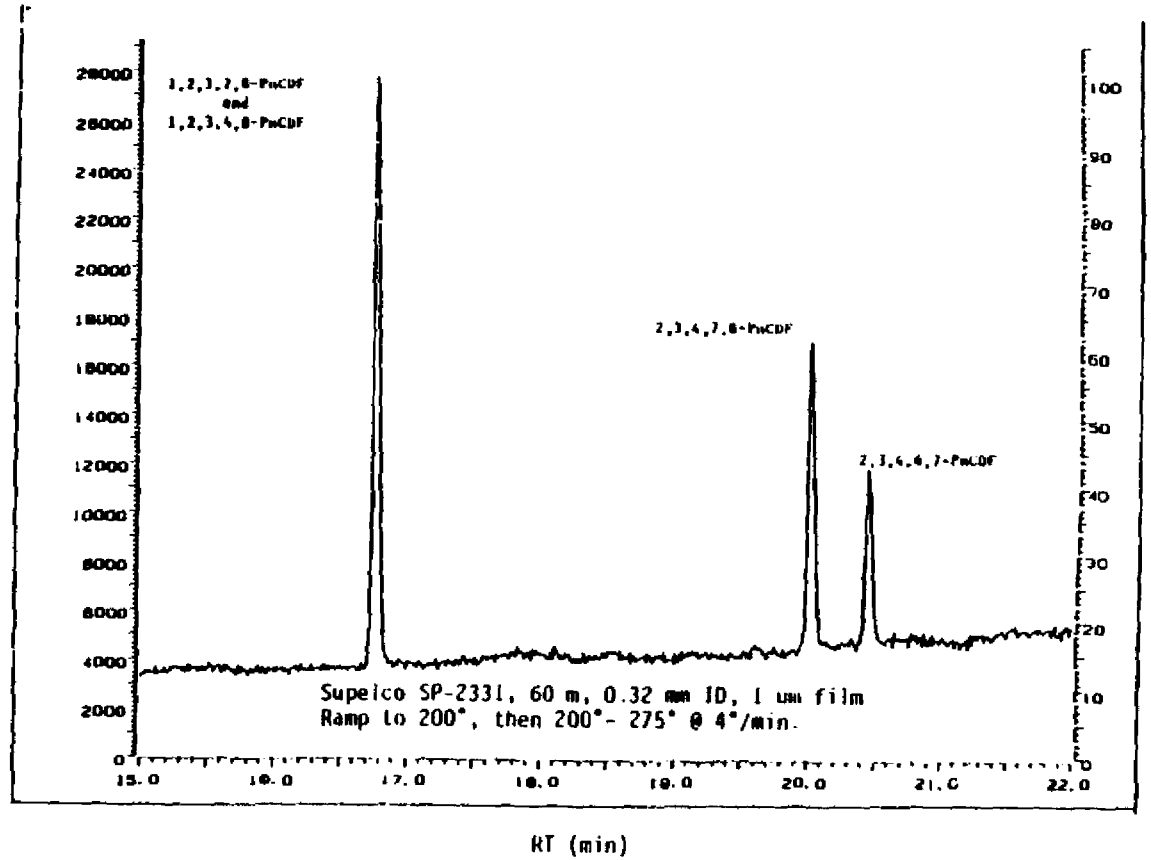

Figure 6-8. PnCDF Test Mixture Chromatogram on SP-2331

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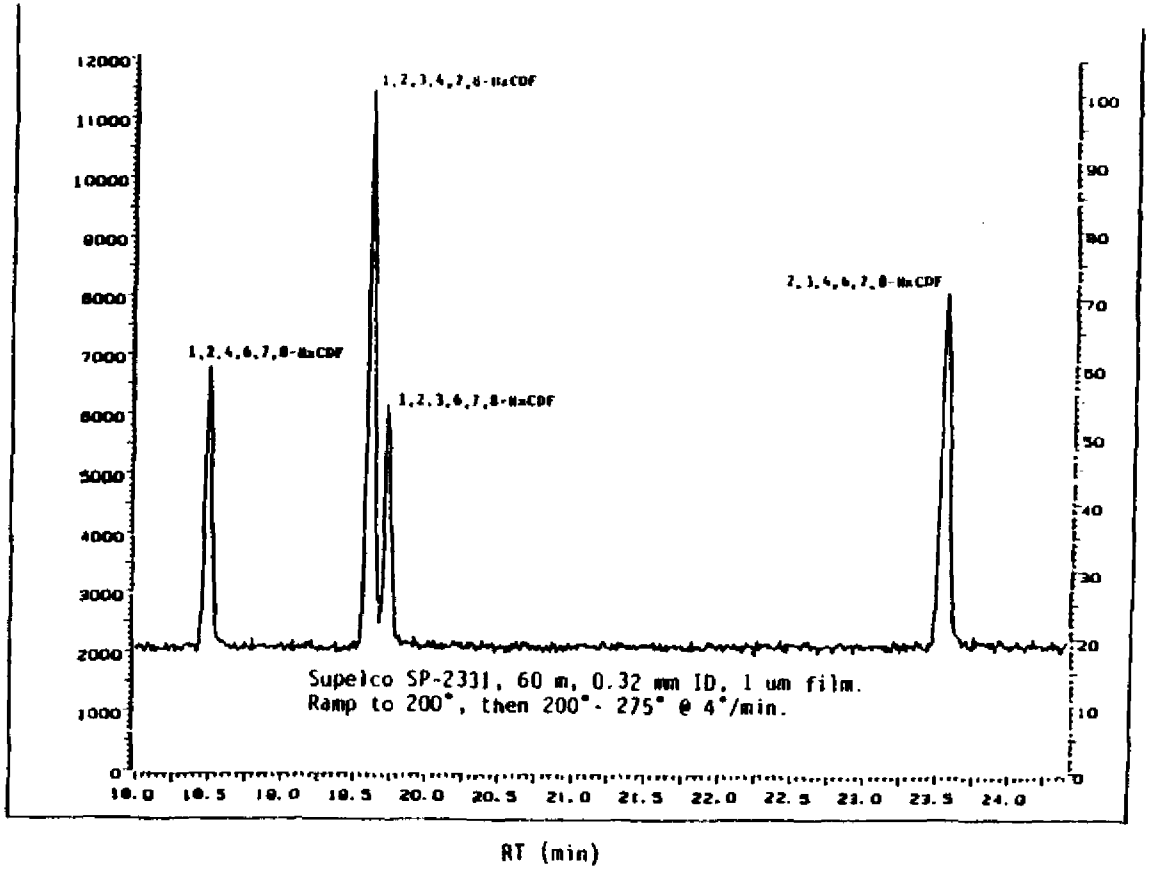

Figure 6-9. HxCDF Test Mixture Chromatogram on SP-2331

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first three calibration curves with each calibration point being the average of three determinations. The fourth calibration curve on SP-2331 also was determined in triplicate for concentrations of 40, 150, 500, and 2500 ng/mL. Calibration curves were generated and statistical analysis performed for the following analyte/^{13}C internal standard sets:

2,3,7,8-TCDD	2,3,7,8-TCDD-^{13}C
2,3,7,8-TCDF	2,3,7,8-TCDF-^{13}C
1,2,3,7,8-PnCDF 2,3,4,7,8-PnCDF	1,2,3,7,8-PnCDF-^{13}C
1,2,3,4,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF	1,2,3,4,7,8-HxCDF-^{13}C
OCDF	OCDD-^{13}C

Calibration curves and regression analyses used in this study are given in Appendices C through F. Overall, the calibration curves were very linear throughout the range tested and exhibited correlation coefficients greater than 0.999 in most cases.

Identification of PCDD/PCDFs

Identification of native PCDFs and PCDDs was performed by measuring chlorine isotope ratios and monitoring the presence of the fragment ions arising from loss of COCl. For PCDFs, two chlorine isotope peaks were monitored in addition to the quantitating ion. Only the most abundant chlorine isotope peak was monitored for the first series of extracts. The acceptance criteria for PCDD/PCDF positive identification required agreement better than ±10% between the theoretical and experimentally determined chlorine isotope ratios. The presence of either a second chlorine isotope peak or a peak arising from the loss of COCl (M-COCl) also was necessary for positive identification.

In the few cases where there was substantial interference in the ion profile of the largest chlorine isotope peak, the second largest chlorine isotope peak was used for evaluation of the isotope ratio acceptance criteria. In a few cases, a ±15% chlorine isotope ratio was accepted. If a peak was not within ±10% of the criteria on one determination but was within ±10% on previous determinations, the determination was accepted.

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Quantitation

Quantitation was performed by isotope dilution using the ^{13}C-labeled internal standards added before extraction. Concentrations were determined from $^{12}C/^{13}C$ ratios of sample analyte to internal standard and a linear fit to the five-point calibration curves (see Appendix C). A separate calibration curve was generated for 2,3,4,7,8-PnCDF. All other PnCDFs were quantitated from the 1,2,3,7,8-PnCDF calibration curve. A separate calibration curve was produced for 2,3,4,6,7,8-HxCDF. All other HxCDFs and HpCDFs were quantitated from the 1,2,3,4,7,8-HxCDF calibration curve. OCDD and OCDF were quantitated from the OCDD-^{13}C calibration curve. Congener class total concentrations were calculated by summing concentrations of each peak which passed the identification criteria for the congener class. Surrogate recoveries were determined using the mean 500 ng/mL response factor from the calibration checks made at the start and end of the analyses.

One sample (41159-11-13), had a severe interference which coeluted with the TCDF-^{13}C peaks. TCDFs in this sample were quantitated versus the area of the PnCDF-^{13}C quantitating ion with a normalization factor of 1.1 (the average ratio of TCDF-^{13}C to PnCDF-^{13}C response).

Detection Limits

Instrumental detection limits were defined as the analyte concentration which gave a 3:1 signal-to-noise ratio. Method detection limits for all analytes were approximately 3 ng/g except OCDF and OCDD which were 10 ng/g due to peak broadening at the later retention times. The lowest point on the calibration curves, 40 ng/mL, corresponds to a sample concentration of 8 ng/g.

Instrumental QA/QC

At the beginning of each working day, the mass spectrometer was tuned for DFTPP (decafluorotriphenylphosphine) in the range of 197-443 amu (atomic mass units) to pass the criteria set forth in EPA SW-846, "Test Methods for Evaluating Solid Waste", (Method 8280). The chromatographic test mixture also was injected at the beginning of each working day to evaluate chromatographic resolution. Baseline resolution of the 1,2,3,4-TCDD/2,3,7,8-TCDD pair was necessary for the chromatography to be accepted.

Standard mixtures (500 ng/mL) were injected at the beginning and end of each working day and the response factors from these runs were compared to the average

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response factors from the 5-point calibration curves. These daily calibration date are presented in Table 6-4. These daily calibrations from the 2,3,7,8-TCDF/2,3,4,8-TCDF seperations on SP-2331 are shown in Table 6-5. Generally, the daily calculated response factors agreed within ±10%. For the first extraction set, response factor agreement was generally ±20%. The gas chromatographic conditions were changed after the analysis of the first extraction set by reducing the initial GC temperature of 80°C to 65°C. The new gas chromatography conditions resulted in improved precision.

BLANK SAMPLES

Nine extraction blanks were obtained in this study. One was generated from the first extraction set, four from the second extraction set, and four from the last extraction set. The results from the analysis of these blanks are shown in Appendix G. Acceptably low concentrations of some furans were found in several of the blanks.

Three blanks of mineral oil, Aroclor 1016, and Aroclor 1260 were analyzed to measure background levels of PCDD/PCDFs in the blind-spiked baseline liquids. These results are shown in Appendix H. No PCDD/PCDFs were found in the mineral oil or Aroclor 1016, but the Aroclor 1260 blank showed high levels of PCDFs.

BASELINE LIQUID RESULTS

To determine the accuracy of PCDD/PCDF determinations in mineral oil, Aroclor 1016, and Aroclor 1260, the baseline samples were reanalyzed. Results for the triplicate determinations on all of the baseline liquids, triplicate extraction results, extraction averages and coeluter separations on SP-2331 are shown in Appendix I. A summary of the Baseline Liquid results compared to the blind spike values is shown in Table 6-6. For the Mineral Oil Sample (E729-53-01), the average experimentally obtained values show good agreement with the spiked values, ranging from 0.6% to 8.5%. For 2,3,7,8-TCDF and 2,3,4,8-TCDF separated on SP-2331, there is a difference of 13% and 27% between the experimental results and the spiked values, while the combined value determined on DB-5 shows a difference of only 8.5%. Results for the spiked Aroclor 1016 sample (E729-53-02), again show good agreement with the spike values, ranging from 1.9% to 15.1%. No 2,3,4,8-TCDF was detected in this sample. For the Aroclor 1260 (E729-53-03), the results showed very little agreement with the spiked values. This result is attributable to high levels of some congeners in the original sample. Interestingly, several congeners including the highly toxic 2,3,7,8-TCDD and 2,3,7,8-TCDF

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Table 6-4

DAILY CALIBRATION DIFFERENCES (ABSOLUTE) FROM FIVE POINT CALIBRATION RESPONSE FACTORS

Analyte:	2378	2378	12378	23478	123478	234678	1234678	
Data	TCDD	TCDF	PnCDF	PnCDF	HxCDF	HxCDF	HxCDF	OCDF
Extraction 1								
2/21/86	27.2	4.8	19.5	24.0	19.2	12.0	14.4	23.8
2/21/86	1.9	14.1	13.1	9.1	13.3	13.8	17.1	NA
2/24/86	6.6	0.0	5.5	5.4	3.6	4.7	26.4	6.8
2/24/86	0.9	3.4	7.1	16.9	68.7	0.3	5.6	14.6
2/25/86	0.5	16.8	10.3	11.4	24.5	33.7	43.6	10.7
2/25/86	9.3	14.3	6.7	4.5	3.5	27.4	39.8	5.0
Extraction 2								
3/18/86	2.7	1.8	0.9	2.5	0.1	2.9	5.7	2.0
3/18/86	4.9	1.4	0.3	2.1	4.5	3.5	3.3	12.1
3/20/86	2.0	1.1	0.9	4.2	4.3	2.2	9.2	7.6
3/20/86	1.1	3.7	1.8	4.2	0.8	0.5	11.4	11.1
3/21/86	2.3	6.1	7.1	8.5	3.0	1.1	12.1	0.5
3/21/86	7.9	1.4	7.1	5.1	1.0	1.1	19.3	30.8
3/22/86	4.4	3.9	0.0	0.9	12.0	3.2	17.9	2.5
3/22/86	1.1	7.9	8.0	9.3	1.0	1.7	5.1	0.5
Extraction 3								
3/30/86*	1.7	1.7	0.7	0.7	0.5	3.8	8.6	1.3
3/31/86	2.4	0.2	1.3	0.7	1.6	0.5	4.2	10.1
3/31/86	0.7	0.4	6.1	0.7	40.3	3.1	23.4	148.6
4/01/86	1.2	0.8	0.1	1.1	1.1	3.9	12.2	5.0
4/01/86	7.8	1.2	6.1	3.3	12.1	2.0	2.7	7.1
4/02/86	9.2	0.9	2.2	2.1	3.0	5.0	0.5	1.9
4/02/86	12.4	0.4	4.1	3.4	5.3	5.9	5.7	4.2

Data as percent deviation from five-point curve.
NA = Not available.
* = Five-point calibration run on morning of the same day.

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Table 6-5

DAILY CALIBRATION PERCENT DIFFERENCES (ABSOLUTE)
FROM FOUR POINT RESPONSE FACTORS ON SP-2331

Date	2,3,7,8-TCDF Percent Difference
4/24/86	5.6
4/24/86	2.3
4/28/86	3.4
4/28/86	0.6
4/29/86	2.4
4/29/86	4.4
4/30/86	3.0
4/30/86	1.4
Average	2.9

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Table 6-6

SUMMARY OF BASELINE LIQUIDS RESULTS

Sample:	E729-53-01 Mineral Oil			E729-53-02 Aroclor 1016			E729-53-03 Aroclor 1260		
	Average Value[a]	Spike Value	% Difference from Spike	Average Value[a]	Spike Value	% Difference from Spike	Average Value[a]	Spike Value	% Difference from Spike
PCDD/PCDF									
Total TCDF	183			61			1509		
Total PnCDF	298			305			1343		
Total HxCDF	480			194			2363		
Total HpCDF	407			558			2252		
OCDF	14	0	--	126	132	4.6	3921	222	1666
Total TCDD	24			50			99		
OCDD	<10			<10			<10		
Congeners									
2,3,7,8-TCDD	24	25	4.0	50	53	5.7	99	111	10.8
2,3,7,8-TCDF	113[183]	100	13.0[8.5]	61	53	15.1	881[1136]	833	5.8[10.2]
2,3,4,8-TCDF	73[183]	100	27.0[8.5]	<3	0	--	150[1136]	194	22.7[10.2]
1,2,3,7,8-PnCDF	141	150	6.0	155	158	1.9	180	111	62.2
1,2,3,4,8-PnCDF	<3	--	--	<3	--	--	132	--	--
2,3,4,7,8-PnCDF	155	150	3.3	150	158	5.1	278	111	150.4
1,2,3,4,7,8-HxCDF	259	250	3.6	<3	0	--	1139	500	127.8
1,2,3,4,7,9-HxCDF	<3	--	--	<3	--	--	33	--	--
1,2,3,7,8,9-HxCDF	<3	--	--	<3	--	--	35	--	--
1,2,3,6,7,8-HxCDF	48	50	4.0	58	53	9.4	299	278	7.6
2,3,4,6,7,8-HxCDF	174	175	0.6	136	132	3.0	327	56	483.9
1,2,3,4,6,7,8-HpCDF	407	400	1.8	558	526	6.1	768	111	591.9

[a] Average of three extractions, three determinations each; expressed in ppb (ng/g).
[ ] 2,3,7,8-TCDF combined; from LAB-5 determination. Separation achieved on SP-2331.

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showed good agreement, indicating the absence of these congeners in the original Aroclor 1260.

The precision from all 27 baseline liquid determinations was 4.0%. The precision for nine extraction averages from the three baseline liquids was 4.2%.

IN-SERVICE LIQUID RESULTS

The remaining part of this project involved analysis of seven liquids from electrical equipment currently in-service. Triplicate determination results, triplicate extraction results, extraction averages and coeluter separations generated from these seven in-service samples are shown in Appendix J. A summary of the results from the in-service samples is shown in Table 6-7.

In-service sample 41159-11-11 contained Aroclor 1260 and tri/tetrachlorobenzene. It had very high concentrations of the higher chlorinated PCDFs and relatively low concentrations of TCDFs. The second extract generated from this sample had an anomalously high surrogate recovery of OCDD-^{13}C (246%). The resulting native OCDF concentration calculated from comparison to the OCDD-^{13}C internal standard was relatively low compared with the other sample extracts. The precision for nine determinations was 7.7%, and the precision for the three extractions, (each the average of three determinations) was 6.3%.

If the anomalously low OCDF concentration for the second extract is omitted, the resulting measured extraction precision is 3.2%. In this sample, the sum of the unresolved 2,3,7,8-TCDF/2,3,4,8-TCDF concentration on DB-5 is 38 ng/g. On SP-2331, the average sum of the separated pair on SP-2331 is 26 ng/g, a 38% difference. The 1,2,3,4,8-PnCDF concentration was much higher than the 1,2,3,7,8-PnCDF concentration and could not be resolved.

Sample 41159-11-12 was a transformer with 67% Aroclor 1260 plus 32% tri/tetrachlorobenzene. The precision for nine determinations on this sample was 8.8%, and the precision for the three extractions was 12.4%. The concentration of the unresolved 2,3,7,8-TCDF/2,3,4,8-TCDF pair on DB-5 was 31 ng/g. On SP-2331 the average sum of the separated pair was 26 ng/mL, an 18% difference. Only in the third extract was the 1,2,3,7,8-PnCDF/1,2,3,4,8-PnCDF pair resolved.

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Table 6-7

SUMMARY OF IN-SERVICE LIQUIDS RESULTS

Sample:	41159-11-11 Avg Value*	41159-11-12 Avg Value*	41159-11-13 Avg Value*	41159-11-14 Avg Value*	41159-11-15 Avg Value*	41159-11-16 Avg Value*	41159-11-17 Avg Value*
PCDD/PCDF							
Total TCDF	160	91	847	882	<3	<3	479
Total PCDF	2053	299	305	9384	<3	<3	1248
Total HxCDF	7055	916	37	9733	18	NA	2313
Total HpCDF	10727	2330	NA	2048	39	NA	2358
OCDF	21814	5904	23	201	87	NA	3205
Total TCDD	<3	<3	<3	<3	<3	<3	<3
OCDD	<10	<10	<10	<10	<10	<10	<10
Congeners							
2,3,7,8-TCDD	<3	<3	<3	<3	<3	<3	<3
2,3,7,8-TCDF	9	18	22	139	<3	NA	109
2,3,4,8-TCDF	17 [38]	8 [31]	68 [245]	74 [338]			35 [182]
1,2,3,7,8-PnCDF	1013**	108**	6	862	<3**	<3**	305**
1,2,3,4,8-PnCDF			12	345			
2,3,4,7,8-PnCDF	109	37	59	1646	<3	<3	196
1,2,3,4,7,8-HxCDF	4755	571	20	3913	18	NA	1345
1,2,3,4,7,9-HxCDF	119	14	<3	293	<3	<3	63
1,2,3,7,8,9-HxCDF	265	38	NA	1175	<3	<3	65
1,2,3,6,7,8-HxCDF	312	37	NA	762	<3	<3	70
2,3,4,6,7,8-HxCDF	199	32	NA	921	<3	<3	108
1,2,3,4,6,7,8-HpCDF	4061	668	NA	678	21	NA	1138

* Average of three extractions, three determinations each expressed in ppb (ng/g).
** Combined value for 1,2,3,7,8-PnCDF and 1,2,3,4,8-PnCDF.
[ ] 2,3,7,8-TCDF, 2,3,4,8-TCDF combined from DB-5 determination. Separation achieved on SP-2331.
NA:Not available; some determinations were less than detection limit and some were greater.

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Sample 41159-11-13 was a capacitor askarel. This sample, unlike the previous two in-service liquids, had higher concentrations of TCDFs and lower concentrations of the higher chlorinated congenars. TCDFs were particularly difficult to quantitate because the major ion from the TCDF-^{13}C internal standard was obscured totally by coeluting interferences at the same mass (see Figure 6-10). The TCDD-^{13}C also was obscured partially by interferences; for this reason, surrogate recoveries could not be calculated. TCDF was quantitated versus the PnCDF-^{13}C internal standard normalized using a factor of 1.1 (the average TCDF-^{13}C/ PnCDF-^{13}C response ratio in the 500 ng/mL standards). The concentration of the unresolved 2,3,7,8-TCDF/2,3,4,8-TCDF pair on DB-5 was 245 ng/g. On the SP-2331, the average sum of the resolved pair was 90 ng/g, a substantial 93% difference. This difference may be due to suppression of ionization by coeluting interferences. On SP-2331, TCDF was quantitated using TCDF-^{13}C as the internal standard, and the suppression of ionization effect should be similar for both the analyte and the standard. However, on DB-5, TCDF was quantitated versus PCDF-^{13}C. This standard elutes in a different portion of the chromatogram where suppression of ionization effects most likely were not similar. An alternate explanation is that evaporation of the extract to dryness resulted in the loss of 2,3,4,8-TCDF relative to 2,3,7,8-TCDF-^{13}C. The average precision for the nine determinations was 12.8%, and the precision for the three extractions was 16.4%.

Sample 41159-11-14 was from an ESP transformer. The average precision for nine determinations was 10.7% and the precision for the three extractions was 6.5%. A large coeluting peak interfered with quantitation of 2,3,7,8-TCDF and 2,3,4,8-TCDF on the SP-2331 column (see Figure 6-11). This accounts for the relatively large coefficient of variance (45%) obtained for the 2,3,4,8-TCDF (see Appendix J).

Sample 41159-11-15 was an arc furnace contaminated mineral oil. The first determination on the first extraction of this sample apparently had carry-over effects from previous injections and, for this reason, higher detection limits were used. Determination and extraction precisions were relatively high in this sample due to concentrations near detection limits. TCDFs were not found in this sample.

Sample 41159-11-16 was a transformer askarel. A few low-level analytes were quantitated in some of the determinations, but when the three extractions were averaged, no significant concentrations of analytes were found.

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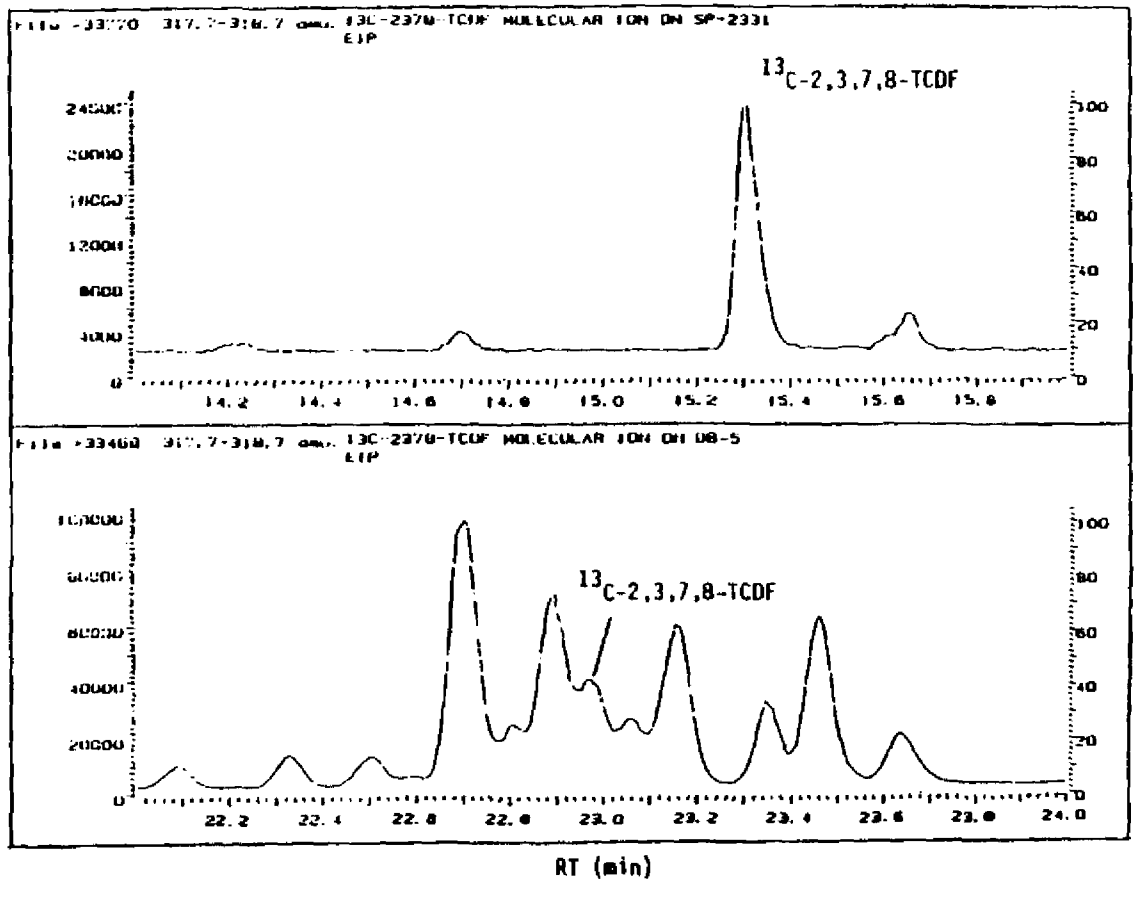

Figure 6-10. Capacitor Oil Interference

MONS 218179

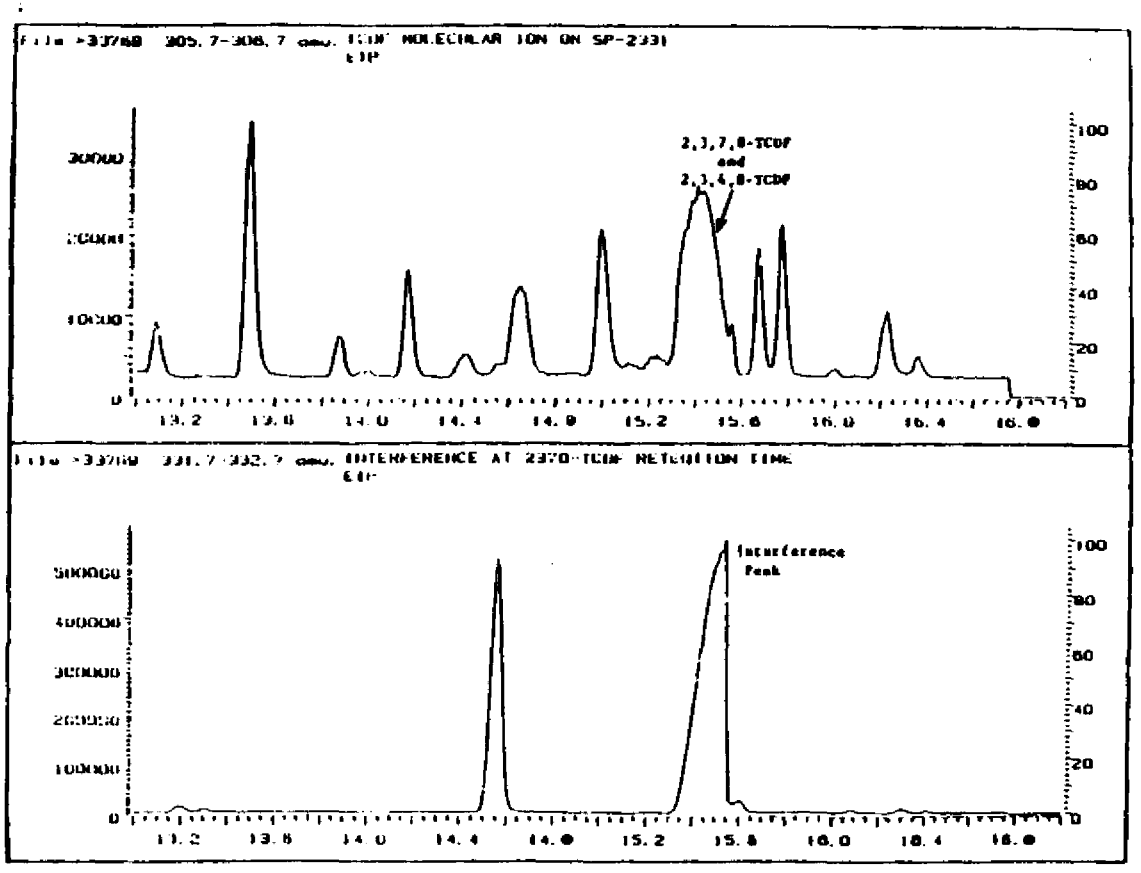

Figure 6-11. ESP Transformer Oil Interference

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in-service liquid 41159-11-17, was an askarel sample. The second extract had a high OCDD-^{13}C surrogate recovery of 1214% and consequently, the OCDF concentration calculated versus the OCDD-^{13}C internal standard was anomalously low. The precision of the three extraction averages was 11.9%. Omitting the anomalously high OCDF value gives an extraction precision of 6.9%. The precision for the nine determinations is 7.6%. The 1,2,3,7,8-PnCDF and 1,2,3,4,8-PnCDF were resolved only in the third extract.

Surrogate Recoveries

Sample surrogate recoveries versus 2,3,7,8-TCDD-^{13}C are shown on Table 6-8. Recoveries were generally between 80-200%. The second extraction for sample 41159-11-14 had low surrogate recoveries of approximately 50%. The second extraction for sample 41159-11-17 had an extremely high OCDF-^{13}C recovery of 1214%, the average of three seperate determinations of close agreement. Preparation of the second extraction sample must have been done incorrectly. Three other extractions had OCDD-^{13}C recoveries between 200-300%.

Surrogate recoveries for the nine extraction blanks were generally between 80-200% and are shown on Table 6-9. Some OCDD-^{13}C surrogate recoveries were as high as 476%. Surrogate recoveries are not as consistent for the blanks as for the actual samples. This is most likely due to TCDD-^{13}C evaporation during the concentration.

Interferences

The sample clean-up procedure did not remove all of the interferences present in the in-service liquids. On DB-5, the capacitor oil sample (41159-11-13) had interferences coeluting throughout the TCDF and PCDF retention region interfering with all major ions of 2,3,7,8-TCDF-^{13}C. On SP-2331, the ESP transformer sample (41159-11-14) had an interference coeluting with 2,3,7,8-TCDF/2,3,4,8-TCDF preventing unambiguous quantitation. Overall, the greater relative extraction precision of the baseline liquids compared to the inservice liquids reflects the greater difficulty of clean-up of the in-service liquid matrix. The average precision for the three baseline liquid extractions on DB-5 was 4.2%. The average precision for the five most complex in-service liquid extractions (omitting two anomalous OCDF values) was 10.7%.

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Table 6-8

SAMPLE SURROGATE RECOVERIES VERSUS ^{13}C-2,3,7,8-TCDD

Sample	Recoveries (%)			
	^{13}C-TCDF	^{13}C-PnCDF	^{13}C-HxCDF	^{13}C-OCDD
41159-11-11A (Extraction 1)	104	120	140	NC
11B (Extraction 2)	105	121	127	246
11C (Extraction 3)	94	107	127	94
41159-11-12A	100	120	115	101
12B	103	121	137	116
12C	80	85	96	76
41159-11-14A	97	108	108	86
14B	50	55	62	43
14C	70	74	84	65
41159-11-15A	84	144	180	180
15B	88	129	149	149
15C	91	137	168	165
41159-11-16A	104	129	133	122
16B	93	142	175	188
16C	94	137	152	153
41159-11-17A	106	113	131	101
17B	99	123	133	1214
17C	97	114	112	88
729-53-01A	93	115	152	180
01B	97	162	226	275
01C	94	136	164	153
729-53-02A	98	122	159	173
02B	101	142	181	212
02C	94	125	150	147
729-53-03A	98	110	144	144
03B	98	115	128	125
03C	90	102	113	100
41159-11-13A*	NC	NC	NC	NC
13B*	NC	NC	NC	NC
13C*	NC	NC	NC	NC

*^{13}C-TCDD could not be determined in this sample due to severe interference.
NC = Not calculated.

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Table 6-9

SURROGATE RECOVERIES VERSUS ^{13}C-2,3,7,8-TCDD IN BLANK SAMPLES

Sample	Recoveries (%)			
	^{13}C-TCDF	^{13}C-PCDF	^{13}C-HxCDF	^{13}C-OCDD
A509049	93	157	195	318
A510015-C-2	82	154	216	254
A510015-D-2	100	125	122	130
A510015-E-2	93	119	131	121
A510015-F-2	78	174	306	476
A510015-C-3	94	126	155	165
A510015-D-3	132	124	136	144
A510015-E-3	94	127	157	168
A510015-F-3	94	114	131	149
E729-54-02	55	144	233	314
E729-54-03	97	115	126	120
E729-54-04	84	115	169	229

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On-Column Injector Sample Carry-Over

Sample carry-over from the on-column injector appeared to cause some problems. Some of the higher molecular weight congeners show greater than 20% variations in the daily response factor calibration checks, particularly at the end of the day. This was most likely due to carry-over from extracts containing high levels of PCDD/PCDF congeners injected over the day. As mentioned earlier, this was a particular problem with the initial set of samples which had the higher (80°C versus 65°C) initial gas chromatograph temperature.

CONCLUSIONS

This work has shown that the analytical methodology employed for quantitation of PCDD/PCDFs in dielectric fluids was appropriate and successful. The GC/MS response was shown to be linear over a wide range of analytical concentrations, the two columns used in this work, SP-2331 and DB-5, separated the specific toxic congeners, and the accuracy of GC/MS quantitation was validated in the blind-spiked baseline liquid study. The in-service sample GC/MS determinations showed that matrix interferences can be a problem with some samples. Difficulties were encountered with some of the higher chlorine number surrogate recoveries, most likely due to TCDD-^{13}C loss during the extract concentration. Since these surrogates were used as multiple internal standards (one for each isomer class except HpCDFs), quantitation of native PCDD/PCDFs should not have been adversely affected by high surrogate recoveries.

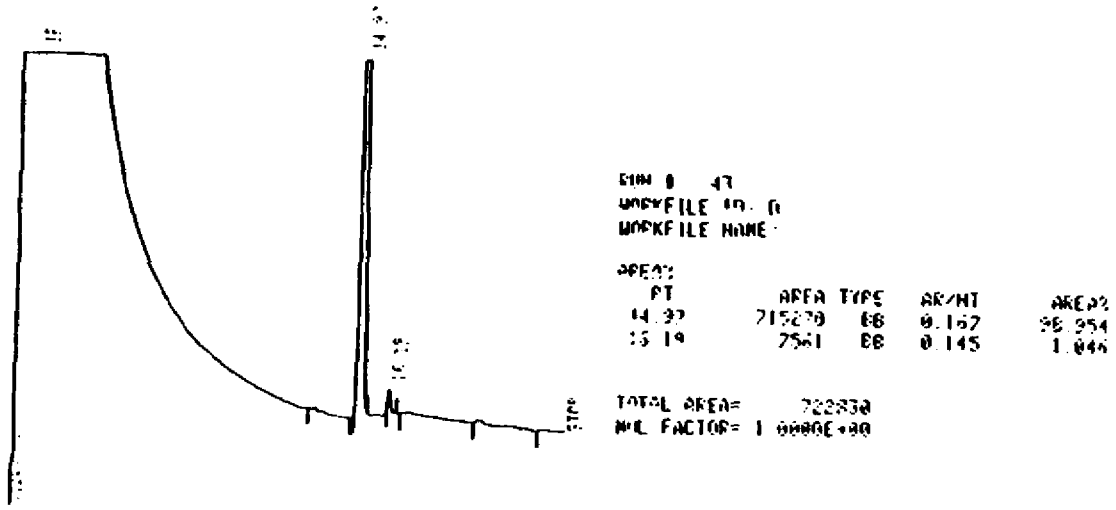
MONS 218184

Appendix A

CHROMATOGRAMS

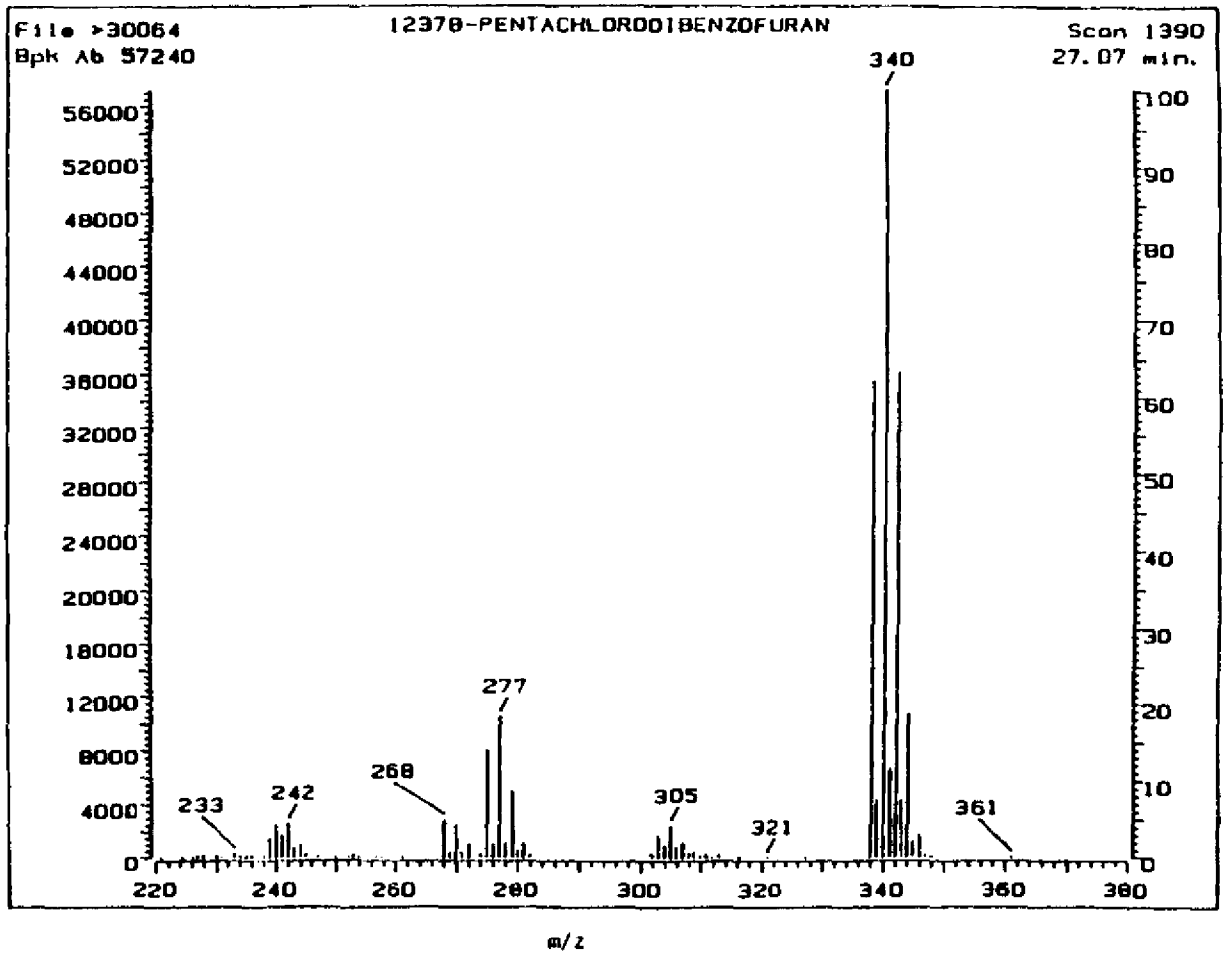
The following pages contain Gas Chromatograms and Mass Spectra of compounds synthesized for this program.

MONS 218185



MONS 218186

Gas Chromatogram of 1,2,3,7,8-Pentachlorodibenzofuran



A-3

MONS 218187

>30064 12378-PENTACHLORODIBENZOFURAN
1390 NRM

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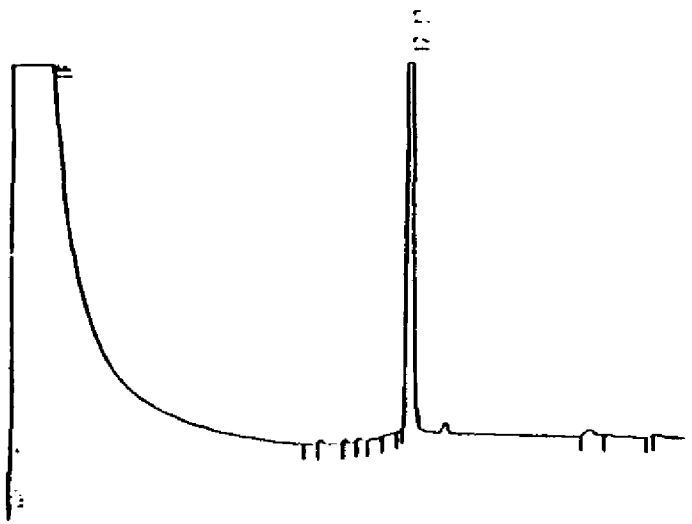
215.00	.00	243.00	1.25	276.95	18.38	316.40	.05	395.65	.04
216.00	.17	244.00	1.56	277.95	1.97	320.90	.05	400.40	.03
216.90	.73	245.00	.33	278.95	8.66	327.15	.04	400.75	.04
219.75	.07	247.00	.08	279.95	1.09	328.00	62.12	409.25	.06
220.90	.05	249.90	.05	280.95	1.89	329.00	7.59	424.65	.06
224.50	.05	252.15	.09	291.95	.26	340.00	100.00	425.00	.04
226.15	.04	253.00	.14	301.90	.39	341.00	11.64	431.40	.04
226.90	.15	253.90	.06	302.90	2.96	342.00	62.24	446.30	.06
227.90	.19	256.70	.05	303.90	1.64	343.00	7.54	448.20	.07
230.00	.21	257.70	.04	304.90	4.13	344.00	18.70	475.20	.04
230.90	.09	261.20	.06	305.90	1.43	345.00	2.20	482.50	.04
233.00	.50	267.95	4.83	306.90	2.00	346.00	3.00	494.75	.04
234.00	.15	268.95	.54	307.90	.74	347.00	.52	497.00	.06
235.00	.23	269.95	4.36	308.90	.69	347.90	.21	499.40	.07
236.00	.23	270.95	.55	309.90	.21	361.05	.05	506.65	.03
238.00	.14	271.95	1.67	310.90	.35	383.55	.10	511.15	.04
239.00	2.46	273.95	.38	311.75	.06	388.45	.05	516.00	.09
240.00	4.28	274.95	14.06	312.90	.23	393.20	.04	519.25	.04
241.00	2.91	275.95	1.96	316.15	.05	399.15	.05	537.00	.05
242.00	4.44								



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WORKFILE NAME:

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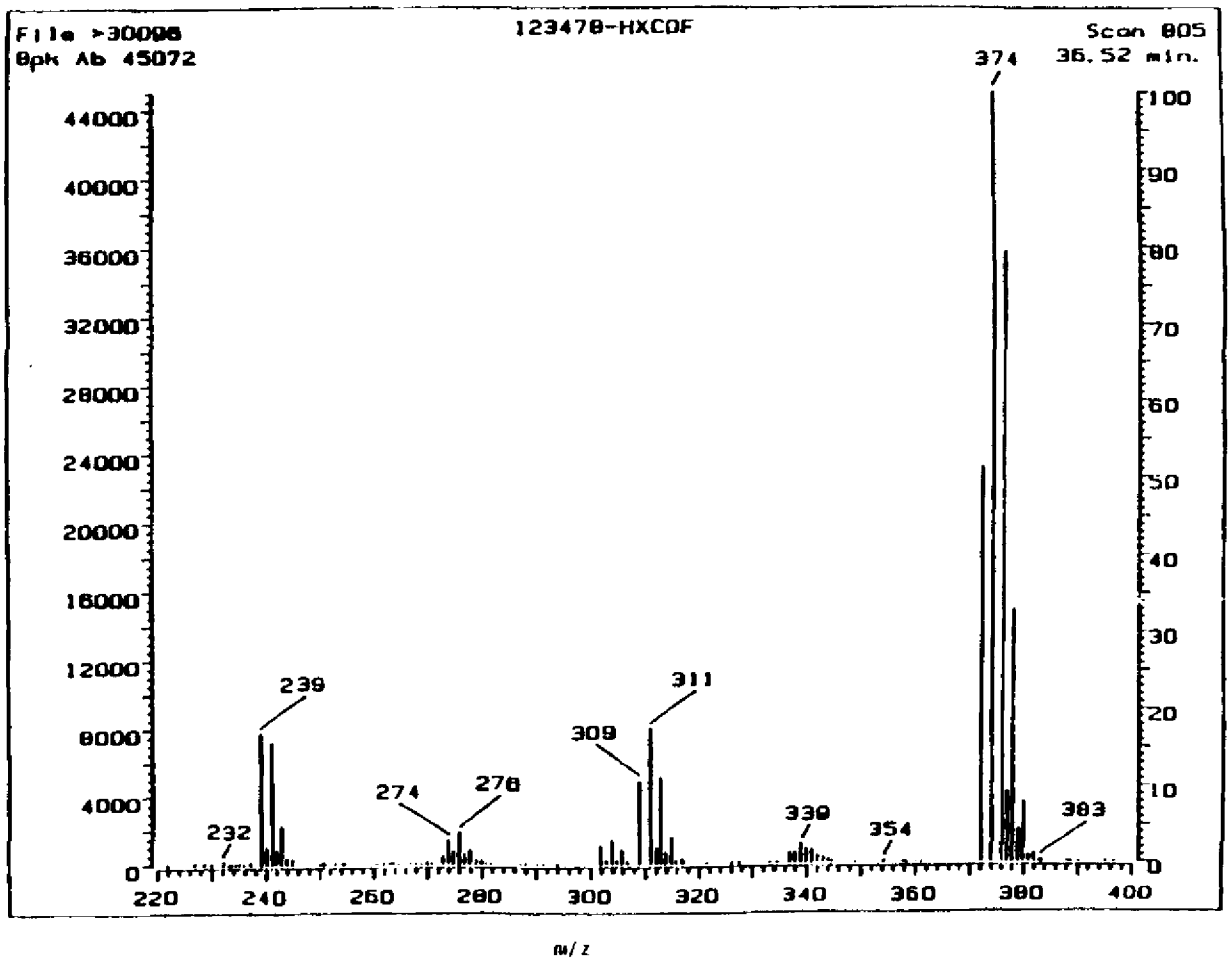
TOTAL AREA= 947950
AMT FACTOR= 1.00000E+00

A-5

MONS 218189

Gas Chromatogram of 1,2,3,4,7,8-Hexachlorodibenzofuran



A-6

MONS 218190

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805 NRM

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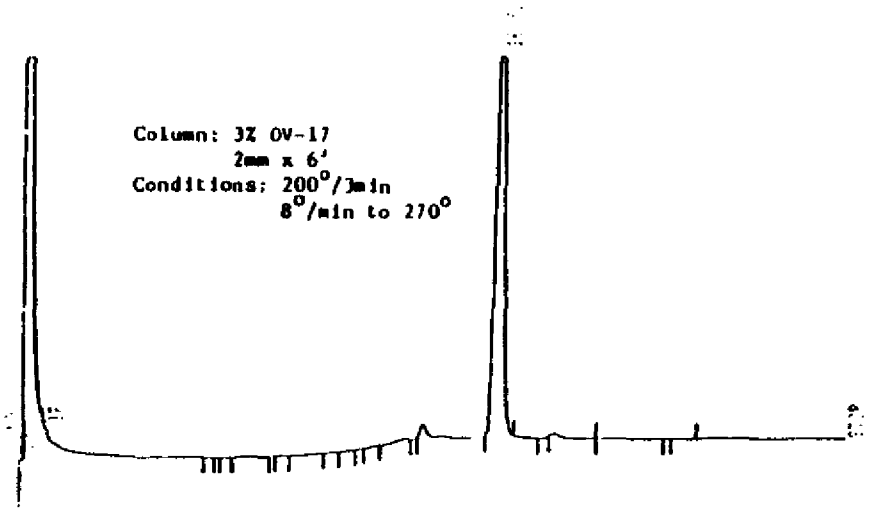
215.80	.19	260.90	.15	304.90	.47	371.90	51.53	425.45	.05
216.80	.54	261.90	.20	305.90	1.83	373.90	100.00	428.55	.00
217.70	.09	263.00	.17	306.90	.24	375.50	79.44	429.30	.10
218.30	.09	263.75	.17	308.80	10.73	376.50	9.57	429.90	.11
221.95	.08	264.50	.07	310.90	17.59	377.50	55.05	432.70	.09
226.95	.20	264.75	.08	311.95	2.16	378.90	4.41	433.05	.07
229.70	.18	265.75	.11	312.90	11.23	379.90	7.54	433.45	.05
229.95	.15	266.20	.19	313.90	1.42	380.90	.93	439.55	.08
231.95	.35	267.90	.19	314.95	3.42	381.90	1.13	444.05	.05
233.20	.11	268.90	.17	315.90	.50	382.90	.20	444.50	.05
233.70	.18	269.50	.09	316.55	.59	383.50	.11	444.80	.07
234.45	.09	270.90	.10	321.45	.07	390.15	.08	446.15	.05
235.20	.19	271.90	.12	326.05	.09	390.75	.07	447.15	.03
235.95	.14	272.90	1.12	327.45	.06	391.15	.06	448.00	.10
237.50	.05	273.90	3.05	333.20	.12	394.60	.09	448.65	.11
238.95	17.10	274.90	1.74	334.55	.08	395.20	.08	449.00	.04
239.95	2.35	275.90	4.06	335.95	1.40	396.55	.13	457.40	.09
240.95	15.89	276.90	1.59	337.95	1.40	400.20	.09	457.75	.07
241.95	1.09	277.90	1.99	339.95	2.40	402.70	.05	463.90	.05
242.85	4.99	278.90	.52	339.95	1.95	403.20	.05	464.40	.08
243.95	.75	279.90	.43	340.65	1.69	407.80	.05	466.40	.06
244.95	.76	280.75	.15	341.95	1.04	408.05	.07	466.75	.04
249.90	.11	281.65	.06	342.95	.55	410.80	.14	469.20	.07
250.05	.12	287.00	.06	343.95	.45	411.55	.07	485.55	.07
250.45	.17	287.90	.08	344.90	.24	412.55	.04	493.30	.11
250.95	.25	290.15	.09	349.00	.05	413.30	.08	496.70	.09
252.95	.20	291.25	.06	354.25	.22	414.55	.05	498.55	.09
254.20	.17	293.90	.08	357.65	.11	416.20	.11	499.30	.06
254.45	.16	301.80	1.35	358.25	.08	416.95	.09		

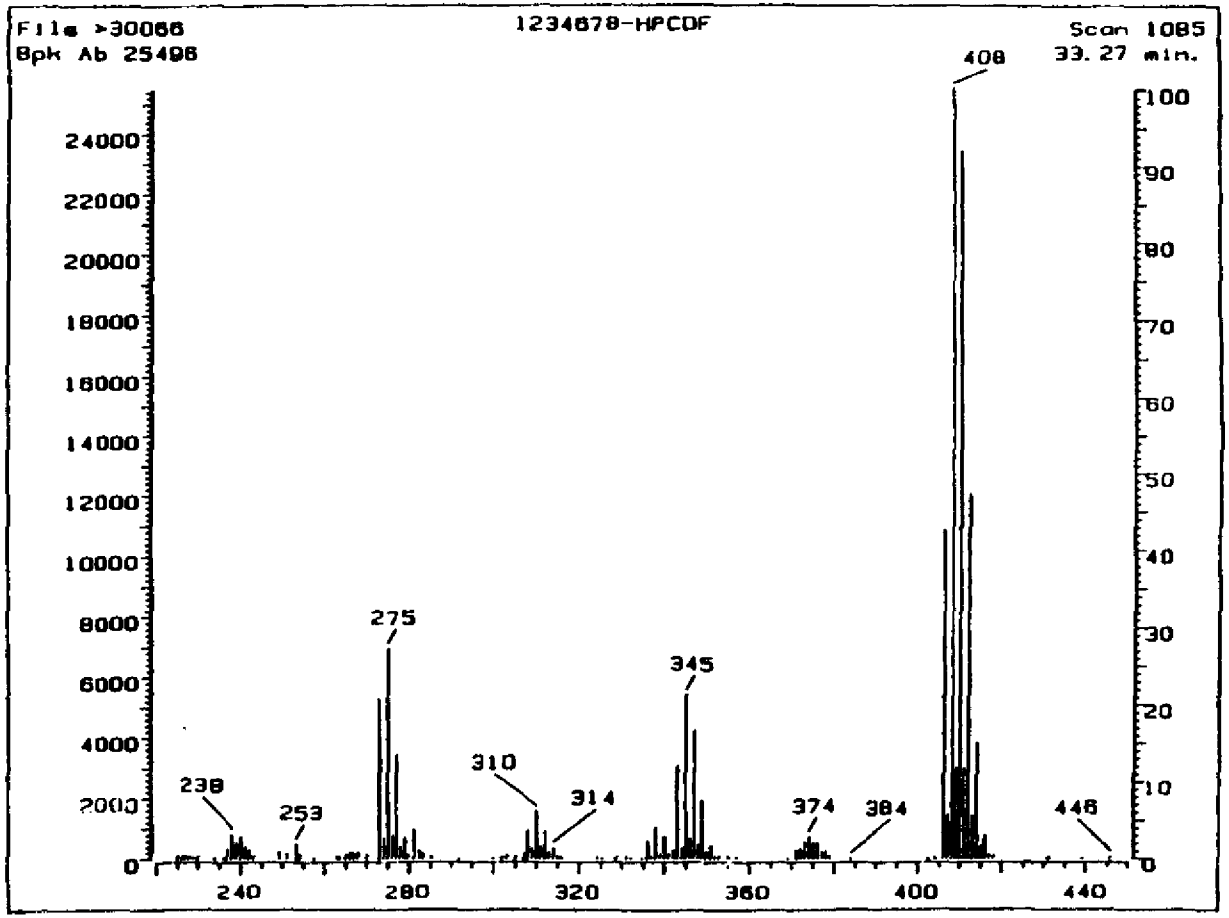
MONS 218191



A-8

MONS 218192

Gas Chromatogram of 1,2,3,4,6,7,8-Heptachlorodibenzofuran

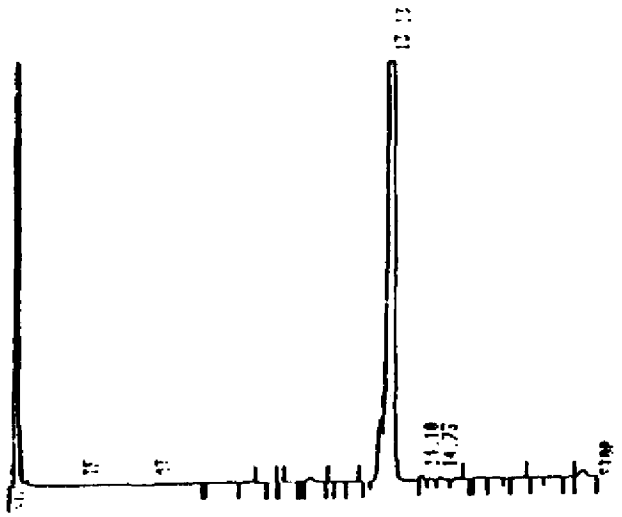

File >30066
Bpk Ab 25498
1234678-HPCDF
Scan 1085
33.27 min.
408
275
345
238
253
310
314
374
384
446
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
100
90
80
70
60
50
40
30
20
10
0
240
280
320
360
400
440
m/z

>30066 1234678-HPCDF
1085 NRM

File: >30066 Scan #: 1085 Retn. time: 33.27

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
212.90	.47	264.95	.56	308.90	1.45	345.90	2.69	407.90	100.00
214.90	.41	265.95	.84	309.90	6.06	346.90	16.61	408.90	11.95
217.00	.21	268.95	.70	310.90	1.76	347.90	2.00	409.90	92.38
224.90	.45	267.95	.81	311.90	3.87	348.80	7.50	410.90	11.73
226.00	.39	269.95	.42	312.90	1.02	349.90	1.04	411.90	47.24
226.90	.41	272.95	20.70	313.90	1.48	350.90	1.70	412.90	5.65
227.90	.30	273.95	2.93	315.00	.41	351.80	.33	413.80	15.05
229.50	.22	274.95	27.26	315.90	.22	352.80	.33	414.90	1.74
229.90	.29	275.95	3.30	324.25	.18	355.05	.36	415.80	2.95
233.90	.13	276.95	13.58	325.40	.08	356.95	.16	416.90	.43
236.00	.16	277.95	1.89	328.50	.15	389.20	.09	417.80	.42
236.90	1.35	278.95	2.72	329.90	.17	370.95	1.14	430.75	.38
239.00	3.15	279.80	.49	331.15	.33	371.95	1.43	431.80	.10
239.00	2.15	281.05	3.75	332.25	.16	372.95	2.22	439.40	.09
240.00	2.63	282.20	1.18	334.75	.15	373.95	2.83	445.70	.14
241.00	1.65	293.05	.74	335.90	2.26	374.95	2.16	467.05	.10
242.90	1.18	294.95	.32	336.90	.40	375.95	2.17	486.45	.08
243.00	.33	291.70	.09	337.90	4.07	376.95	.96	489.65	.12
249.00	.99	299.95	.14	339.00	.70	377.95	1.03	516.50	.14
251.00	.65	301.70	.22	340.00	2.87	378.95	.35	530.40	.10
253.15	1.90	301.90	.21	340.90	.73	383.95	.13	542.70	.11
254.15	.53	302.90	.49	342.00	1.15	402.50	.10	542.95	.12
257.70	.11	304.90	.23	342.90	11.96	404.15	.12	545.05	.10
262.95	.25	306.90	.75	344.00	1.50	405.80	42.57	545.85	.28
263.70	.18	307.90	3.82	344.90	21.23	406.80	5.79	547.55	.11

MONS 218194



RUN # 127 OCT/01/84 16:18:33
WORKFILE ID: A1
WORKFILE NAME:

AREA%

RT	AREA	TYPE	AR-HT	AREA%
12.73	21213	PV	0.151	3.674
13.13	553480	VB	0.188	95.918
14.10	1163	BP	0.122	0.202
14.23	1179	PP	0.159	0.204

TOTAL AREA= 577040
MIN FACTOR= 1.0000E+00

Gas Chromatogram of 2,3,7,8-Tetrachlorodibenzofuran-^{13}C

A-11

MONS 218195

File #30521
Bph Ab 8188
2378-TCDF-13C
Scan 977
25.53 min.
217
241
252
281
318
322
m/z

MONS 218196

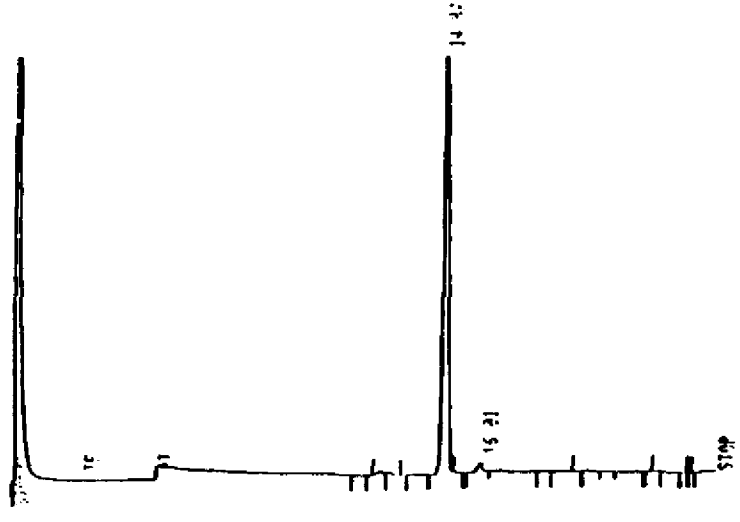
>30521 2378-TCDF-13C AWN 60M-DB5W: 35-200@30; 200-310@4.IH1,1uL.

S77 MFM

File: >30521 Scan #: S77 Retn. time: 25.53

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
207.25	5.27	246.15	3.36	254.05	14.25	284.20	1.27	317.15	13.82
208.25	1.03	247.25	.56	255.05	.79	285.05	1.15	318.00	100.00
209.15	.48	248.25	3.75	256.05	4.31	286.05	.60	319.15	7.25
216.25	3.44	250.25	1.36	280.05	.74	314.00	.78	320.15	48.45
217.15	3.96	251.25	1.65	281.20	4.55	315.00	9.70	321.15	7.31
218.25	2.54	252.05	14.26	282.05	3.00	316.15	80.31	322.15	4.21
219.25	2.27	253.20	2.35	283.05	3.35				

MONS 218197



PRN # 122 SEP/27/84 14:32:55
WORKFILE ID: C1
WORKFILE NAME:

AREAS:

RT	AREA	TYPE	AR/HT	AREA%
14.97	236850	PB	0.179	99.235
16.01	2791	PB	0.149	1.165

TOTAL AREA= 239640
AMT FACTOR= 1.0000E+00

A-14

Gas Chromatogram of 1,2,3,7,8-Pentachlorodibenzofuran-^{13}C

MONS 218198

File =30522
Bph Ab 42182
12378-PCDF-13C
Scan 1318
28.67 min.
216
245
253
282
288
317
350
352
m/z

MONS 210199

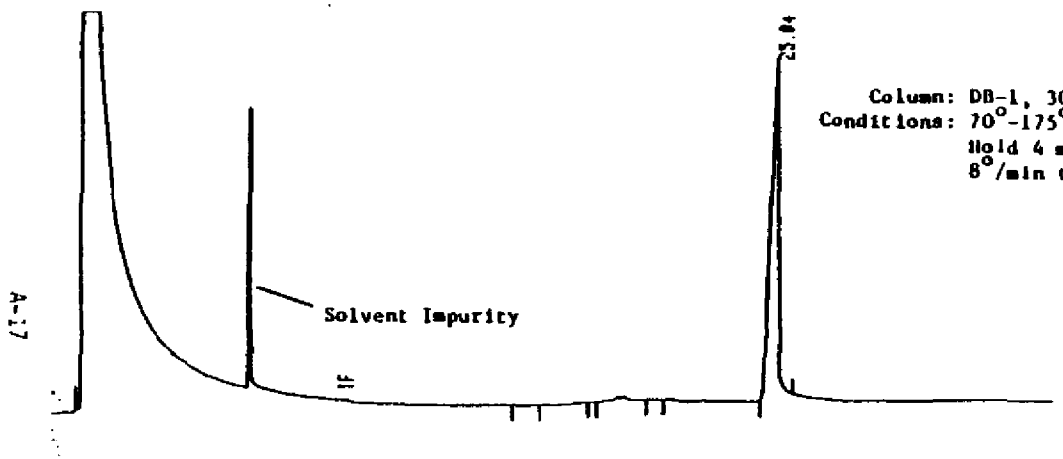
>30522 12378-FCCF-13C AWM60M-DE5W: 35-200@30; 200-310@4.IH1.tuL.

1318 MRM

File: >30522 Scan #: 1318 Retn. time: 28.67

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
214.25	.44	246.15	.15	267.20	.15	290.05	7.12	346.15	.42
215.25	2.16	247.25	.22	279.05	.59	291.05	.31	348.15	.89
216.15	16.97	248.25	.43	280.20	4.05	292.05	1.33	349.15	7.62
217.15	1.83	249.25	.49	281.20	2.42	314.00	.63	350.15	61.72
218.15	10.86	250.15	2.56	282.20	4.77	315.00	2.86	351.15	12.44
219.15	.46	251.15	3.51	283.20	.66	316.15	1.78	352.15	100.00
220.15	1.94	252.20	2.52	284.20	1.82	317.15	3.07	353.15	9.15
224.15	.56	253.05	3.55	285.05	1.48	318.15	1.59	354.15	60.50
226.15	.52	254.05	.98	286.05	12.55	319.00	1.50	355.15	3.31
228.15	.19	255.05	1.11	287.05	1.99	320.15	.85	356.15	19.80
238.15	.13	256.05	.16	288.05	14.68	321.00	.34	357.00	.66
244.25	.13	265.05	.11	289.05	1.09	322.15	.30	358.15	3.09
245.25	.28								

MONS 218200



Column: DB-1, 30M
Conditions: 70°-175°C @ 30°/min,
Hold 4 min.
8°/min to 270°C

RUN # 466 JAN/31/85 10:51:05
WORKFILE ID: 85
WORKFILE NAME:

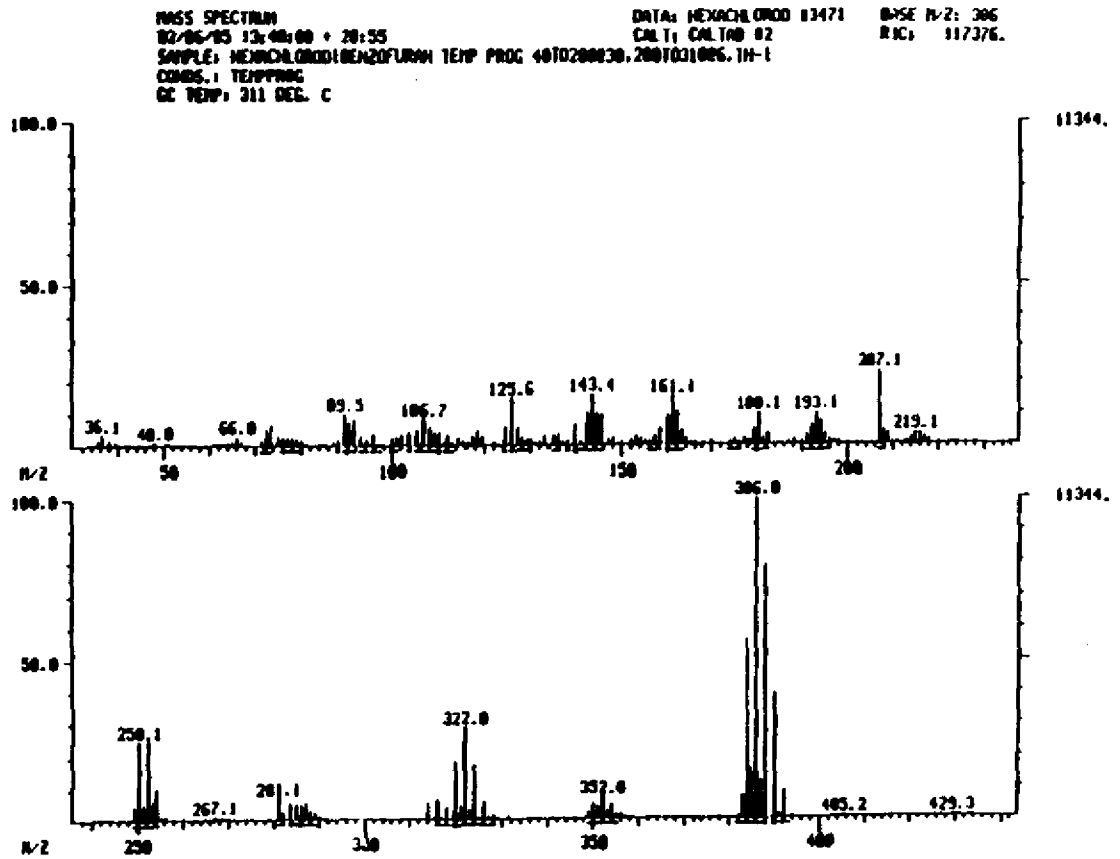
AREA%

RT	AREA	TYPE	AR/HT	AREA%
25.04	89399	PV	0.237	100.00

TOTAL AREA= 89398
MUL FACTOR= 1.0000E+00

Gas Chromatogram of 1,2,3,4,7,8-Hexachlorodibenzofuran-^{13}C

MONS 218201


MASS SPECTRUM
02/06/95 13:48:00 + 20:55
SAMPLE: HEXACHLORODIBENZOFURAN TEMP PROG 40T0200030.200T031005.1H-1
COMDS.: TEMPPROG
GC TEMP: 311 DEG. C
DATA: HEXACHLORDD 03471
CALI: CALTAB 02
BASE M/Z: 306
RIC: 117376.
100.0
50.0
M/Z
36.1
40.0
66.0
89.5
105.7
125.6
143.4
161.1
180.1
193.1
207.1
219.1
50
100
150
200
11344.
100.0
50.0
M/Z
250.1
267.1
281.1
322.0
352.0
306.0
405.2
429.3
250
300
350
400
11344.

MONS 218202

Mass List Data: HEXACHLOROD 83471 Base m/z: 284
02/06/85 13:40:00 + 28:55 Call: CALTAB # 2 RIC: 117376.
Sample: HEXACHLORODIBENZOFURAN TEMP PROG 40T0200830.200T0310B4.IN-1
Comm.: TEMPPROG

35 0.00 0.00 0. Minima Min Inten: 236.
429 0 0 Maxima

Mass		% RA	% RIC	Inten.	Mass		% RA	% RIC	Inten.
36.067		3.48	0.34	353.	182.09	F	3.31	0.32	329.
44.047		3.06	0.30	347.	191.11	F	3.29	0.39	407.
72.48	F	5.27	0.51	598.	192.08	F	6.77	0.61	711.
73.43	F	6.37	0.63	745.	193.08	F	9.98	0.96	1132.
75.03		3.01	0.29	341.	194.03	F	7.33	0.71	832.
76.86	F	2.46	0.24	279.	199.03	F	3.31	0.34	398.
78.02	F	2.34	0.23	283.	207.12	F	22.67	2.19	2572.
79.12	F	2.24	0.22	256.	208.11	F	6.70	0.65	333.
89.46	F	9.71	0.94	1102.	209.11		3.49	0.34	396.
90.29	F	6.83	0.66	775.	214.09		2.58	0.25	293.
91.60	F	8.37	0.81	950.	215.06		3.68	0.36	418.
93.29	F	3.07	0.30	348.	216.06		3.43	0.33	389.
95.88	F	3.63	0.35	412.	217.08		2.12	0.21	241.
100.01	F	2.27	0.22	258.	249.08	F	4.77	0.46	541.
101.05	F	2.11	0.20	239.	250.06	F	25.28	2.44	2868.
102.02		2.96	0.29	336.	251.08	F	8.14	0.50	383.
103.64	F	4.24	0.41	481.	252.06	F	24.79	2.40	2812.
105.08	F	4.71	0.45	534.	253.11	F	6.31	0.61	716.
106.73	F	8.07	0.78	915.	254.06	F	9.43	0.93	1092.
107.84	F	5.93	0.58	673.	281.09	F	6.77	0.69	768.
108.85	F	4.06	0.39	460.	282.12	F	2.29	0.22	260.
110.01	F	4.19	0.40	475.	283.69	F	5.31	0.51	602.
112.03	F	3.44	0.33	390.	285.03	F	4.76	0.46	540.
114.05		2.38	0.23	270.	286.00	F	4.43	0.43	503.
117.11		2.66	0.26	302.	287.00	F	5.10	0.49	578.
118.06		4.41	0.43	500.	288.03	F	3.14	0.30	356.
119.08		2.83	0.27	321.	289.03	F	2.59	0.25	294.
124.41	F	5.61	0.54	636.	313.91		5.15	0.50	584.
125.65	F	14.69	1.42	1666.	316.03	F	6.26	0.60	710.
127.01	F	3.14	0.30	383.	318.03	F	3.91	0.38	444.
128.08	F	2.45	0.24	278.	319.06	F	2.79	0.27	317.
133.09		2.79	0.27	316.	320.00	F	18.37	1.78	2084.
135.09	F	3.06	0.30	347.	321.00	F	4.53	0.44	514.
136.03	F	3.24	0.31	367.	322.00	F	28.88	2.79	3276.
139.83	F	6.52	0.63	740.	323.00	F	3.37	0.33	382.
142.24	F	9.87	0.99	1120.	324.00	F	17.72	1.71	2010.
143.39	F	13.57	1.30	1784.	326.00	F	5.80	0.56	658.
144.25	F	10.03	0.97	1138.	348.97	F	2.43	0.24	276.
145.34	F	9.13	0.88	1036.	350.00	F	4.67	0.45	530.
148.05		2.13	0.21	244.	351.00	F	3.83	0.37	434.
153.00		3.19	0.31	362.	352.00	F	7.05	0.68	800.
157.12	F	2.90	0.28	329.	353.03	F	3.00	0.29	340.
158.47	F	5.37	0.52	609.	354.00	F	4.79	0.46	543.
160.06	F	7.34	0.92	1082.	383.00	F	7.73	0.73	879.
161.06	F	13.57	1.49	1744.	385.97	F	36.28	3.44	4084.
162.05	F	10.39	1.00	1174.	389.00	F	16.04	1.55	1820.
163.02	F	4.87	0.47	553.	386.00	F	100.00	9.64	11344.
164.06		2.33	0.22	264.	387.03	F	13.03	1.26	1480.
177.08		2.93	0.24	287.	388.00	F	78.84	7.62	8944.
179.11	F	5.39	0.52	611.	389.84	F	39.36	3.82	4488.
180.11	F	9.86	0.95	1118.	391.97	F	8.80	0.85	998.

MONS 218203

Appendix B

SAMPLE CONTROL

The following pages contain sample numbers, log-in codes and other sample control information generated during this program.

MONS 218204

PAGE 1 Radian Corp WORK SHEET ORO # AS-09-049
CLIENT: EPRI CON: GARRETSOR CAT: RCVD: 09/26/85 DUE: 10/10/85
PROJ: 01/23/86 11:10:44 STAT: TRANSMITTED 01/23/86

DASH	SAMPLE IDENTIFICATION	STORED	DEPT	START	DUE BY	TESTS in DEPT: MS
01B	E729-53-01 MINERAL OIL	D.HAYES	MS	09/10	10/10	DIOFUR
01D	E729-53-01 MINERAL OIL	D.HAYES	MS	09/10	10/10	DIOFUR
01F	E729-53-01 MINERAL OIL	D.HAYES	MS	09/10	10/10	DIOFUR
02B	E729-53-01 AROCLOR 1016	D.HAYES	MS	09/10	10/10	DIOFUR
02D	E729-53-02 AROCLOR 1016	D.HAYES	MS	09/10	10/10	DIOFUR
02F	E729-53-02 AROCLOR 1016	D.HAYES	MS	09/10	10/10	DIOFUR
03B	E729-53-03 AROCLOR 1260	D.HAYES	MS	09/10	10/10	DIOFUR
03D	E729-53-03 AROCLOR 1260	D.HAYES	MS	09/10	10/10	DIOFUR
03F	E729-53-03 AROCLOR 1260	D.HAYES	MS	09/10	10/10	DIOFUR

MONS 218205

PAGE 1 Radian Corp WORK SHEET ORD # A5-10-015
CLIENT: EPRI CON: NICHOLS CAT: RCVD: 10/14/05 DUE: 12/01/05
PROJ: 01/22/06 16:50:56 STAT: TRANSMITTED 01/22/06

DASH	SAMPLE IDENTIFICATION	STORED	DEPT	START	DUE BY	TESTS in DEPT: MS
01B	41159-11-14	REFRIG 11	MS	11/01	12/01	DIOXIN
01D	41159-11-14	REFRIG 11	MS	11/01	12/01	DIOXIN
01F	41159-11-14	REFRIG 11	MS	11/01	12/01	DIOXIN
02B	41159-11-11	REFRIG 11	MS	11/01	12/01	DIOXIN
02D	41159-11-11	REFRIG 11	MS	11/01	12/01	DIOXIN
02F	41159-11-11	REFRIG 11	MS	11/01	12/01	DIOXIN
03B	41159-11-12	REFRIG 11	MS	11/01	12/01	DIOXIN
03D	41159-11-12	REFRIG 11	MS	11/01	12/01	DIOXIN
03F	41159-11-12	REFRIG 11	MS	11/01	12/01	DIOXIN
04B	41159-11-16	REFRIG 11	MS	11/01	12/01	DIOXIN
04D	41159-11-16	REFRIG 11	MS	11/01	12/01	DIOXIN
04F	41159-11-16	REFRIG 11	MS	11/01	12/01	DIOXIN
05B	41159-11-17	REFRIG 11	MS	11/01	12/01	DIOXIN
05D	41159-11-17	REFRIG 11	MS	11/01	12/01	DIOXIN
05F	41159-11-17	REFRIG 11	MS	11/01	12/01	DIOXIN
06B	41159-11-15	REFRIG 11	MS	11/01	12/01	DIOXIN
06D	41159-11-15	REFRIG 11	MS	11/01	12/01	DIOXIN
06F	41159-11-15	REFRIG 11	MS	11/01	12/01	DIOXIN
07B	41159-11-13	REFRIG 11	MS	11/01	12/01	DIOXIN
07D	41159-11-13	REFRIG 11	MS	11/01	12/01	DIOXIN
07F	41159-11-13	REFRIG 11	MS	11/01	12/01	DIOXIN

MONS 218206

PAGE 1 Radian Corp DATA SHEET ORG # A6-03-007-MS

CLIENT: EPRI CON: AMNICHOLS CAT: RCVD: 03/06/86 DUE: 03/31/86

PROJ: STAT: TRANSMITTED 03/06/86

COMPANY ELECTRIC POWER RESEARCH INST.

FACIL

REP NA

PHONE NA

WORK IO 236-043-04-01

TAKEN

TRANS

TYPE

CREATED 03/06/86 TRANSMITTED 03/06/86

WRITTEN 03/06/86 PREV TRANS

QUOTED $ FIRST TRANS 03/06/86

SAMPLE $ 0.00 SAMPLES 2

NOTES

DEPTS/ TESTS		DASH	SAMPLE IDENTIFICATION	STORED	JOB
		01A	E-729-54-02 AGED MIN. OIL	MNL	
		01B	E-729-54-02 AGED MIN. OIL	MNL	
MS	1	02A	E-729-54-03 AROCHLOR 1260	MNL	
EX	1	02B	E-729-54-03 AROCHLOR 1260	MNL	
		03A	E7-29-54-04 AROCLOR 1016	MNL	
		03B	E7-29-54-04 AROCLOR 1016	MNL	

MONS 218207

PAGE 2 Radian Corp DATA SHEET ORD # A6-03-007-MS
CLIENT: EPRI CON: AMNICHOLS CAT: RCVD: 03/08/86 DUE: 03/31/86
PROJ: STAT: TRANSMITTED 03/08/86

DEPT MS DASH #01B ID E-729-54-02 AGED MIN. OIL STORED HHL JOB
CATEGORY WHEN COLLECTED MS-DIOFUR
SAMPLE AND WORK DESCRIPTION
INFO TO D HAYES AND D HARDIN

DIOFUR

DEPT MS DASH #02B ID E-729-54-01 AROCHLOR 1260 STORED HHL JOB
CATEGORY WHEN COLLECTED MS-DIOFUR
SAMPLE AND WORK DESCRIPTION
INFO TO D HAYES AND D HARDIN

DIOFUR

DEPT MS DASH #01B ID E7-29-54-01 AROCLOR 1016 STORED HHL JOB
CATEGORY WHEN COLLECTED MS-DIOFUR
SAMPLE AND WORK DESCRIPTION
INFO TO D HAYES AND D HARDIN

DIOFUR

MONS 218208

Appendix C

CALIBRATION DATA: ULTRA-2

The following pages contain the first set of calibration curves and linear regression data generated on Ultra-2.

FIVE-POINT CALIBRATION IN TRIPLICATE [ULTRA-2 50m H-P]

AREA RATIOS RELATIVE TO CORRESPONDING 13C ISOMER

	2378 TCDD	2378 TCDF	12378 PeCDF	23478 PeCDF	123478 HxCDF	234678 HxCDF	1234678 HpCDF	OCDF
40 ng								
VALUE	0.0750	0.1650	0.1150	0.1010	0.0940	0.0814	0.0910	0.1900
VALUE	0.0796	0.1660	0.1050	0.1030	0.1120	0.1044	0.0891	0.1810
VALUE	0.0770	0.1170	0.1100	0.1020	0.0954	0.10e0	0.0759	0.1020
AVG.	0.0772	0.1493	0.1083	0.1019	0.1011	0.1005	0.0850	0.1510
S.D.	0.0026	0.0067	0.0029	0.0010	0.0143	0.0079	0.0087	0.0105
C.V.	4.7275	6.0899	2.e647	0.9901	10.2207	7.8786	10.2794	5.2160
150 ng								
VALUE	0.3090	0.4e0e	0.3699	0.4130	0.3810	0.3220	0.3229	0.5880
VALUE	0.2620	0.3420	0.3250	0.3350	0.3740	0.3960	0.3500	0.5790
VALUE	0.3110	0.4580	0.4220	0.4240	0.4e6e	0.3360	0.3250	0.7420
AVG.	0.2940	0.4033	0.3857	0.3907	0.4070	0.3487	0.3317	0.6420
S.D.	0.0277	0.0582	0.0481	0.0465	0.0512	0.0371	0.0206	0.0859
C.V.	9.4310	14.4497	12.4e85	12.4292	12.5836	11.2161	9.1559	13.3812
500 ng								
VALUE	0.9790	1.1410	1.1160	1.1500	1.0410	0.9500	0.9210	1.5900
VALUE	0.8910	1.0360	0.9550	1.0090	0.9e80	0.9e40	0.9e50	1.4e00
VALUE	0.9560	1.0e00	0.9750	1.0510	0.9710	0.9e50	0.9390	1.36e0
AVG.	0.94e0	1.0e70	1.0487	1.0700	1.0000	0.9507	0.9417	1.4e33
S.D.	0.0404	0.0634	0.0e9e	0.0641	0.0e65	0.0255	0.0211	0.1521
C.V.	4.2e08	5.8237	5.1e77	5.9594	5.e210	2.7e78	2.1e77	9.e478
2500 ng								
VALUE	3.7490	5.3100	4.8e20	5.1e00	4.7100	4.5e00	2.1000	8.2e00
VALUE	3.8110	5.1e50	4.8e00	5.1500	4.8200	4.e000	2.0940	8.25e0
VALUE	NA	NA	NA	NA	NA	NA	NA	NA
AVG.	3.7755	5.2275	4.8e10	5.1250	4.7650	4.5e90	2.0970	8.1503
S.D.	0.0502	0.1025	0.0014	0.0354	0.0849	0.0785	0.0042	0.1044
C.V.	1.3297	1.9576	0.0291	0.6897	1.7799	0.e176	0.1970	1.e475
7500 ng								
VALUE	13.0000	17.0e00	15.4900	16.4800	14.6700	14.5400	6.7900	24.5700
VALUE	11.7690	16.0800	14.7e00	15.7500	14.7500	14.4700	6.5900	21.e600
VALUE	11.5209	15.2900	14.e500	15.4500	14.0900	13.7e00	6.3000	20.1200
AVG.	11.7e97	16.4767	14.9767	15.8900	14.5400	14.2e67	6.4e00	22.79e0
S.D.	0.2407	0.51e0	0.4464	0.5072	0.4715	0.4e42	0.4137	1.5571
C.V.	2.0429	3.1216	2.9e07	3.2155	3.2427	2.e0e9	6.2419	6.e72e

MONS 218210

FIVE-POINT CALIBRATION IN TRIPLICATE (ULTRA-2 50m H-P)

AVERAGE AREA RATIOS RELATIVE TO CORRESPONDING IGC ISOMER

NG	2378 TCDD	2378 TCDF	12378 PnCDF	23478 PnCDF	123478 HxCDF	234678 HxCDF	1234678 HpCDF	OCDF
40	0.0772	0.1093	0.1083	0.1010	0.1011	0.1005	0.0850	0.1910
150	0.2940	0.4030	0.3857	0.3907	0.4070	0.3487	0.2517	0.6430
500	0.8460	1.0690	1.0357	1.0770	1.0000	0.9207	0.6410	1.6833
2500	3.7755	5.2375	4.3610	5.1250	4.7700	4.5800	3.0970	8.1550
7500	11.7667	16.4767	14.9767	15.9000	14.5400	14.2667	9.4600	22.7900

.....

LOG (ng)	LOG (average response factor)							
1.6021	-1.1124	-0.9614	-0.9654	-0.9957	-0.9952	-0.9978	-1.0706	-0.7190
2.1761	-0.5317	-0.3947	-0.4138	-0.4082	-0.3904	-0.4575	-0.5991	-0.1918
2.6990	-0.0726	0.0290	0.0152	0.0322	0.0000	-0.0359	-0.1931	0.2262
3.3979	0.5770	0.7191	0.6867	0.7097	0.6785	0.6609	0.4909	0.9114
3.8751	1.0707	1.2169	1.1754	1.2014	1.1626	1.1543	0.9759	1.3577

Y-ESTIMATES

-1.1028	-0.9671	-0.9715	-0.9891	-0.9808	-1.0148	-1.1113	-0.7301
-0.5582	-0.4227	-0.4359	-0.4406	-0.4448	-0.4749	-0.5952	-0.2066
-0.0822	0.0733	0.0220	0.0590	0.0434	0.0169	-0.1251	0.2703
0.6009	0.7362	0.7042	0.7268	0.6961	0.6743	0.5034	0.9077
1.0536	1.1888	1.1494	1.1827	1.1415	1.1230	0.9324	1.3429

MONS 218211

FIVE-POINT CALIBRATION IN TRIPLICATE IULTRA-2 50m H-P1

TCDD

Regression Output:

Constant	-2.6227
Std Err of Y Est	0.0243
R Squared	0.9994
No. of Observations	5.0000
Degrees of Freedom	3.0000

X Coefficient(s)	0.9487
Std Err of Coef.	0.0133

TCDF

Regression Output:

Constant	-2.4867
Std Err of Y Est	0.0359
R Squared	0.9987
No. of Observations	5.0000
Degrees of Freedom	3.0000

X Coefficient(s)	0.9485
Std Err of Coef.	0.0196

1237B-PnCDF

Regression Output:

Constant	-2.4664
Std Err of Y Est	0.0309
R Squared	0.9990
No. of Observations	5.0000
Degrees of Freedom	3.0000

X Coefficient(s)	0.9331
Std Err of Coef.	0.0169

2347B-PnCDF

Regression Output:

Constant	-2.5199
Std Err of Y Est	0.0296
R Squared	0.9992
No. of Observations	5.0000
Degrees of Freedom	3.0000

X Coefficient(s)	0.9555
Std Err of Coef.	0.0157

MONS 218212

FIVE-POINT CALIBRATION IN TRIPLICATE [ULTRA-2 50m H-P]

123478-HxCDF
Regression Output:

Constant	-2.4766
Std Err of Y Est	0.0440
R Squared	0.9980
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.9827
Std Err of Coef.	0.0241

123678-HxCDF
Regression Output:

Constant	-2.5215
Std Err of Y Est	0.0389
R Squared	0.9985
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.9405
Std Err of Coef.	0.0213

123478-HpCDF
Regression Output:

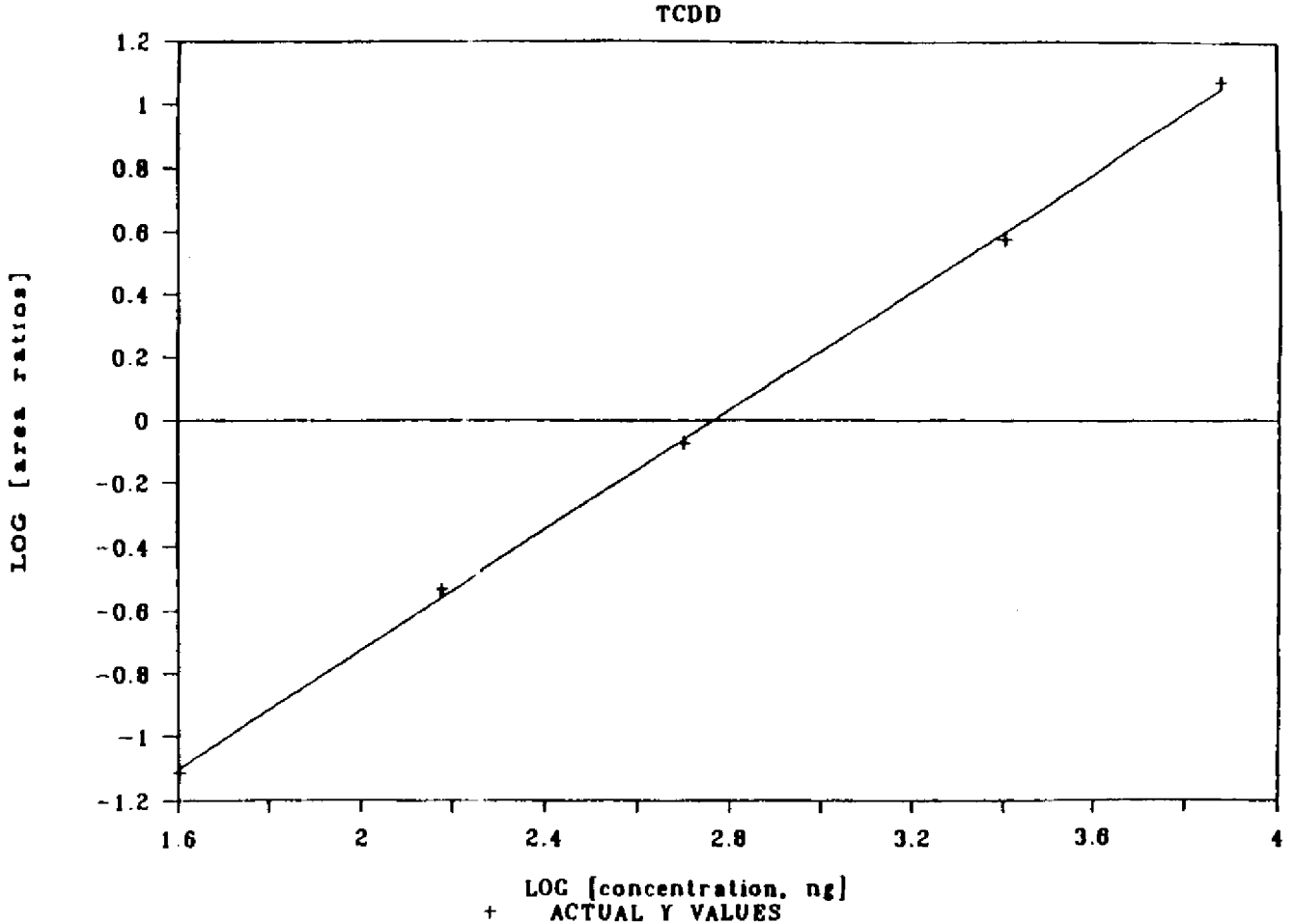
Constant	-2.5517
Std Err of Y Est	0.0528
R Squared	0.9969
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.8991
Std Err of Coef.	0.0289

OCDF
Regression Output:

Constant	-2.1912
Std Err of Y Est	0.0290
R Squared	0.9991
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.9120
Std Err of Coef.	0.0159

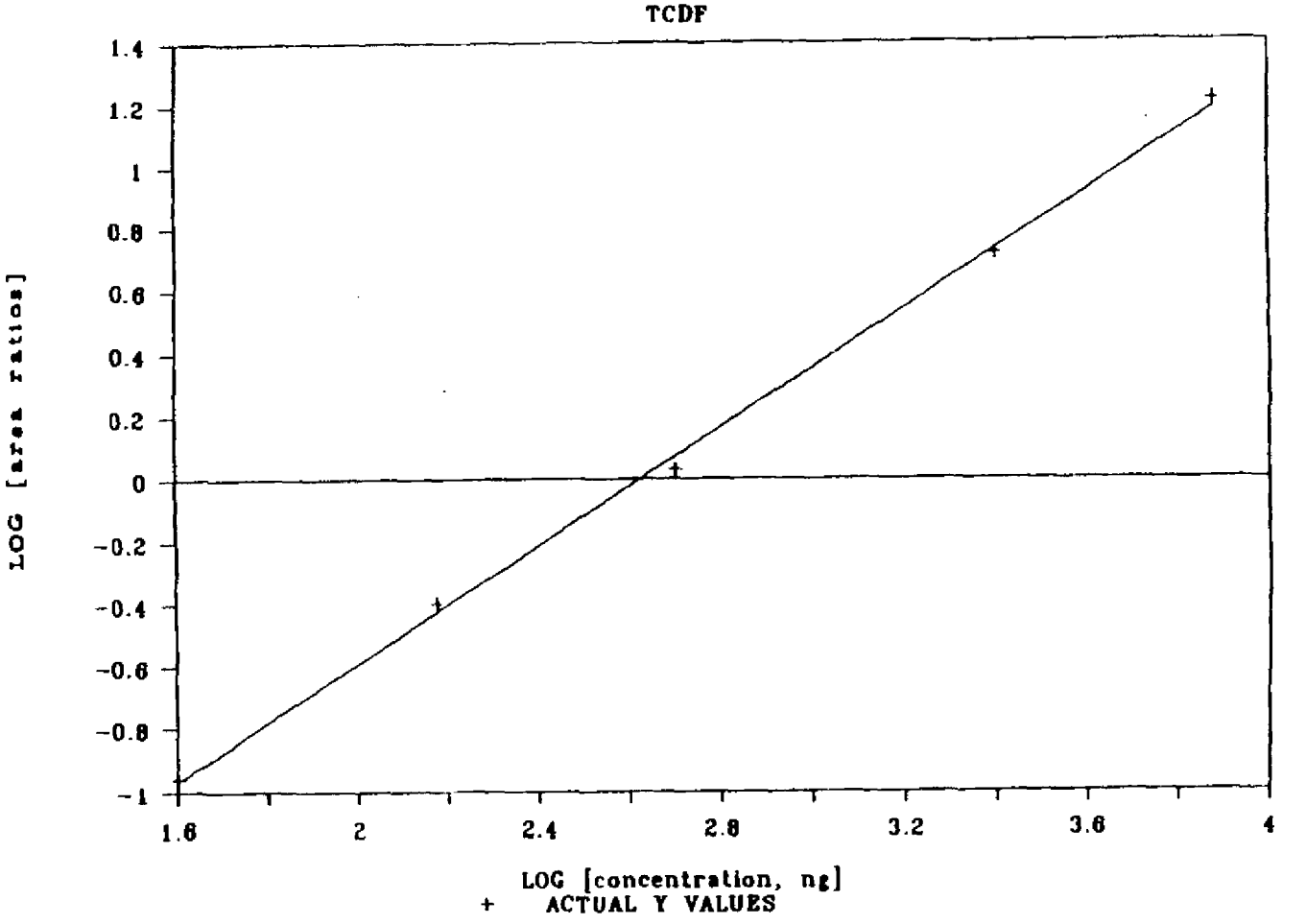
MONS 218213

CALCULATED ULTRA-2 CALIBRATION CURVE



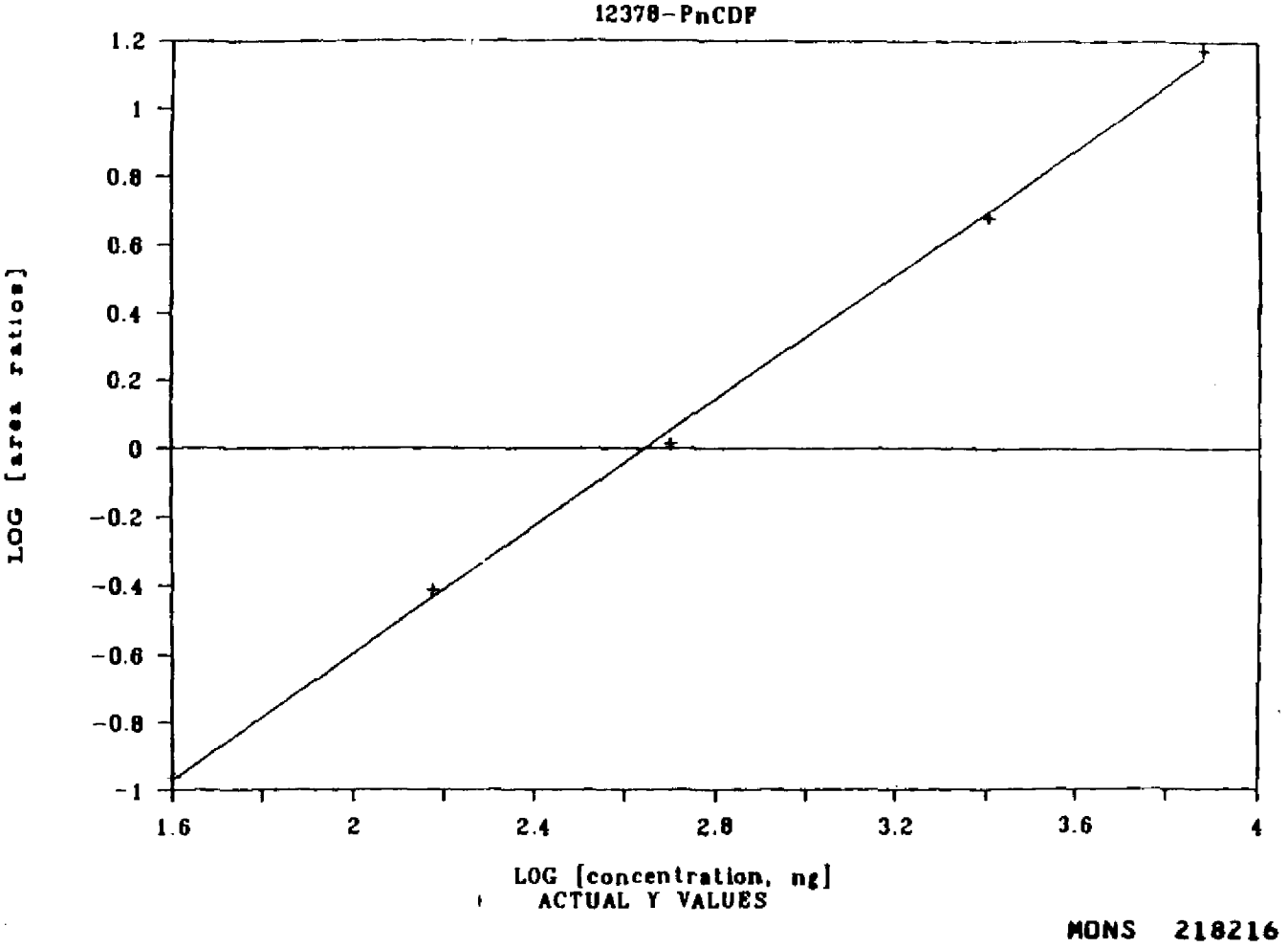
MONS 218214

CALCULATED ULTRA-2 CALIBRATION CURVE



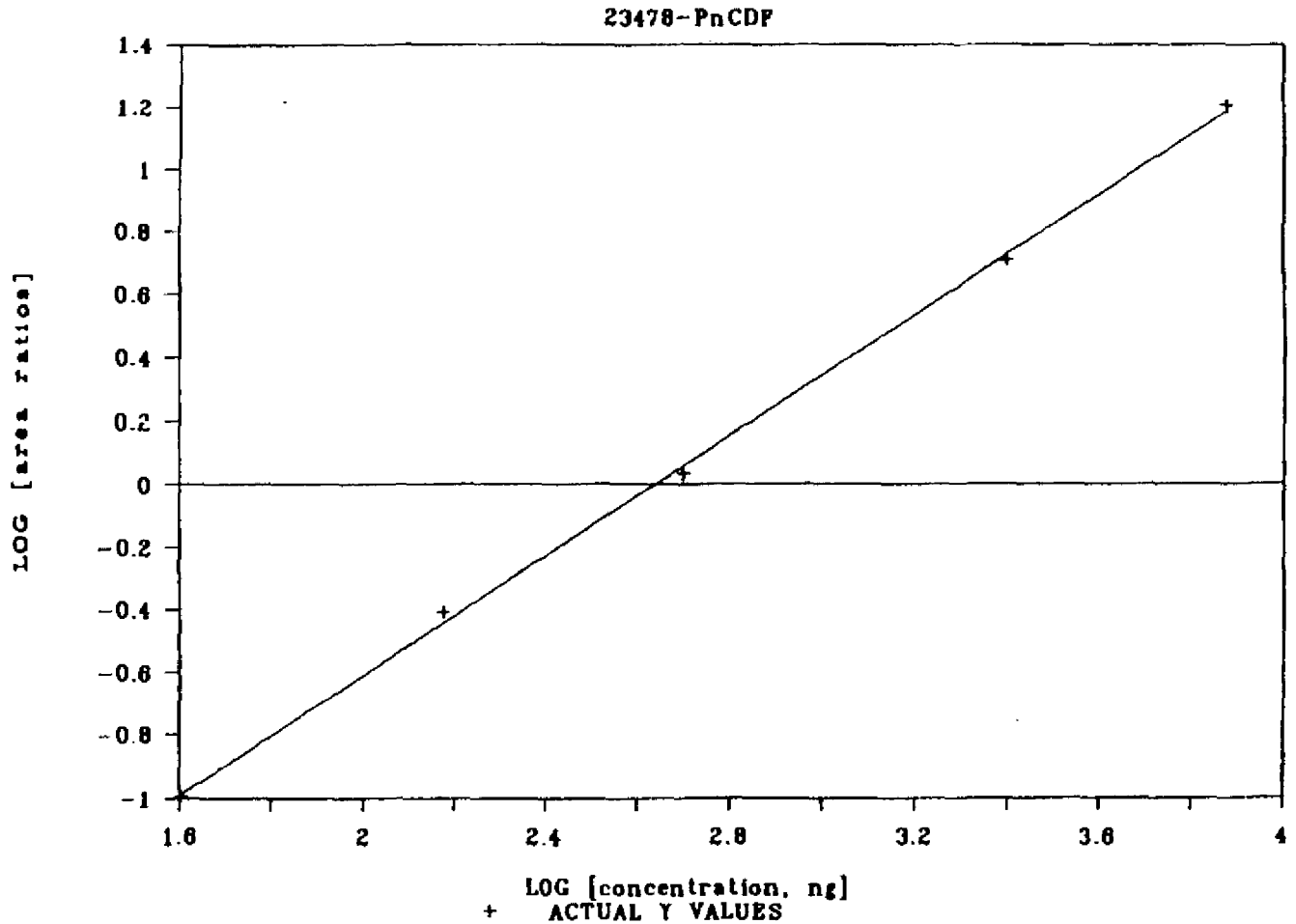
MONS 218215

CALCULATED ULTRA-2 CALIBRATION CURVE



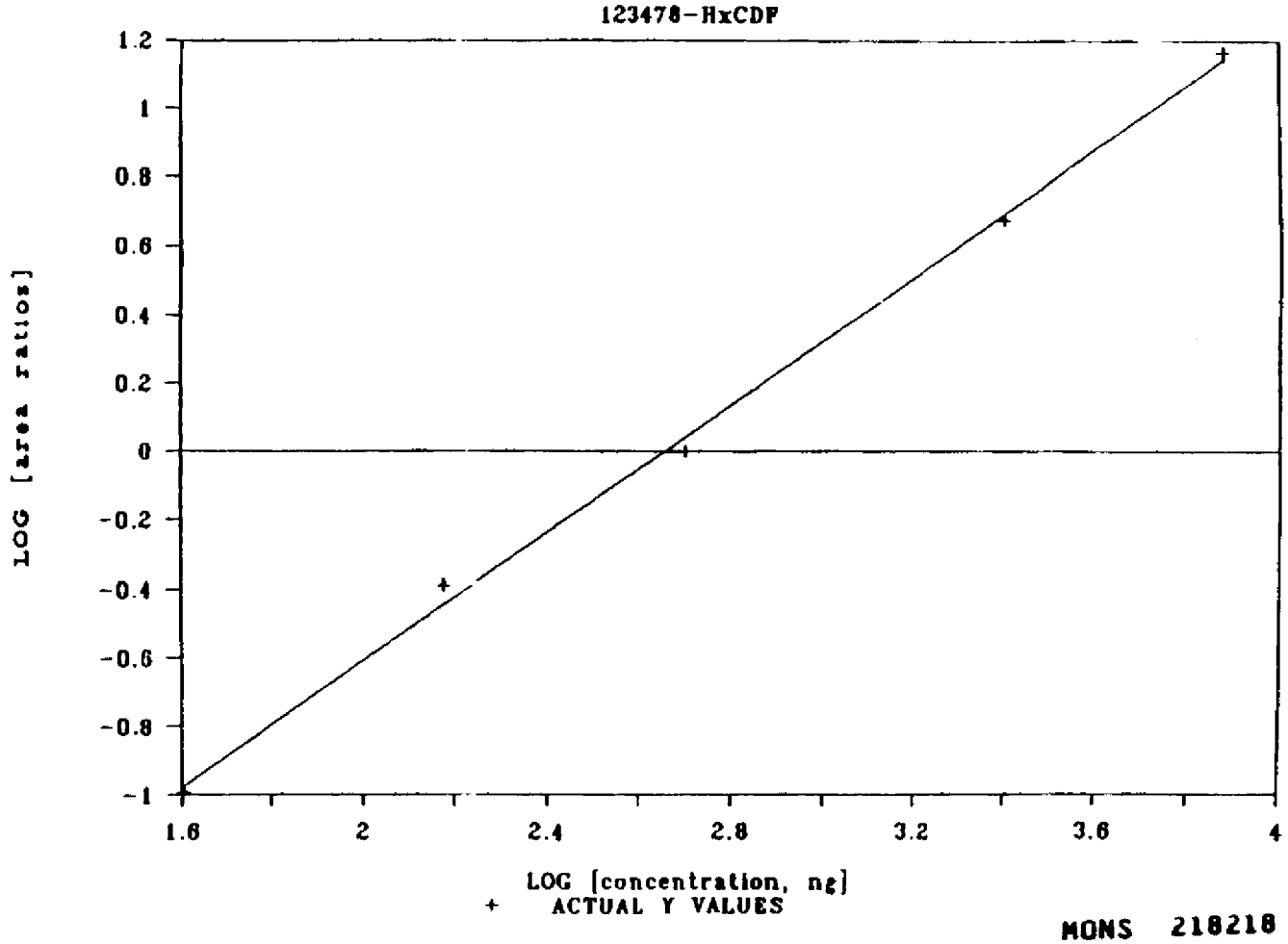
MONS 218216

CALCULATED ULTRA-2 CALIBRATION CURVE

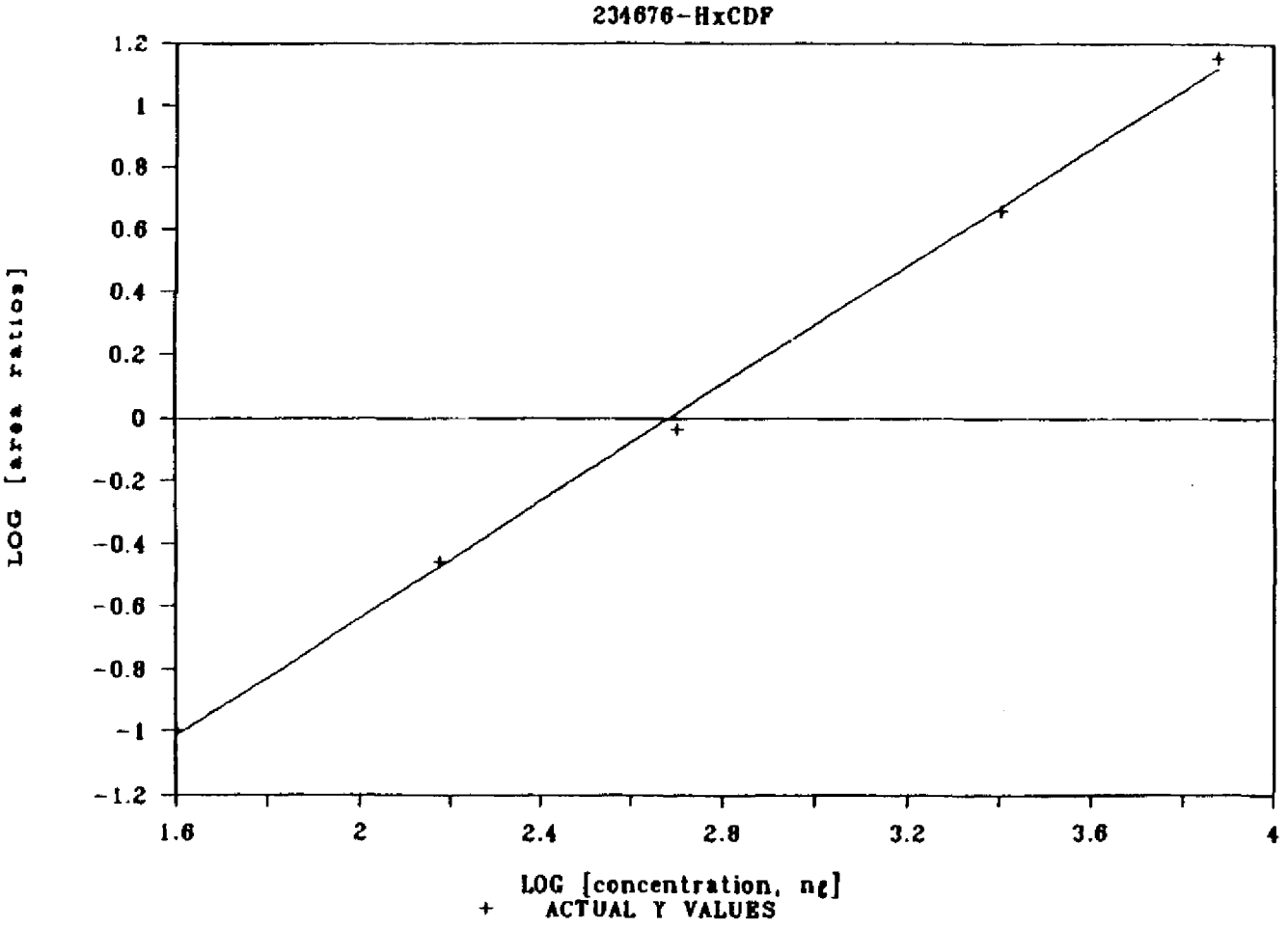


MONS 218217

CALCULATED ULTRA-2 CALIBRATION CURVE

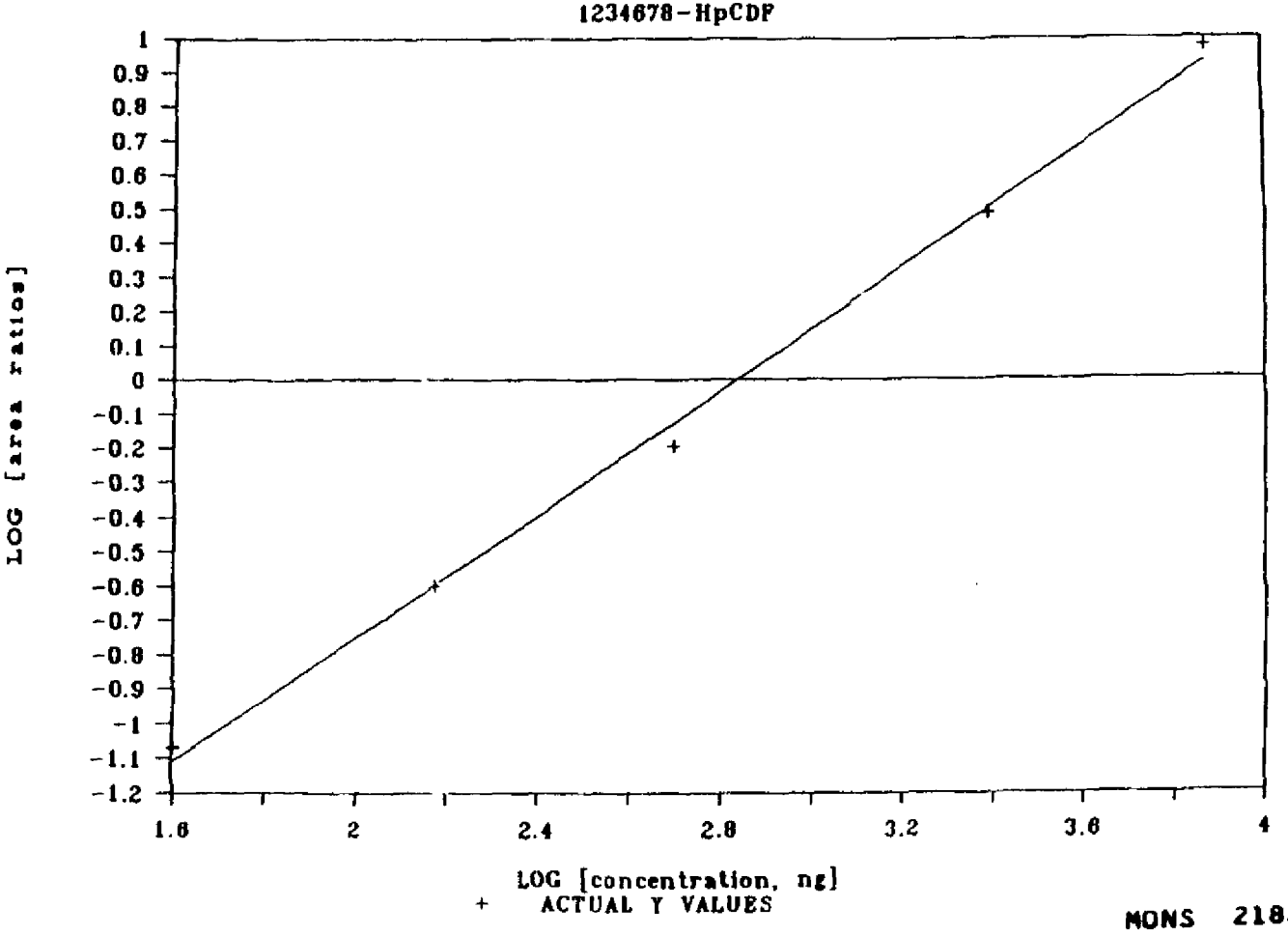


CALCULATED ULTRA-2 CALIBRATION CURVE

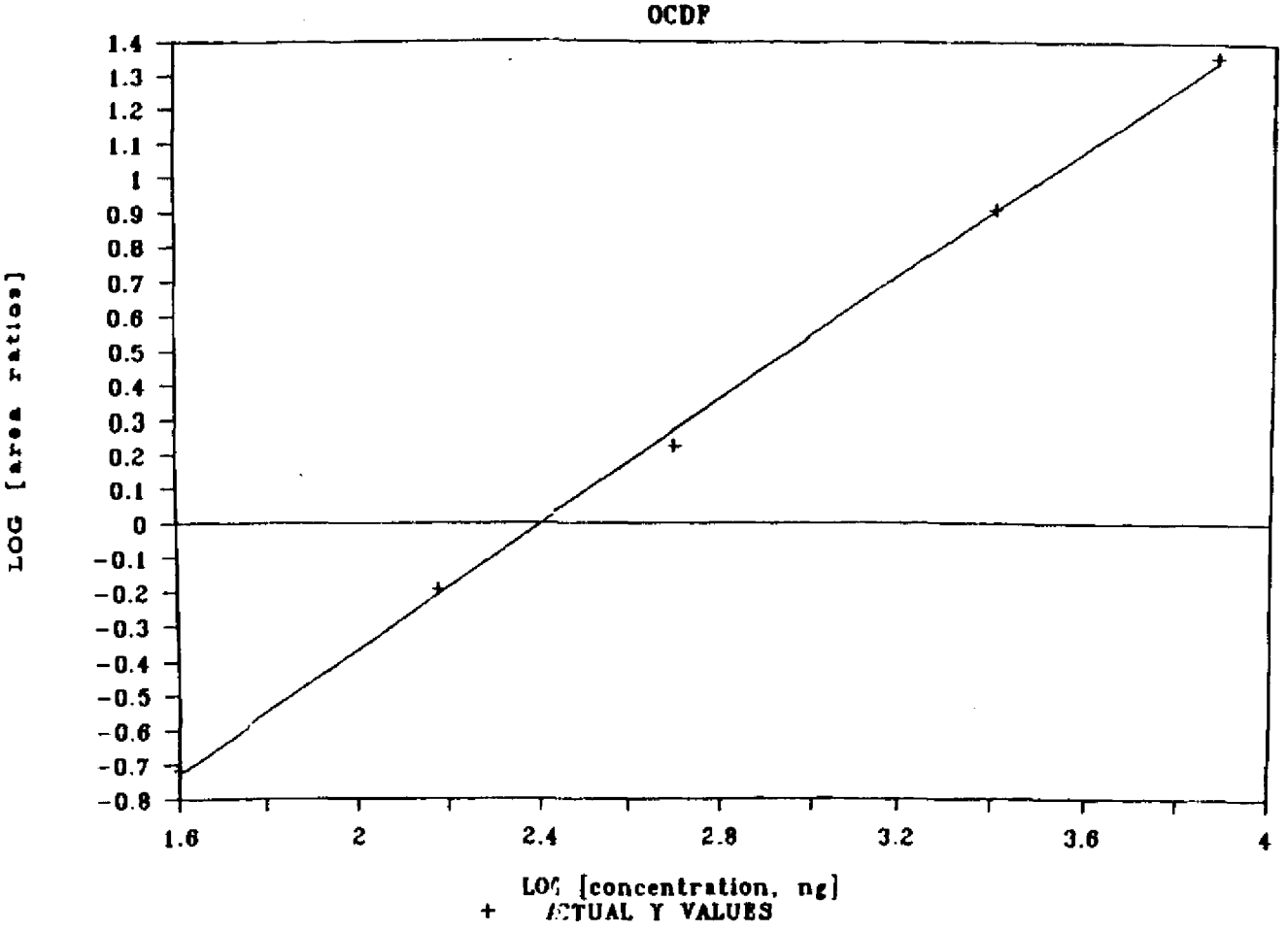


MONS 218219

CALCULATED ULTRA-2 CALIBRATION CURVE



CALCULATED ULTRA-2 CALIBRATION CURVE



MONS 218221

Appendix D

CALIBRATION DATA: DB-5

The following pages contain the second set of calibration curves and linear regression data generated on DB-5.

FIVE-POINT CALIBRATION IN TRIPLICATE (DB-5 60m J&W)

AREA RATIOS RELATIVE TO CORRESPONDING 13C ISOMER

	2378 TCDD	2378 TCDF	12378 PeCDF	23478 PeCDF	123478 HxCDF	234678 HxCDF	1234678 HpCDF	OCDF
40 ng								
VALUE	0.0650	0.0820	0.0960	0.0830	0.0840	0.0890	0.0520	0.1400
VALUE	0.0660	0.0880	0.0990	0.1030	0.0970	0.0770	0.0550	0.2620
VALUE	0.0820	0.0920	0.0930	0.1050	0.1030	0.0870	0.0670	0.1260
AVG.	0.0710	0.0873	0.0927	0.0970	0.0947	0.0843	0.0580	0.1760
S.D.	0.0095	0.0050	0.0052	0.0122	0.0097	0.0064	0.0079	0.0404
C.V.	13.4358	5.7632	5.4319	12.5418	10.2597	7.6234	13.6849	25.9279
150 ng								
VALUE	0.2770	0.3210	0.3430	0.3680	0.2670	0.2560	0.2240	0.5870
VALUE	0.2820	0.3270	0.3320	0.3590	0.2690	0.2470	0.2020	0.5560
VALUE	0.2850	0.3080	0.3130	0.3220	0.2940	0.2620	0.2040	0.5490
AVG.	0.2813	0.3187	0.3293	0.3497	0.2833	0.2570	0.2100	0.5640
S.D.	0.0040	0.0097	0.0152	0.0244	0.0139	0.0105	0.0122	0.0202
C.V.	1.4365	3.0479	4.6083	6.9721	4.9519	4.0995	5.7931	3.5858
500 ng								
VALUE	0.8690	1.0720	1.0810	1.1470	0.9560	0.9540	0.7190	1.9120
VALUE	0.8860	1.1000	1.1140	1.1520	0.9180	0.9010	0.6700	1.9680
VALUE	0.8880	1.0300	1.1020	1.1170	0.9600	0.8930	0.6630	1.9910
AVG.	0.8810	1.0673	1.0990	1.1397	0.9447	0.9160	0.6840	1.9570
S.D.	0.0104	0.0352	0.0167	0.0189	0.0232	0.0332	0.0305	0.0406
C.V.	1.1851	3.3010	1.5199	1.6624	2.4558	3.6191	4.4609	2.0763
2500 ng								
VALUE	4.3310	5.3610	5.5130	5.9060	4.7220	4.6450	3.5020	10.3000
VALUE	4.3210	5.5560	5.7910	6.1310	4.8680	4.7230	3.5400	10.8900
VALUE	4.2950	5.0560	5.3690	5.8740	4.7740	4.7770	3.5260	9.9590
AVG.	4.2857	5.2243	5.5577	5.9703	4.7880	4.7150	3.5220	10.4497
S.D.	0.0700	0.2520	0.2155	0.1401	0.0740	0.0664	0.0187	0.4723
C.V.	1.6342	4.7231	3.8776	2.3459	1.5455	1.4075	0.5303	4.5207
7500 ng								
VALUE	13.0300	16.2700	15.9300	17.7500	14.2900	14.4900	10.6500	29.6700
VALUE	13.4300	16.6000	16.7800	18.0600	14.2800	14.4800	10.6500	30.4300
VALUE	13.2000	16.2200	16.5200	17.6500	15.0900	14.9700	10.5900	31.1200
AVG.	13.2200	16.4300	16.4100	17.8200	14.5533	14.6467	10.6300	30.4967
S.D.	0.2007	0.1457	0.1472	0.2138	0.4648	0.2801	0.0346	0.7252
C.V.	1.5185	0.8867	0.8969	1.1996	3.1937	1.9121	0.3259	2.3800

MONS 218223

FIVE-POINT CALIBRATION IN TRIPLICATE (DB-5 60m JEW)

AVERAGE AREA RATIOS RELATIVE TO CORRESPONDING ISC ISOMER

NG	2378 TCDD	2378 TCDF	12378 PnCDF	23478 PnCDF	123478 HxCDF	234678 HxCDF	1234678 HpCDF	OCDF
40	0.7100	0.0872	0.0940	0.0970	0.0950	0.0840	0.0980	0.1560
150	0.2810	0.3190	0.3290	0.3300	0.2830	0.2570	0.2100	0.5640
500	0.8810	1.0700	1.1000	1.1400	0.9450	0.9160	0.6840	1.9600
2500	4.2900	5.3200	5.5500	5.9700	4.7900	4.7200	3.5200	10.6500
7500	13.2200	16.4300	16.6100	17.8200	14.5500	14.6500	10.6300	30.4100

LOG (ng)	LOG (average area ratio)							
1.602	-1.1487	-1.0595	-1.0269	-1.0132	-1.0223	-1.0757	-1.2366	-0.8069
2.176	-0.5513	-0.4962	-0.4828	-0.4559	-0.5482	-0.5901	-0.6778	-0.2487
2.699	-0.0550	0.0294	0.0414	0.0569	-0.0246	-0.0381	-0.1649	0.2923
3.398	0.6325	0.7259	0.7443	0.7760	0.6803	0.6739	0.5465	1.0191
3.875	1.1212	1.2156	1.2204	1.2509	1.1629	1.1658	1.0265	1.4830

Y-ESTIMATED

-1.1393	-1.0656	-1.0396	-1.0239	-1.0650	-1.1168	-1.2459	-0.8166
-0.5698	-0.4912	-0.4701	-0.4505	-0.5076	-0.5448	-0.6735	-0.2344
-0.0511	0.0321	0.0486	0.0719	0.0001	-0.0238	-0.1521	0.2959
0.6424	0.7215	0.7421	0.7701	0.6788	0.6727	0.5449	1.0048
1.1157	1.2090	1.2154	1.2468	1.1421	1.1482	1.0267	1.4887

MONS 218224

FIVE-POINT CALIBRATION IN TRIPLICATE (DB-5 60m J&W)

TCDD

Regression Output:

Constant	-2.7287
Std Err of Y Est	0.0139
R Squared	0.9998
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.9921
Std Err of Coef.	0.0076

TCDF

Regression Output:

Constant	-2.6608
Std Err of Y Est	0.0070
R Squared	1.0000
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	1.0007
Std Err of Coef.	0.0038

12378-PnCDF

Regression Output:

Constant	-2.6290
Std Err of Y Est	0.0116
R Squared	0.9999
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.9921
Std Err of Coef.	0.0064

23478-PnCDF

Regression Output:

Constant	-2.6244
Std Err of Y Est	0.0118
R Squared	0.9999
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.9990
Std Err of Coef.	0.0065

MONS 218225

FIVE-POINT CALIBRATION IN TRIPLICATE [DB-5 60m J&W]

123478-HxCDF
Regression Output:

Constant	-2.6206
Std Err of Y Est	0.0388
R Squared	0.9986
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.9710
Std Err of Coef.	0.0212

234678-HxCDF
Regression Output:

Constant	-2.7133
Std Err of Y Est	0.0377
R Squared	0.9987
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.9965
Std Err of Coef.	0.0206

1234678-HpCDF
Regression Output:

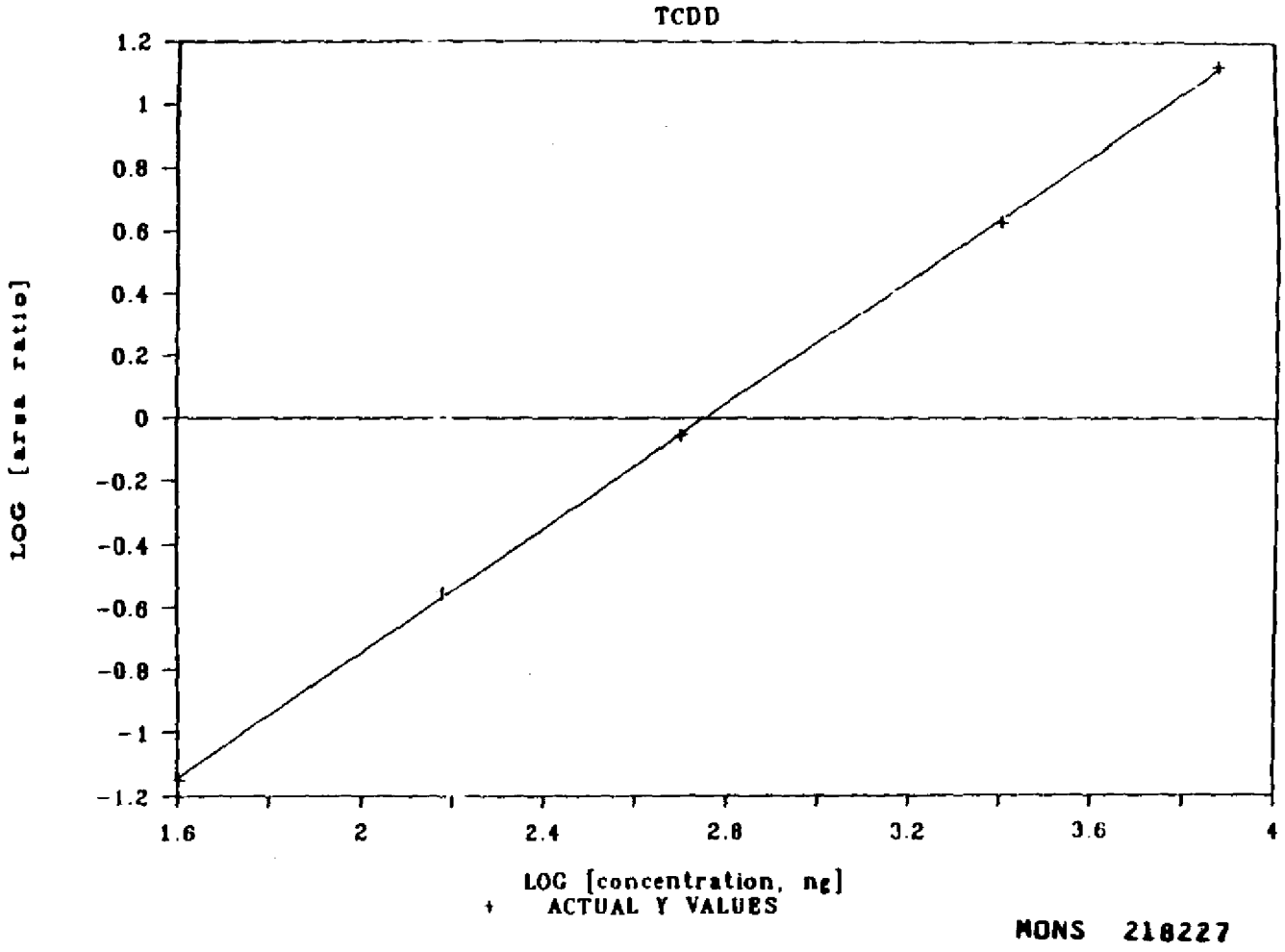
Constant	-2.8435
Std Err of Y Est	0.0101
R Squared	0.9999
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.9972
Std Err of Coef.	0.0055

OCDF
Regression Output:

Constant	-2.4414
Std Err of Y Est	0.0135
R Squared	0.9998
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	1.0142
Std Err of Coef.	0.0074

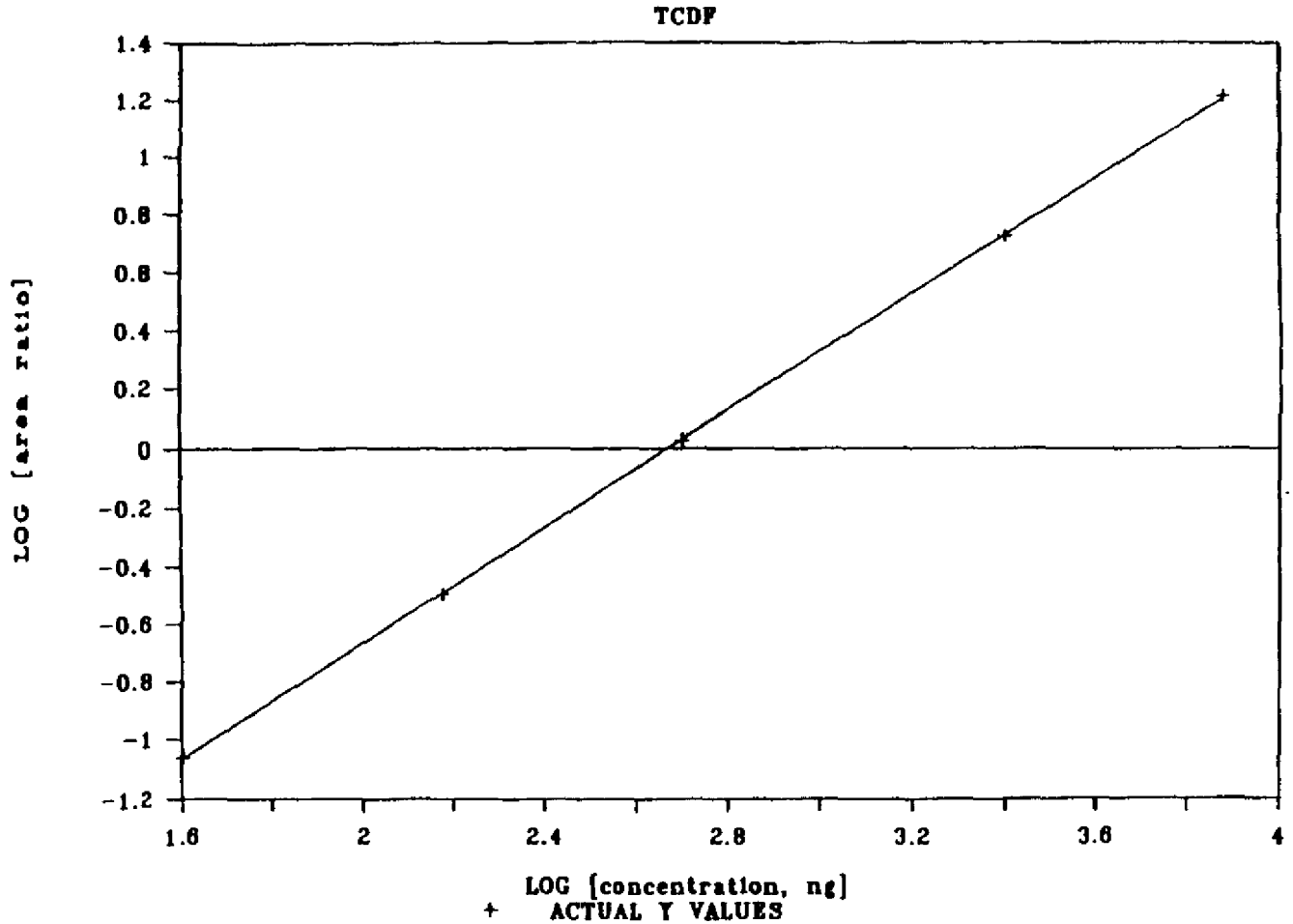
MONS 218226

CALCULATED DB-5 CALIBRATION CURVE



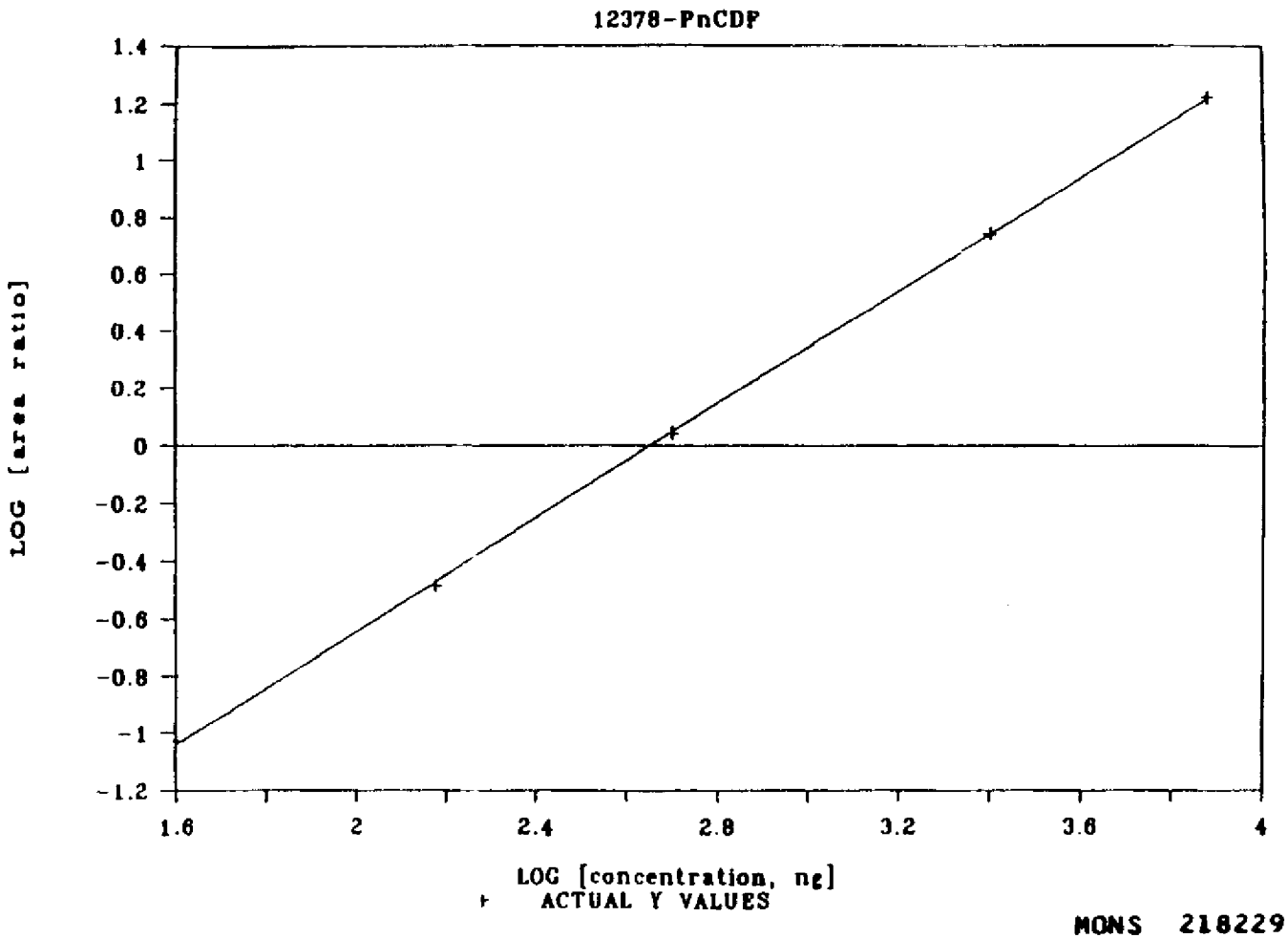
MONS 218227

CALCULATED DB-5 CALIBRATION CURVE

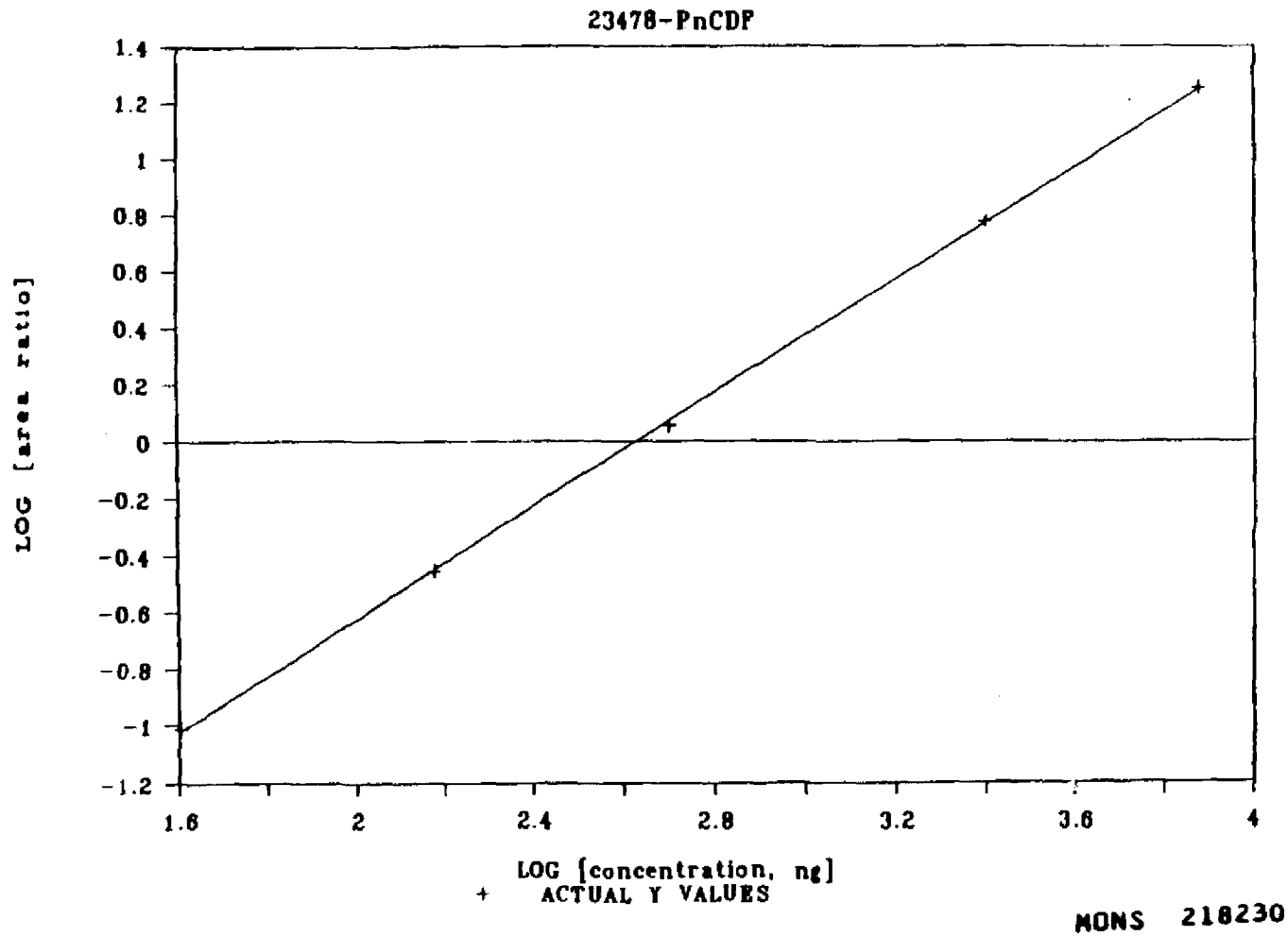


MONS 218228

CALCULATED DB-5 CALIBRATION CURVE

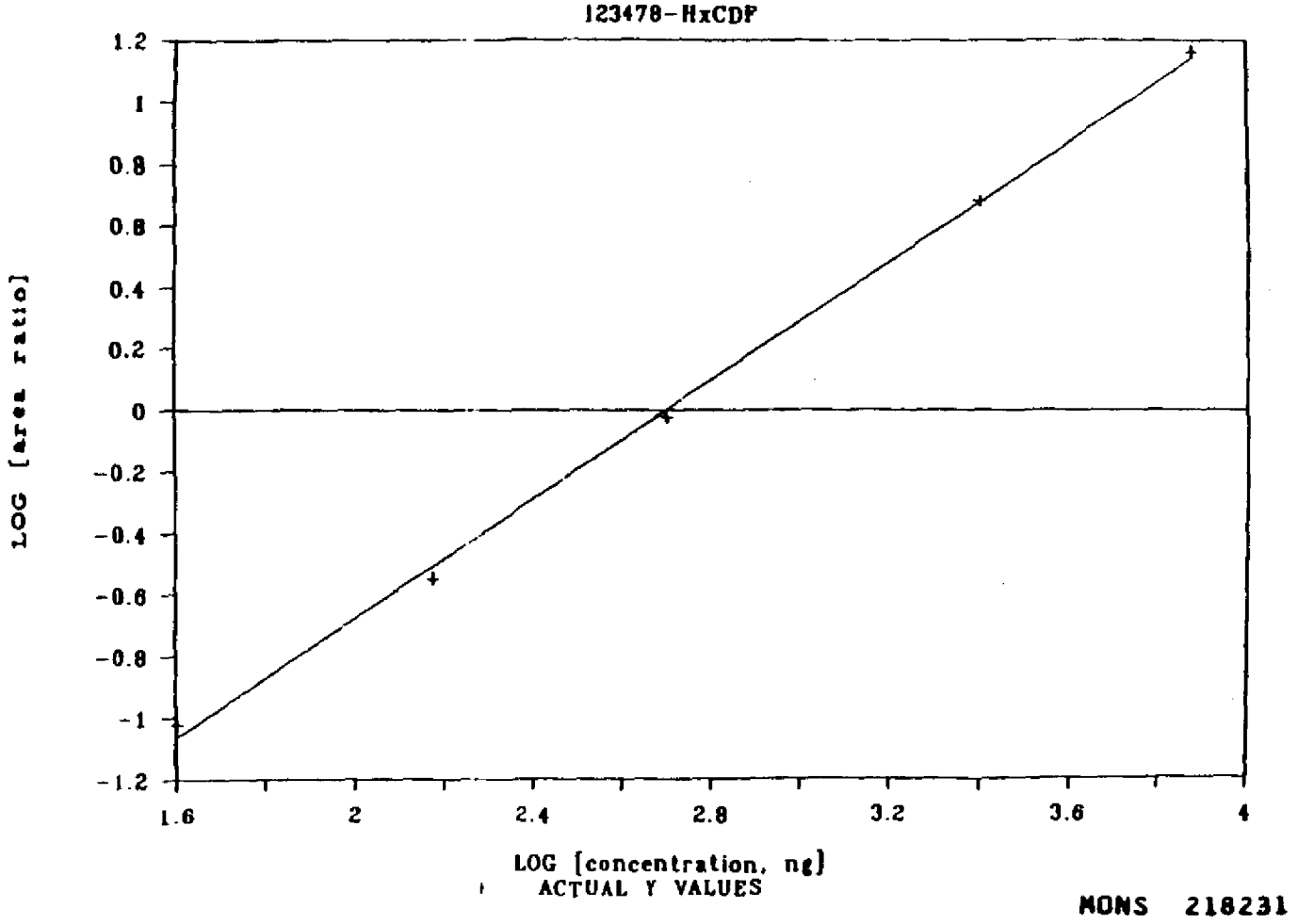


CALCULATED DB-5 CALIBRATION CURVE



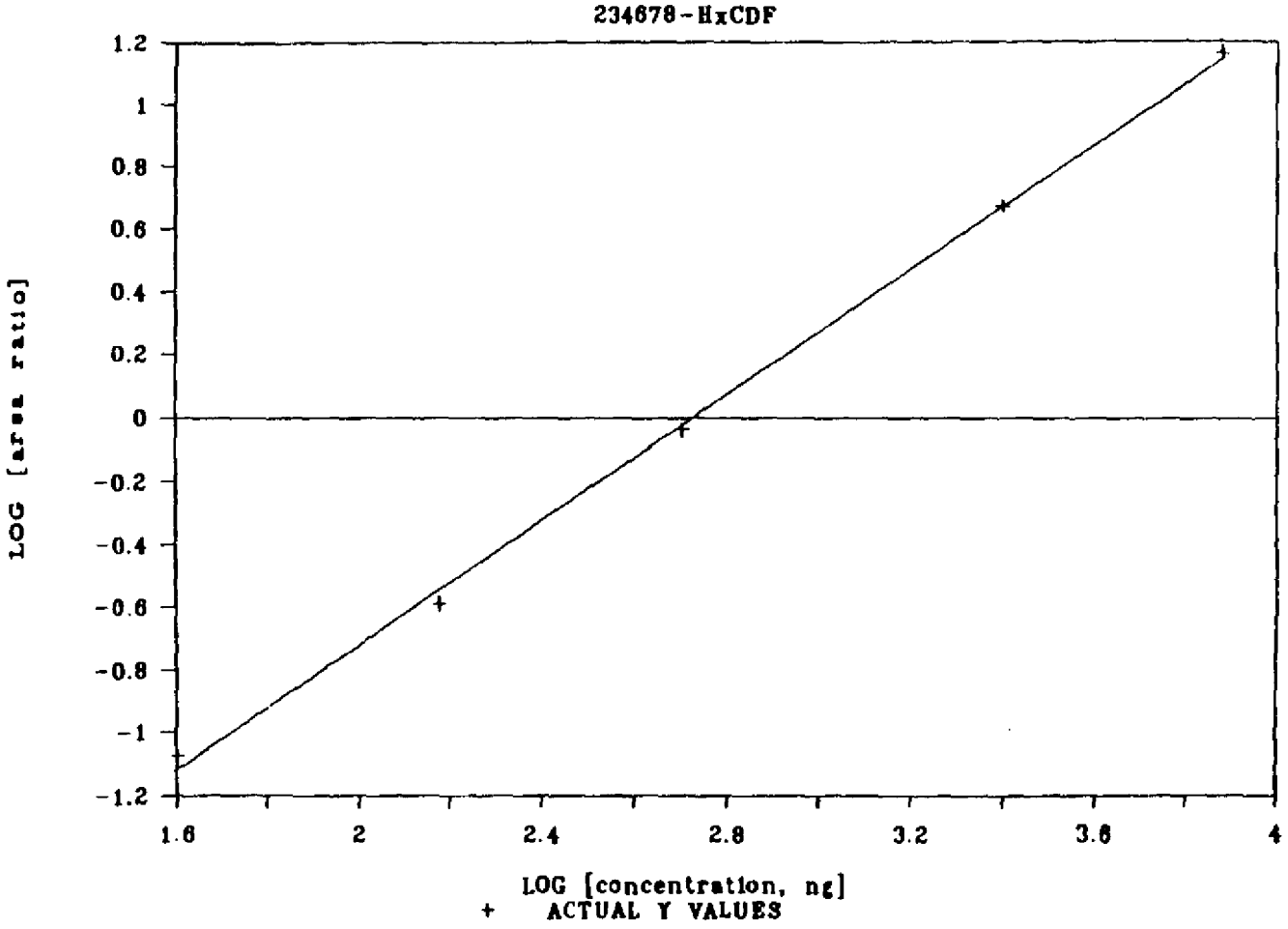
MONS 218230

CALCULATED DB-5 CALIBRATION CURVE

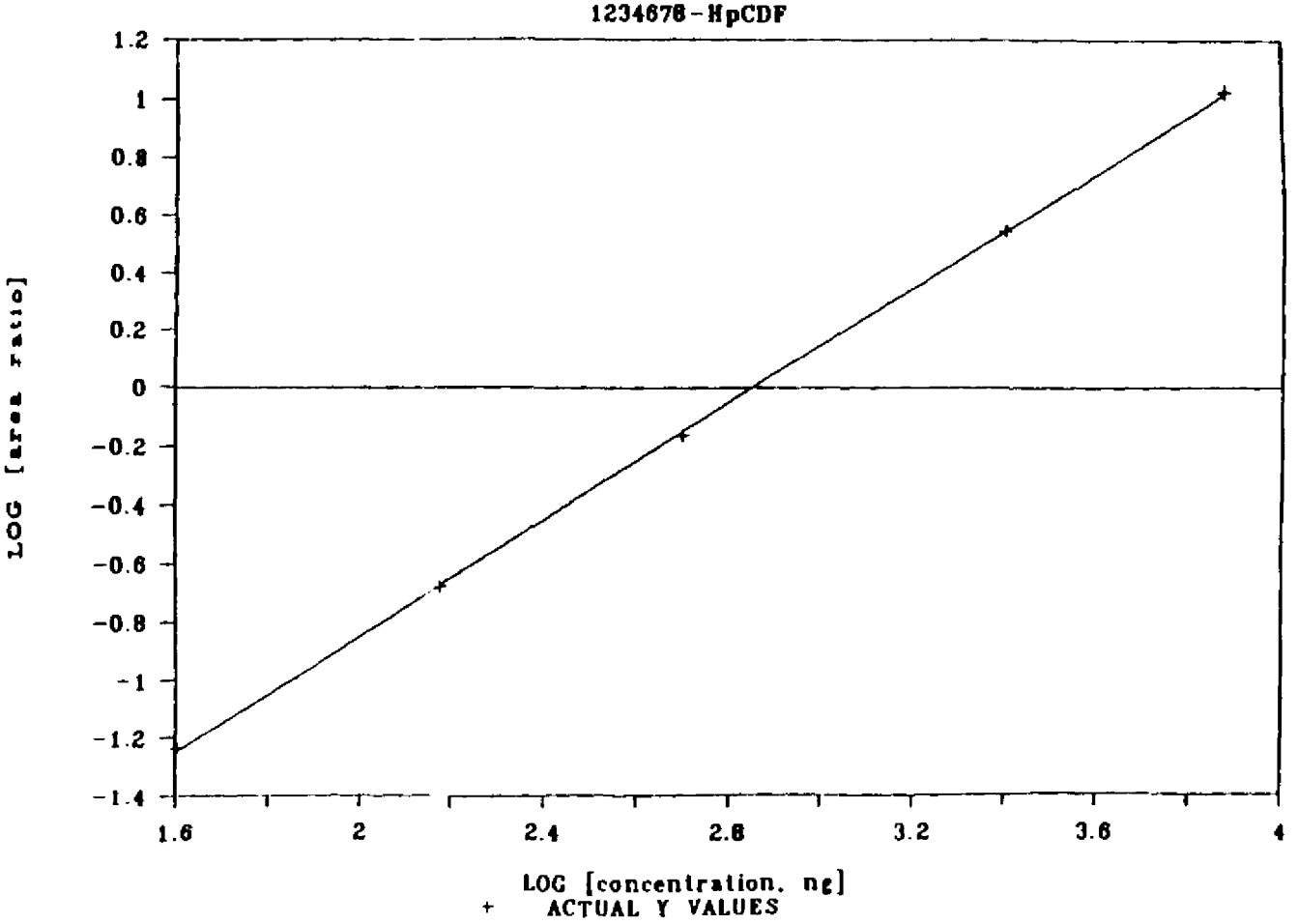


MONS 218231

CALCULATED DB-5 CALIBRATION CURVE

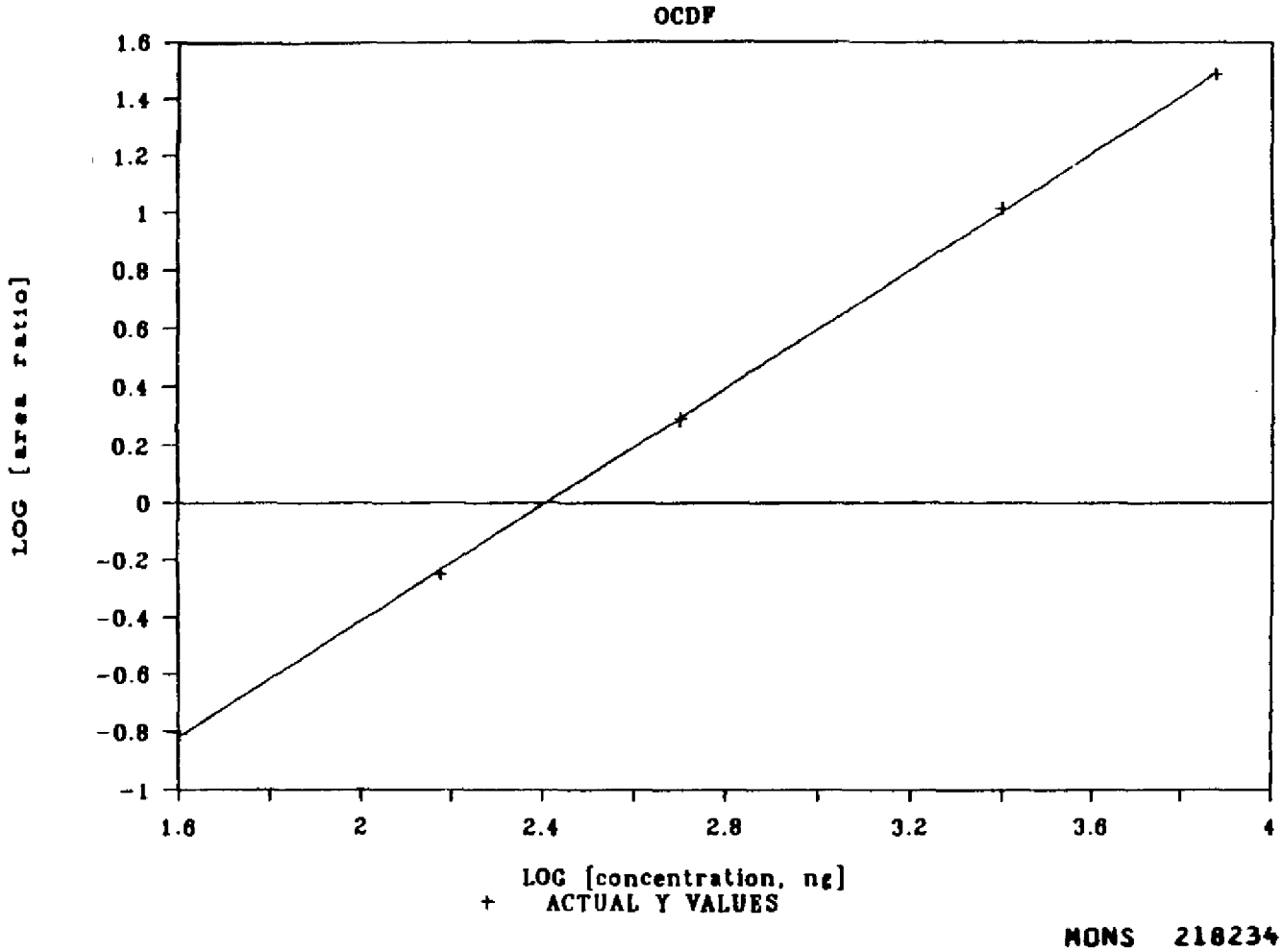


CALCULATED DB-5 CALIBRATION CURVE



MONS 218233

CALCULATED DB-5 CALIBRATION CURVE



Appendix E

CALIBRATION DATA: DB-5

The following pages contain the third set of calibration curves and linear regression data generated on DB-5.

MONS 218235

FIVE-POINT CALIBRATION IN TRIPLICATE (DB-5 60m J&W)

AREA RATIOS RELATIVE TO CORRESPONDING 13C ISOMER

	2378 TCDD	2378 TCDF	12378 PnCDF	23478 PnCDF	123478 HxCDF	234678 HxCDF	1234678 HpCDF	OCDF
40 ng								
VALUE	0.0650	0.0930	0.1060	0.1100	0.1050	0.0980	0.0820	0.2230
VALUE	0.0620	0.0860	0.0950	0.1000	0.0880	0.0880	0.0870	0.1850
VALUE	0.0730	0.0930	0.1090	0.1070	0.0980	0.1010	0.0670	0.2000
AVG.	0.0667	0.0907	0.1033	0.1057	0.0970	0.0957	0.0787	0.2027
S.D.	0.0057	0.0040	0.0074	0.0051	0.0085	0.0068	0.0104	0.0191
C.V.	8.5294	4.4575	7.1333	4.8564	8.8083	7.1152	13.2309	9.4440
150 ng								
VALUE	0.2500	0.3290	0.3660	0.3610	0.3550	0.3270	0.2150	0.5400
VALUE	0.2660	0.3370	0.3470	0.3700	0.3450	0.3330	0.2520	0.6590
VALUE	0.2540	0.3370	0.3390	0.3820	0.3380	0.3160	0.2430	0.6460
AVG.	0.2567	0.3343	0.3507	0.3710	0.3427	0.3253	0.2367	0.6150
S.D.	0.0083	0.0046	0.0139	0.0105	0.0137	0.0086	0.0193	0.0653
C.V.	3.2442	1.3815	3.9549	2.8398	3.9836	2.6501	8.1532	10.6149
500 ng								
VALUE	0.9110	1.1380	1.1720	1.2490	1.1440	1.0890	0.8440	2.4080
VALUE	0.9130	1.1130	1.1720	1.2380	1.1430	1.1100	0.8570	2.1860
VALUE	0.8770	1.1210	1.1810	1.2460	1.0750	1.0530	0.7710	2.0450
AVG.	0.9003	1.1240	1.1750	1.2443	1.1207	1.0840	0.8240	2.2130
S.D.	0.0202	0.0129	0.0052	0.0057	0.0396	0.0288	0.0464	0.1830
C.V.	2.2472	1.1559	0.4422	0.4570	3.5293	2.6593	5.6259	8.2593
2500 ng								
VALUE	4.5290	5.5660	5.9470	6.1770	5.5160	5.2190	4.1690	10.8950
VALUE	4.6940	5.6140	6.0360	6.3490	5.5430	5.4410	4.1410	11.2270
VALUE	4.4840	5.6270	5.8960	6.2770	5.3370	5.2500	3.9300	10.7550
AVG.	4.5690	5.6023	5.9597	6.2677	5.4653	5.3033	4.0800	10.9590
S.D.	0.1106	0.0321	0.0709	0.0864	0.1120	0.1202	0.1307	0.2424
C.V.	2.4199	0.5735	1.1889	1.3782	2.0485	2.2670	3.2024	2.2121
7500 ng								
VALUE	13.9630	17.0440	17.4870	18.6230	17.1560	17.1570	12.8720	30.6290
VALUE	14.0720	17.0410	18.3480	19.2450	17.3660	17.6670	13.4960	31.2780
VALUE	13.7280	17.2600	17.6070	18.5600	15.6260	15.6260	11.1540	31.1810
AVG.	13.9210	17.1150	17.9473	18.8093	16.7193	16.8167	12.5073	31.0293
S.D.	0.1758	0.1256	0.3223	0.3786	0.9441	1.0622	1.2129	0.3501
C.V.	1.2629	0.7338	1.7960	2.0129	5.6465	6.7164	9.6970	1.1282

MONS 218236

FIVE-POINT CALIBRATION IN TRIPLICATE (DB-5 60m JEW)

AVERAGE AREA RATIOS RELATIVE TO CORRESPONDING 13C ISOMER

NG	2378 TCDD	2378 TCDF	12378 PeCDF	23478 PeCDF	123478 HxCDF	234678 HxCDF	1234678 HpCDF	OCDF
40	0.0667	0.0907	0.1033	0.1057	0.0970	0.0957	0.0787	0.2027
150	0.2567	0.3343	0.3507	0.3710	0.3427	0.3253	0.2367	0.6150
500	0.9003	1.1240	1.1750	1.2443	1.1207	1.0840	0.8240	2.2130
2500	4.5690	5.6023	5.9597	6.2677	5.4653	5.3033	4.0800	10.9590
7500	13.9210	17.1150	17.9473	18.9093	16.7193	16.0167	12.5073	31.0293

.....

LOG (ng)	LOG (average area ratio)							
1.602	-1.1759	-1.0424	-0.9859	-0.9759	-1.0132	-1.0191	-1.1040	-0.6931
2.176	-0.5906	-0.4759	-0.4551	-0.4306	-0.4651	-0.4877	-0.6258	-0.2111
2.699	-0.0456	0.0508	0.0700	0.0949	0.0495	0.0350	-0.0841	0.3450
3.398	0.6598	0.7484	0.7752	0.7971	0.7376	0.7245	0.6107	1.0398
3.875	1.1437	1.2334	1.2540	1.2744	1.2232	1.2257	1.0972	1.4918

Y-ESTIMATES

-1.1737	-1.0466	-1.0051	-0.9883	-1.0230	-1.0390	-1.1435	-0.7245
-0.5876	-0.4718	-0.4368	-0.4182	-0.4582	-0.4717	-0.5822	-0.1649
-0.0538	0.0518	0.0810	0.1011	0.0562	0.0431	-0.0710	0.3448
0.6599	0.7517	0.7770	0.7953	0.7459	0.7259	0.6124	1.0261
1.1470	1.2294	1.2454	1.2692	1.2134	1.2074	1.0788	1.4912

MONS 218237

FIVE-POINT CALIBRATION IN TRIPLICATE (DB-5 60m J&W)

TCDD

Regression Output:

Constant	-2.8094
Std Err of Y Est	0.0055
R Squared	1.0000
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	1.0210
Std Err of Coef.	0.0030

TCDF

Regression Output:

Constant	-2.6507
Std Err of Y Est	0.0045
R Squared	1.0000
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	1.0013
Std Err of Coef.	0.0025

12378-PeCDF

Regression Output:

Constant	-3.5913
Std Err of Y Est	0.0173
R Squared	0.9997
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.9901
Std Err of Coef.	0.0095

23478-PeCDF

Regression Output:

Constant	-3.5795
Std Err of Y Est	0.0112
R Squared	0.9999
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.9927
Std Err of Coef.	0.0061

MONS 218238

FIVE-POINT CALIBRATION IN TRIPLICATE [DB-5 60m JWH]

123478-HxCDF
Regression Output:

Constant	-2.5993
Std Err of Y Est	0.0104
R Squared	0.9999
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.9839
Std Err of Coef.	0.0057

234678-HxCDF
Regression Output:

Constant	-2.6223
Std Err of Y Est	0.0202
R Squared	0.9996
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.9883
Std Err of Coef.	0.0110

1234678-HpCDF
Regression Output:

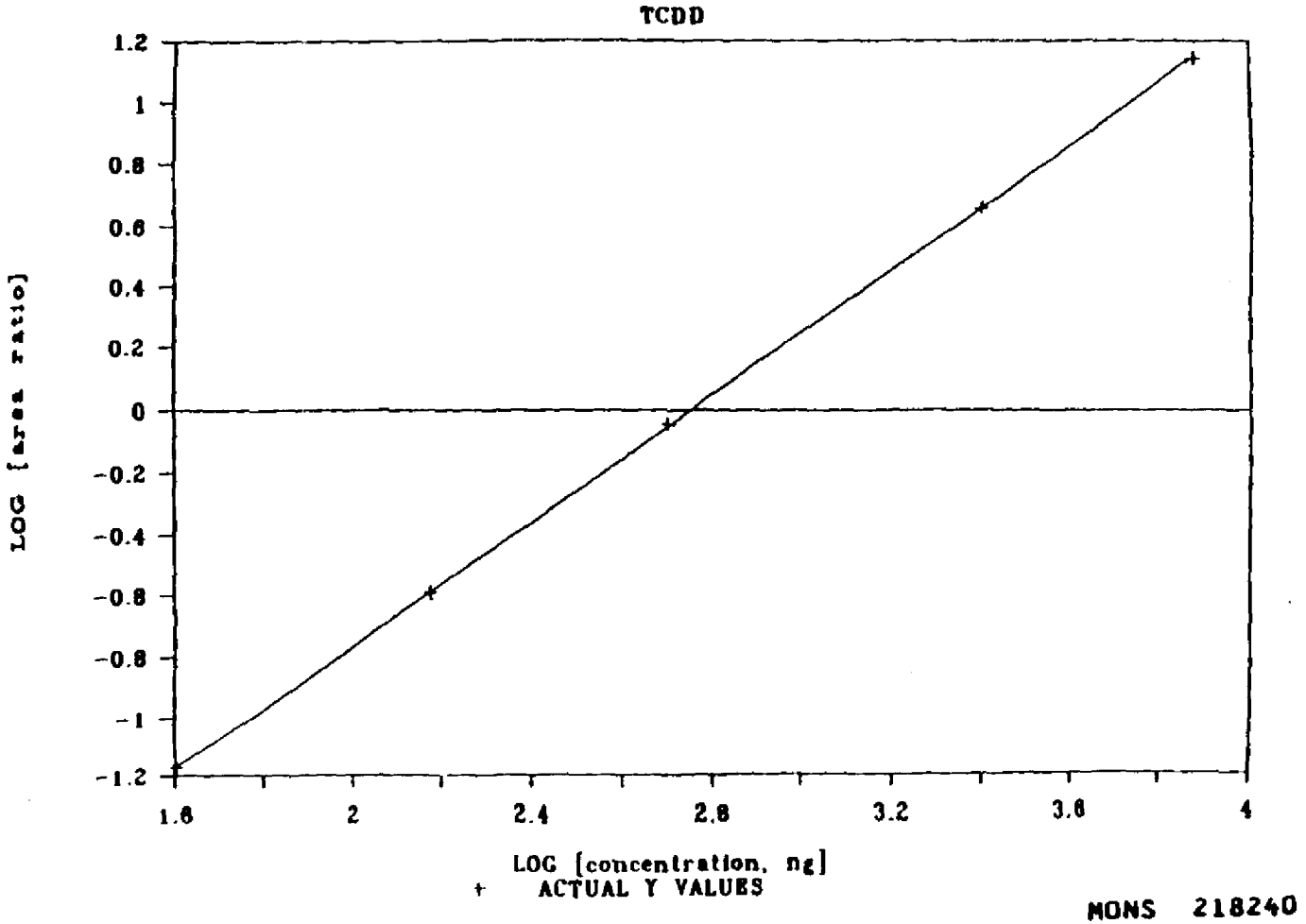
Constant	-2.7098
Std Err of Y Est	0.0363
R Squared	0.9998
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.9777
Std Err of Coef.	0.0199

OCDF
Regression Output:

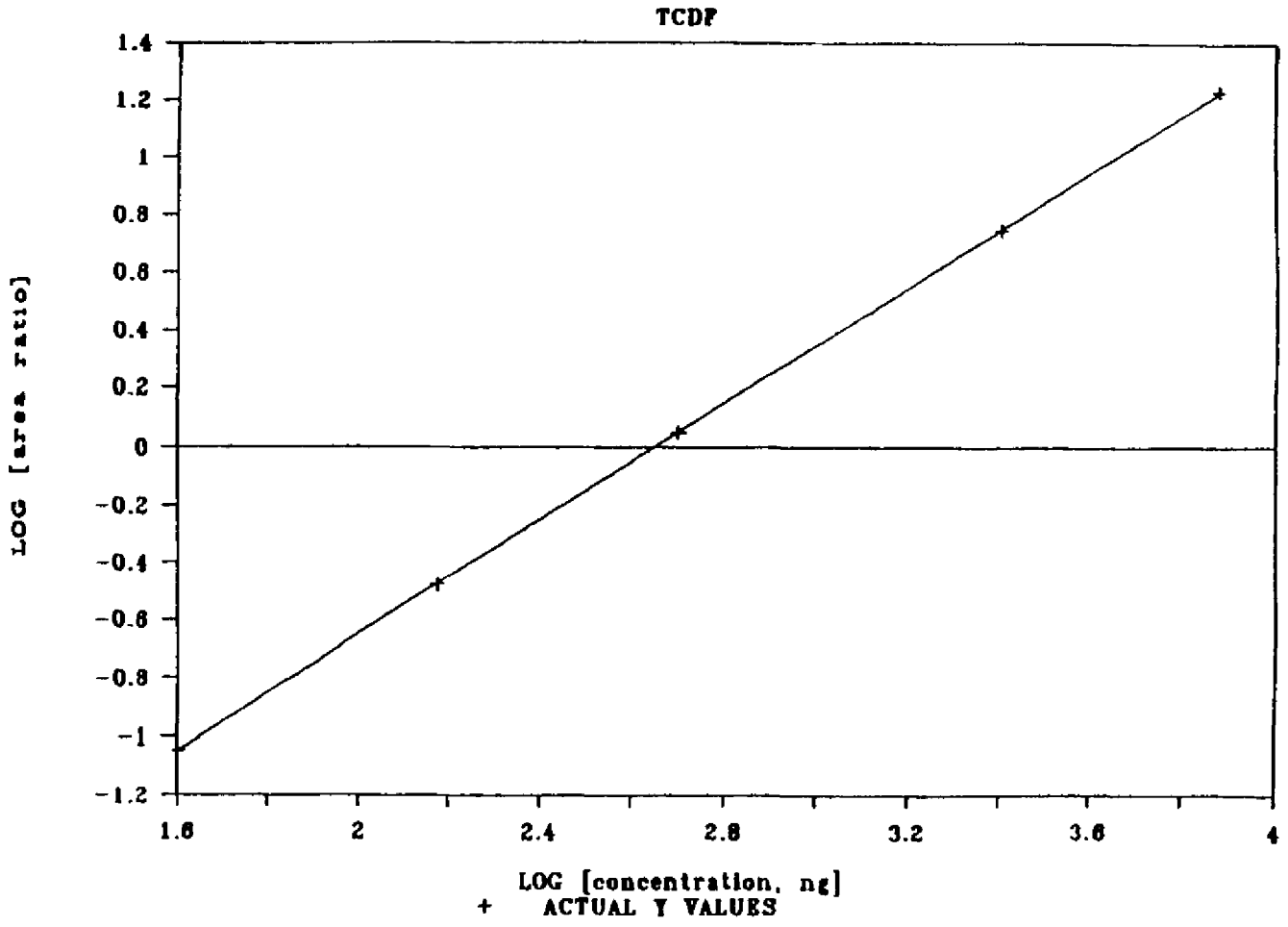
Constant	-2.2862
Std Err of Y Est	0.0552
R Squared	0.9990
No. of Observations	5.0000
Degrees of Freedom	3.0000
X Coefficient(s)	0.9748
Std Err of Coef.	0.0302

MONS 218239

CALCULATED DB-5 CALIBRATION CURVE

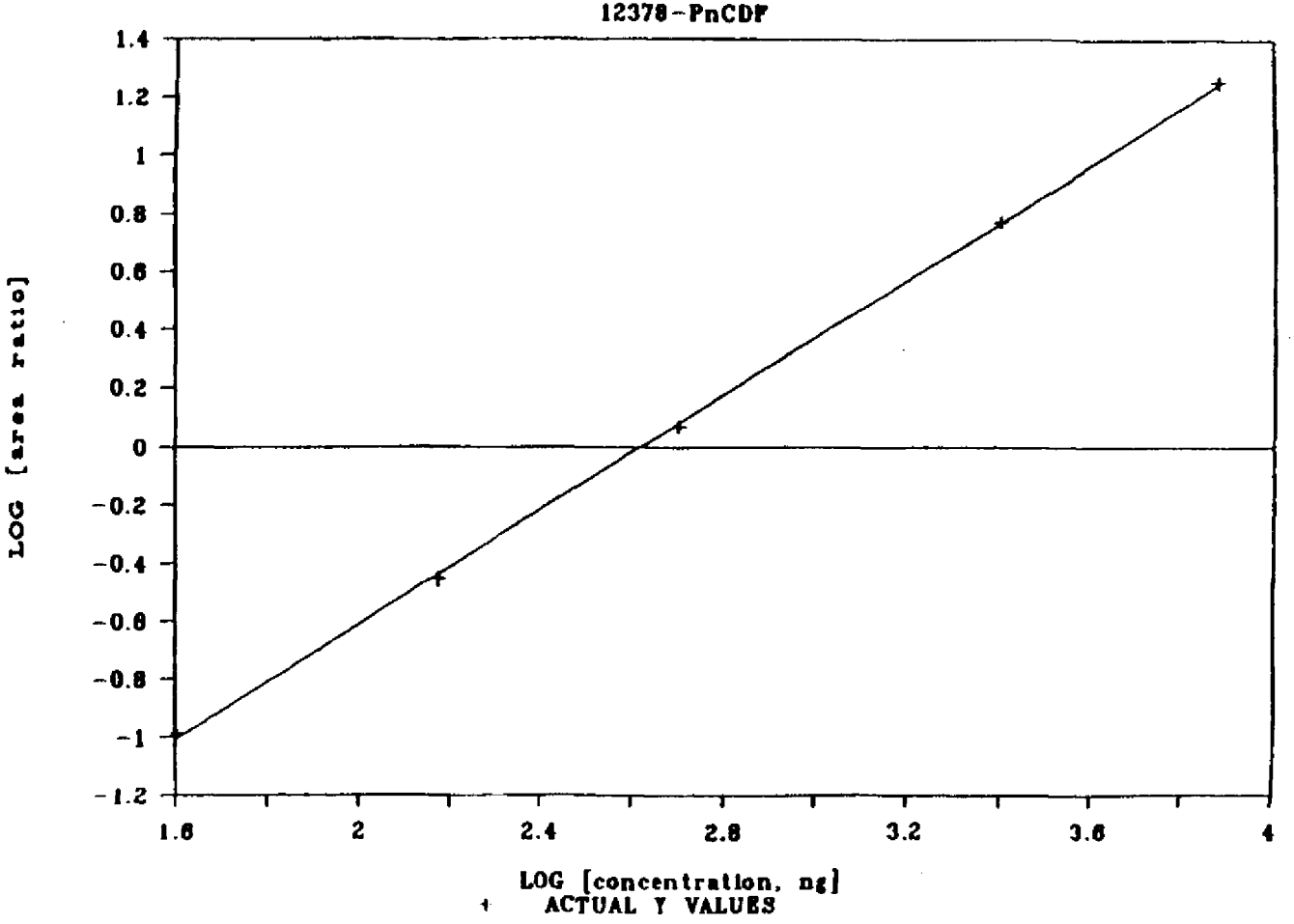


CALCULATED DB-5 CALIBRATION CURVE



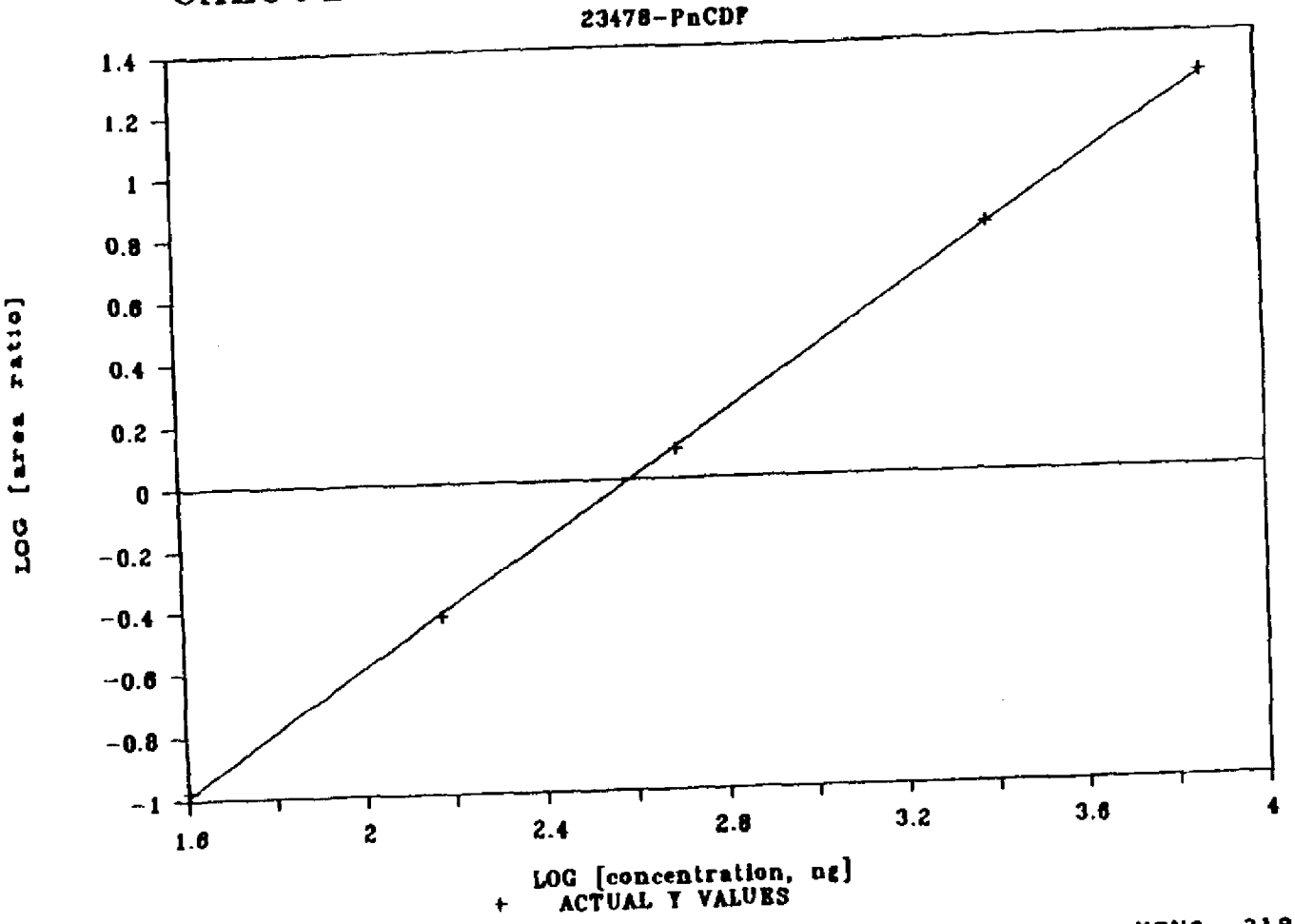
NONS 218241

CALCULATED DB-5 CALIBRATION CURVE



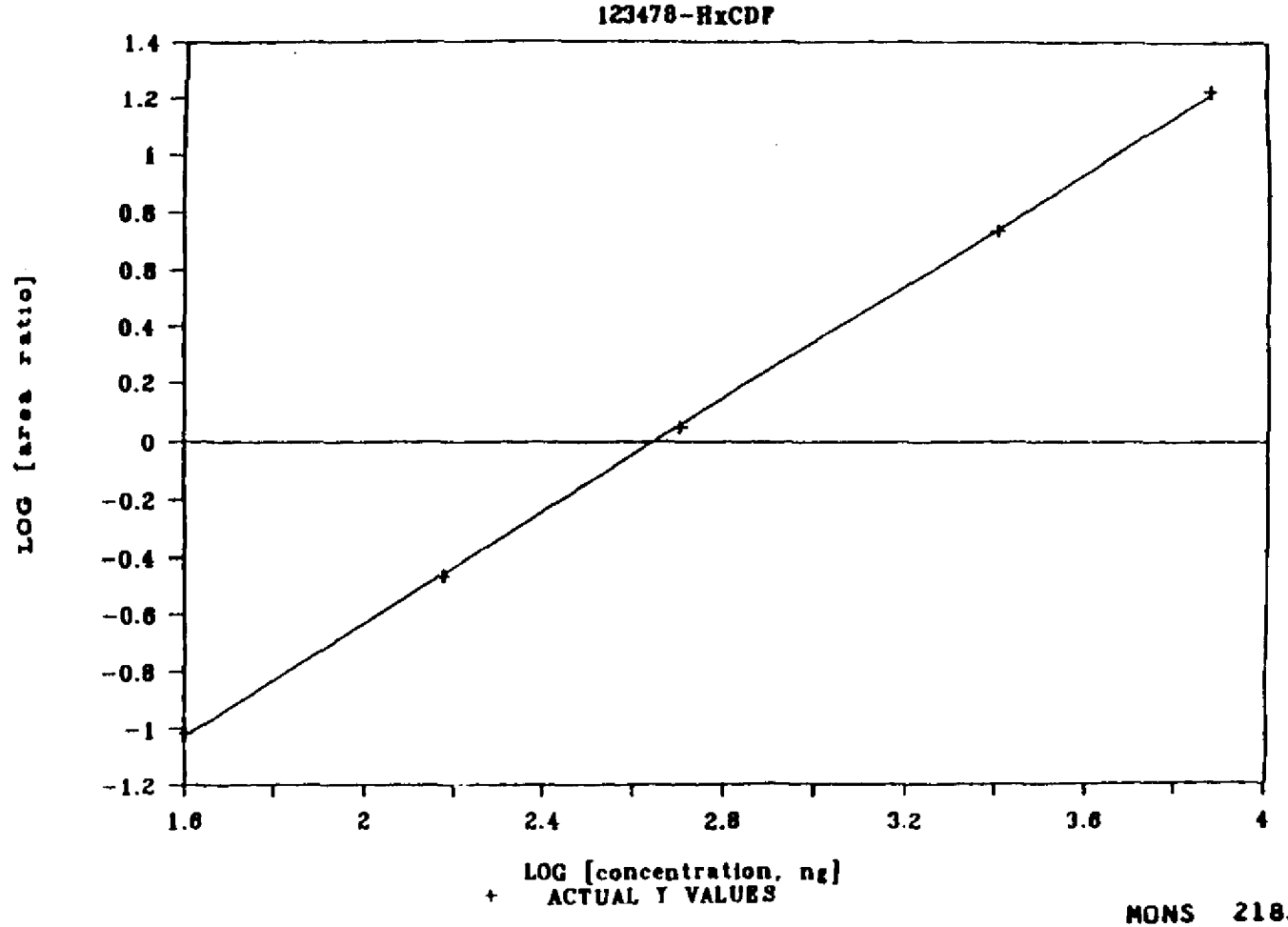
MONS 218242

CALCULATED DB-5 CALIBRATION CURVE



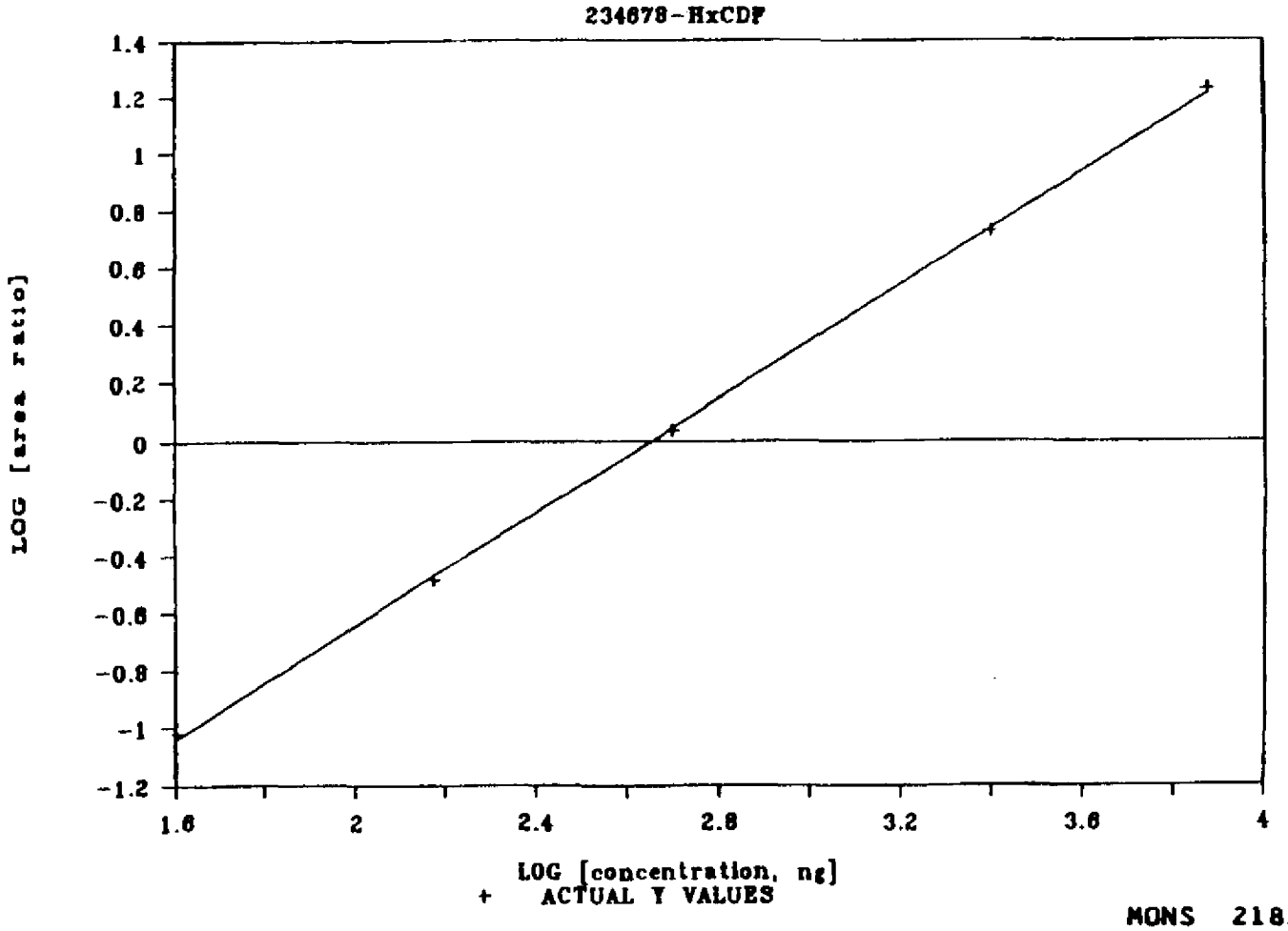
MONS 218243

CALCULATED DB-5 CALIBRATION CURVE



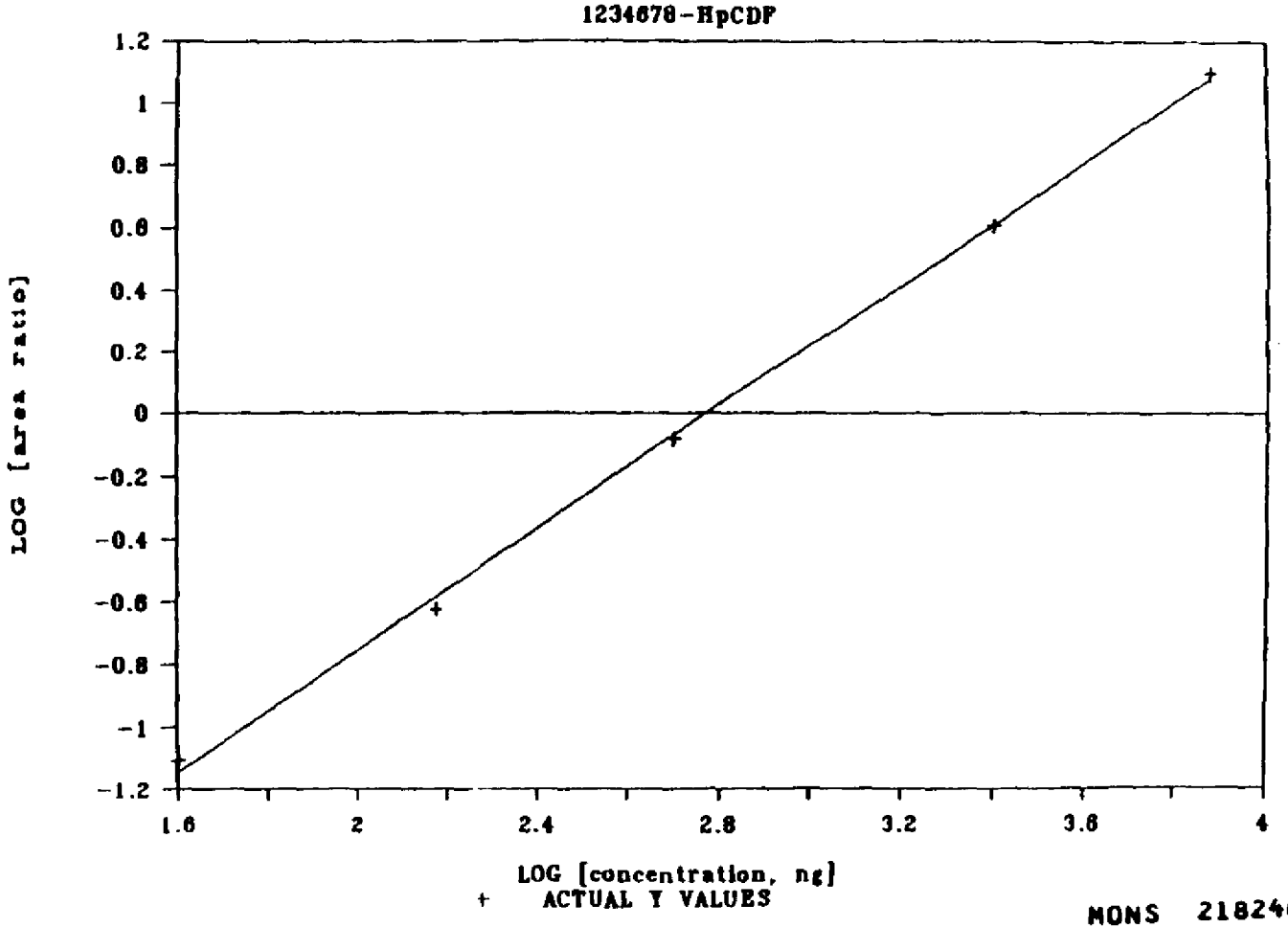
MONS 218244

CALCULATED DB-5 CALIBRATION CURVE



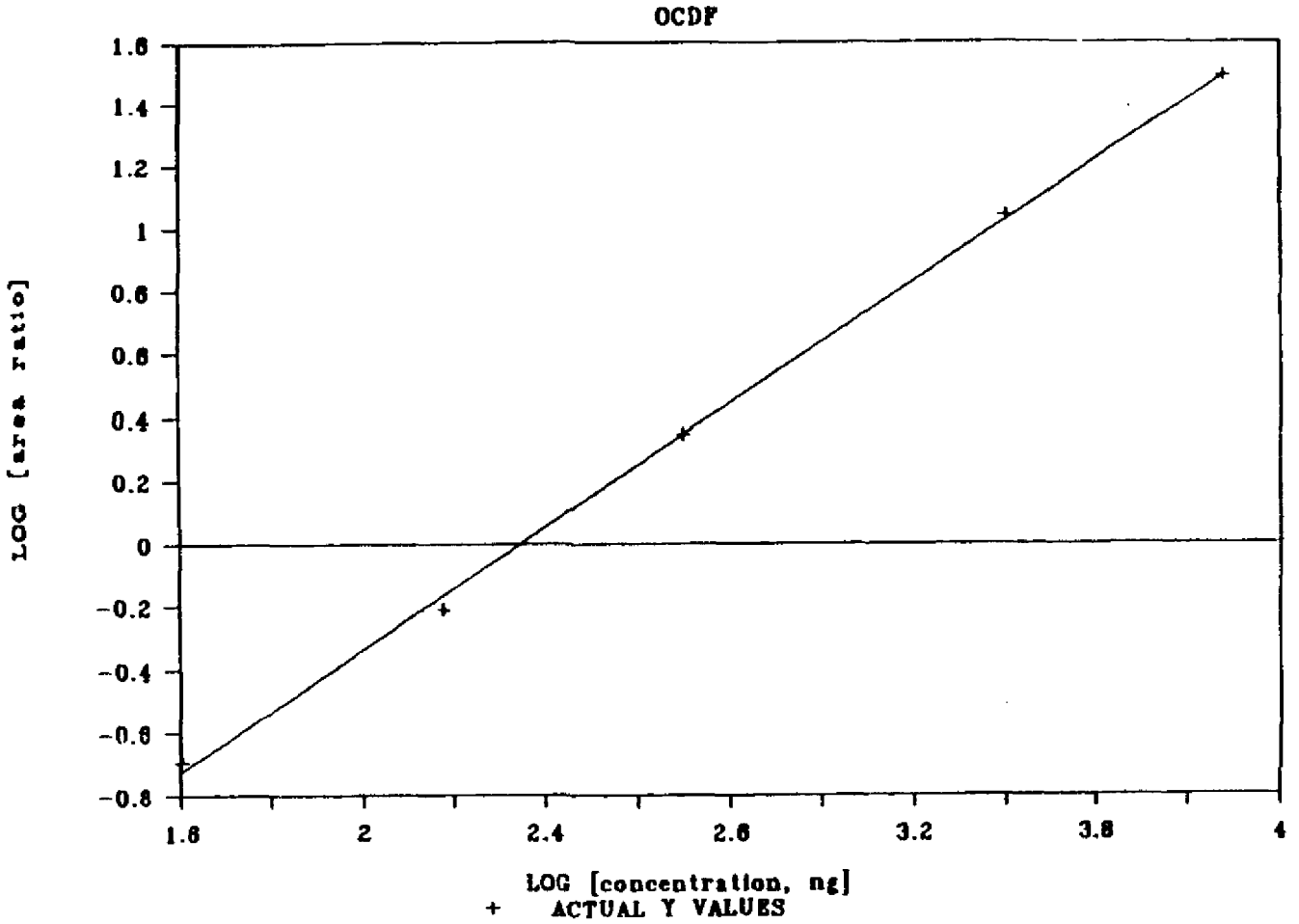
MONS 218245

CALCULATED DB-5 CALIBRATION CURVE



MONS 218246

CALCULATED DB-5 CALIBRATION CURVE



MONS 218247

Appendix F

CALIBRATION DATA: SP-2331

The following pages contain the fourth set of calibration curves and linear regression data generated on SP-2331.

FOUR-POINT CALIBRATION IN TRIPLICATE (SP-2331 5061

AREA RATIOS RELATIVE TO CORRESPONDING 13C ISOMER

	2378 TCDD	2378 TCDF	12378 PnCDF	23478 PnCDF	123478 HxCDF	234678 HxCDF	1234678 HpCDF	OCDF
40 ng								
VALUE	0.0857	0.0953	0.1064	0.0963	0.0911	0.0973	0.0710	NA
VALUE	0.0811	0.0960	0.1015	0.1055	0.0938	0.0918	0.0689	NA
VALUE	0.0789	0.0955	0.0968	0.1050	0.0840	0.0916	0.0600	NA
AVG.	0.0819	0.0956	0.1016	0.1023	0.0896	0.0936	0.0666	NA
S.D.	0.0035	0.0004	0.0048	0.0052	0.0051	0.0032	0.0058	NA
C.V.	4.2367	0.3771	4.7263	5.0587	5.6474	3.4571	8.7641	NA
150 ng								
VALUE	0.2850	0.3350	0.3770	0.3060	0.3390	0.3440	0.2560	0.2600
VALUE	0.3010	0.3380	0.3670	0.3870	0.3410	0.3370	0.2510	0.2100
VALUE	0.2900	0.3410	0.3750	0.3960	0.3320	0.3480	0.2520	0.1860
AVG.	0.2920	0.3380	0.3730	0.3630	0.3373	0.3430	0.2530	0.2190
S.D.	0.0082	0.0030	0.0053	0.0496	0.0047	0.0056	0.0036	0.0376
C.V.	2.8032	0.8876	1.4186	13.6551	1.4009	1.6233	1.0458	17.1886
500 ng								
VALUE	1.0110	1.1380	1.2230	1.3630	1.1060	1.1120	0.8140	0.7420
VALUE	0.9850	1.1500	1.2450	1.3510	1.1100	1.1340	0.9020	0.7160
VALUE	0.9840	1.1340	1.2680	1.3700	1.1500	1.0960	1.2115	0.6400
AVG.	0.9933	1.1407	1.2453	1.3613	1.0887	1.1140	0.9425	0.6993
S.D.	0.0153	0.0083	0.0225	0.0095	0.0335	0.0191	0.0515	0.0530
C.V.	1.5411	0.7300	1.8069	0.7059	3.0814	1.7126	6.1141	7.5791
2500 ng								
VALUE	5.0160	5.6810	6.4280	6.7360	5.3520	5.4880	4.1060	3.4160
VALUE	4.9950	5.6360	6.2840	6.7060	5.3680	5.6870	4.1770	3.0890
VALUE	4.9650	5.5450	6.3190	6.8320	5.5930	5.6630	4.3010	3.4420
AVG.	4.9920	5.6207	6.3437	6.7580	5.4377	5.6127	4.1947	3.3157
S.D.	0.0256	0.0693	0.0751	0.0658	0.1348	0.1086	0.0987	0.1967
C.V.	0.5175	1.2327	1.1839	0.9739	2.4783	1.9354	2.3529	5.9333

MONS 218249

FOUR-POINT CALIBRATION IN TRIPLICATE (SP-2331 50m)

AVERAGE AREA RATIOS RELATIVE TO CORRESPONDING 13C ISOMER

NG	2378 TCDD	2378 TCDF	12378 PnCDF	23478 PnCDF	123478 HxCDF	234678 HxCDF	1234678 HpCDF	OCDF
40	0.0819	0.0956	0.1016	0.1023	0.0896	0.0936	0.0666	NA
150	0.2920	0.3380	0.3730	0.3630	0.3373	0.3430	0.2530	0.2190
500	0.9933	1.1407	1.2453	1.2613	1.0887	1.1146	0.8425	0.6993
2500	4.9920	5.6207	6.2437	6.2580	5.4377	5.6127	4.1947	3.3157

LOG (ng) LOG (average area ratio)

1.602	-1.0867	-1.0195	-0.9931	-0.9901	-1.0477	-1.0287	-1.1765	NA
2.176	-0.5346	-0.4711	-0.4283	-0.4401	-0.4720	-0.4647	-0.5969	-0.6596
2.699	-0.0029	0.0572	0.0953	0.1340	0.0369	0.0469	-0.0744	-0.1553
3.398	0.6993	0.7498	0.8023	0.8298	0.7354	0.7492	0.6227	0.5204

Y-ESTIMATES

-1.0948	-1.0269	-0.9978	-1.0010	-1.0461	-1.0319	-1.1743	NA
-0.5230	-0.4600	-0.4237	-0.4153	-0.4770	-0.4640	-0.5994	-0.6599
-0.0021	0.0563	0.0993	0.1182	0.0413	0.0533	-0.0758	-0.1549
0.6942	0.7466	0.7984	0.8213	0.7342	0.7448	0.6242	0.5203

MONS 218250

FOUR-POINT CALIBRATION IN TRIPLICATE (SP-2331 Only)

TCDD

Regression Output:

Constant	-2.6708
Std Err of Y Est	0.0104
R Squares	0.9999
No. of Observations	4.0000
Degrees of Freedom	2.0000
X Coefficient(s)	0.9962
Std Err of Coef.	0.0079

TCDF

Regression Output:

Constant	-2.6089
Std Err of Y Est	0.0097
R Squared	0.9999
No. of Observations	4.0000
Degrees of Freedom	2.0000
X Coefficient(s)	0.9875
Std Err of Coef.	0.0073

[illegible]

Regression Output:

Constant	-2.6002
Std Err of Y Est	0.0061
R Squared	1.0000
No. of Observations	4.0000
Degrees of Freedom	2.0000
X Coefficient(s)	1.0002
Std Err of Coef.	0.0046

[illegible]

Regression Output:

Constant	-2.6356
Std Err of Y Est	0.0222
R Squared	0.9995
No. of Observations	4.0000
Degrees of Freedom	2.0000
X Coefficient(s)	1.0203
Std Err of Coef.	0.0167

MONS 218251

FOUR-POINT CALIBRATION IN TRIPLICATE (SP-2331 50s)

123478-HxCDF
Regression Output:

Constant	-2.6342
Std Err of Y Est	0.0049
R Squared	1.0000
No. of Observations	4.0000
Degrees of Freedom	2.0000
X Coefficient(s)	0.9913
Std Err of Coef.	0.0037

234678-HxCDF
Regression Output:

Constant	-2.6168
Std Err of Y Est	0.0059
R Squared	1.0000
No. of Observations	4.0000
Degrees of Freedom	2.0000
X Coefficient(s)	0.9893
Std Err of Coef.	0.0045

1234678-HpCDF
Regression Output:

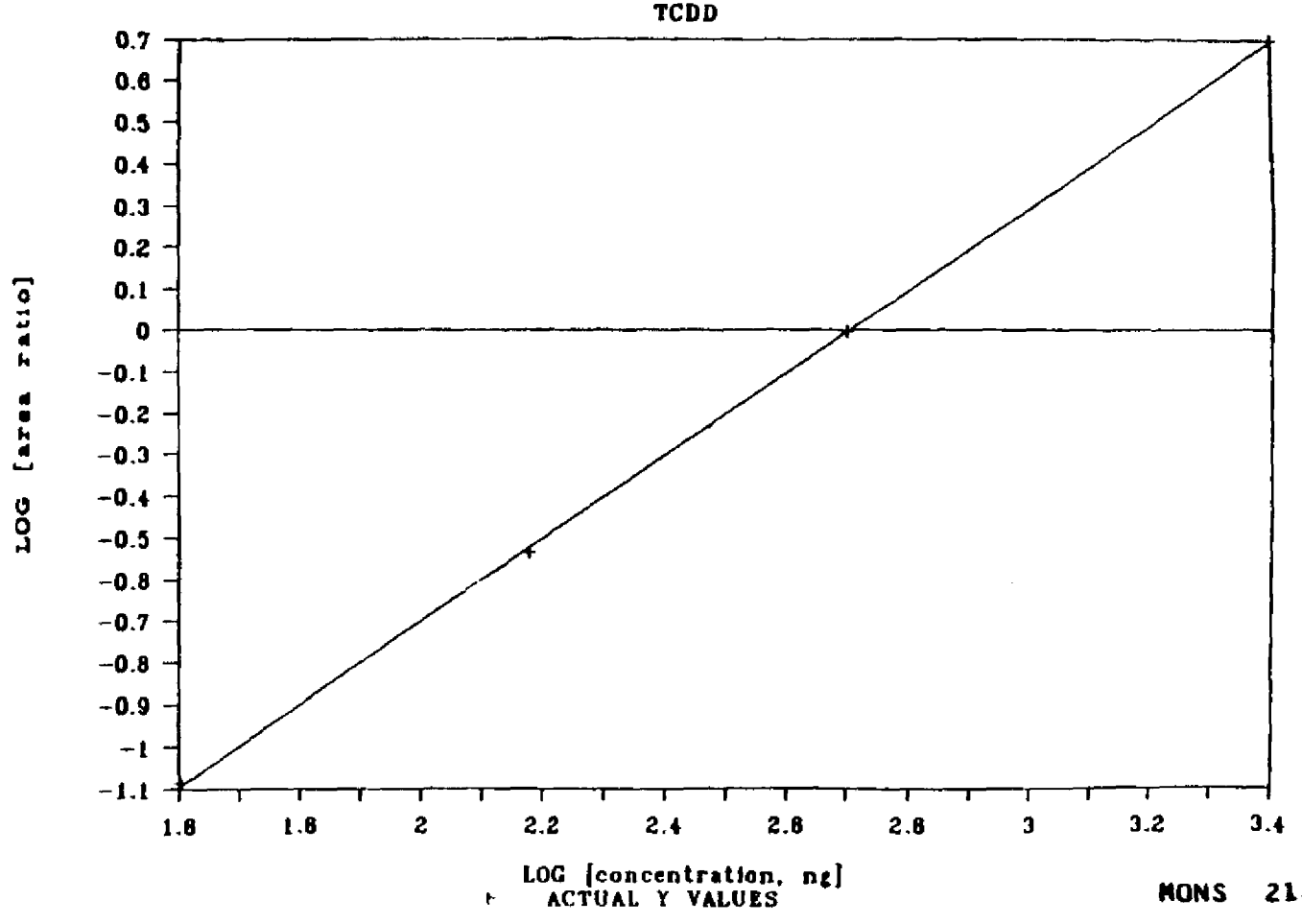
Constant	-2.7788
Std Err of Y Est	0.0028
R Squared	1.0000
No. of Observations	4.0000
Degrees of Freedom	2.0000
X Coefficient(s)	1.0015
Std Err of Coef.	0.0021

OCDF
Regression Output:

Constant	-2.7618
Std Err of Y Est	0.0007
R Squared	1.0000
No. of Observations	3.0000
Degrees of Freedom	1.0000
X Coefficient(s)	0.9659
Std Err of Coef.	0.0008

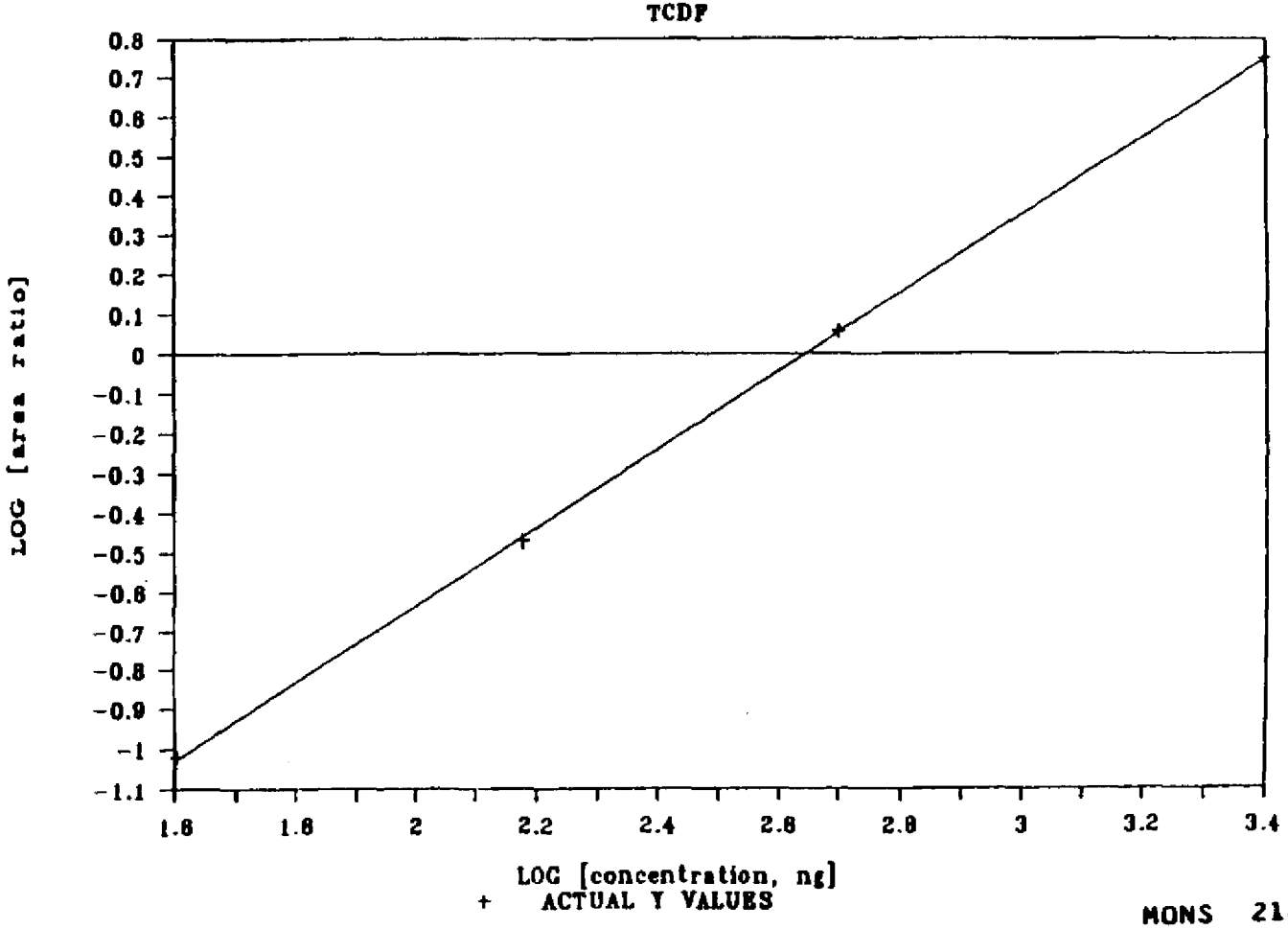
MONS 218252

CALCULATED SP-2331 CALIBRATION CURVE



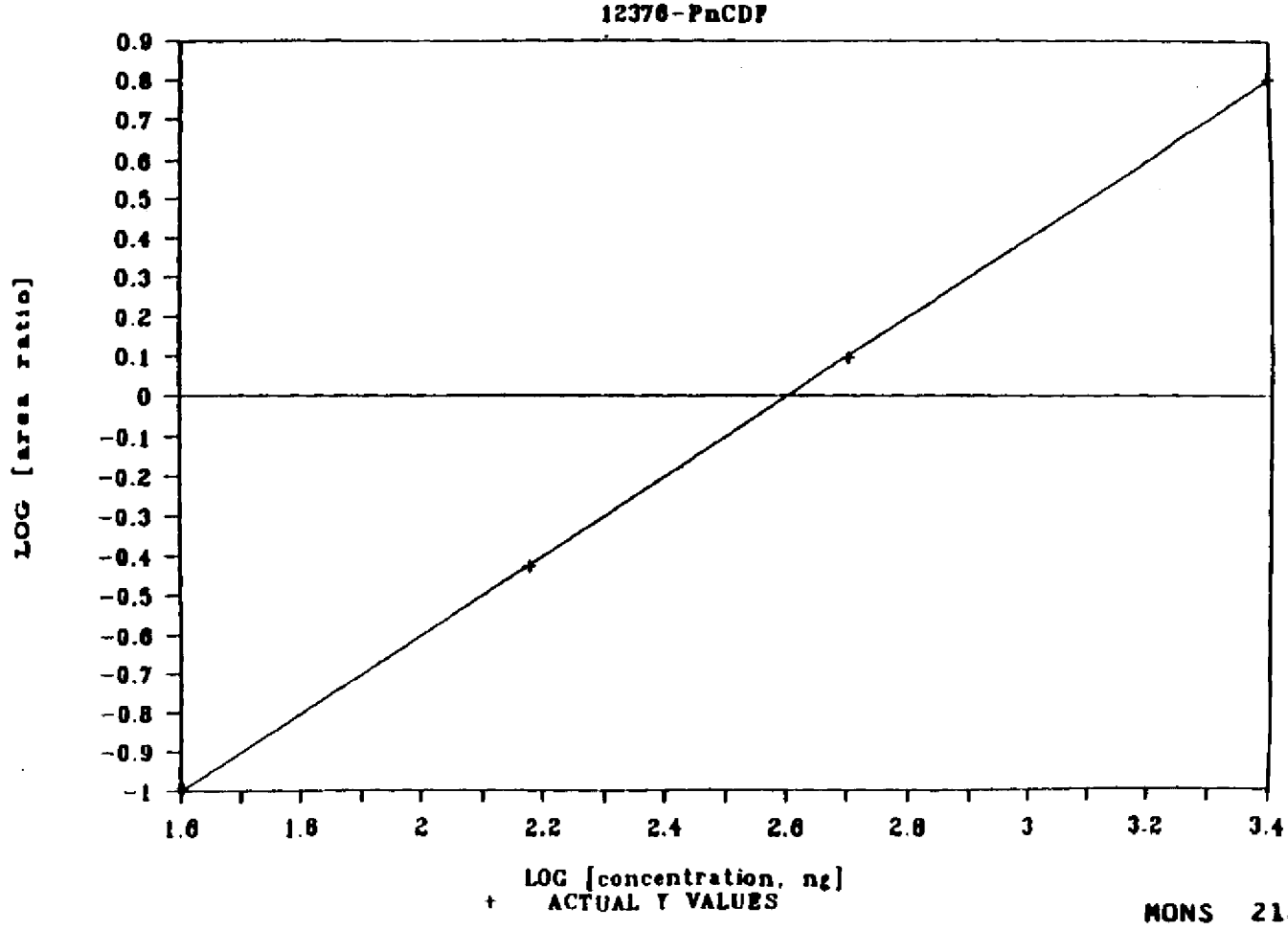
MONS 218253

CALCULATED SP-2331 CALIBRATION CURVE



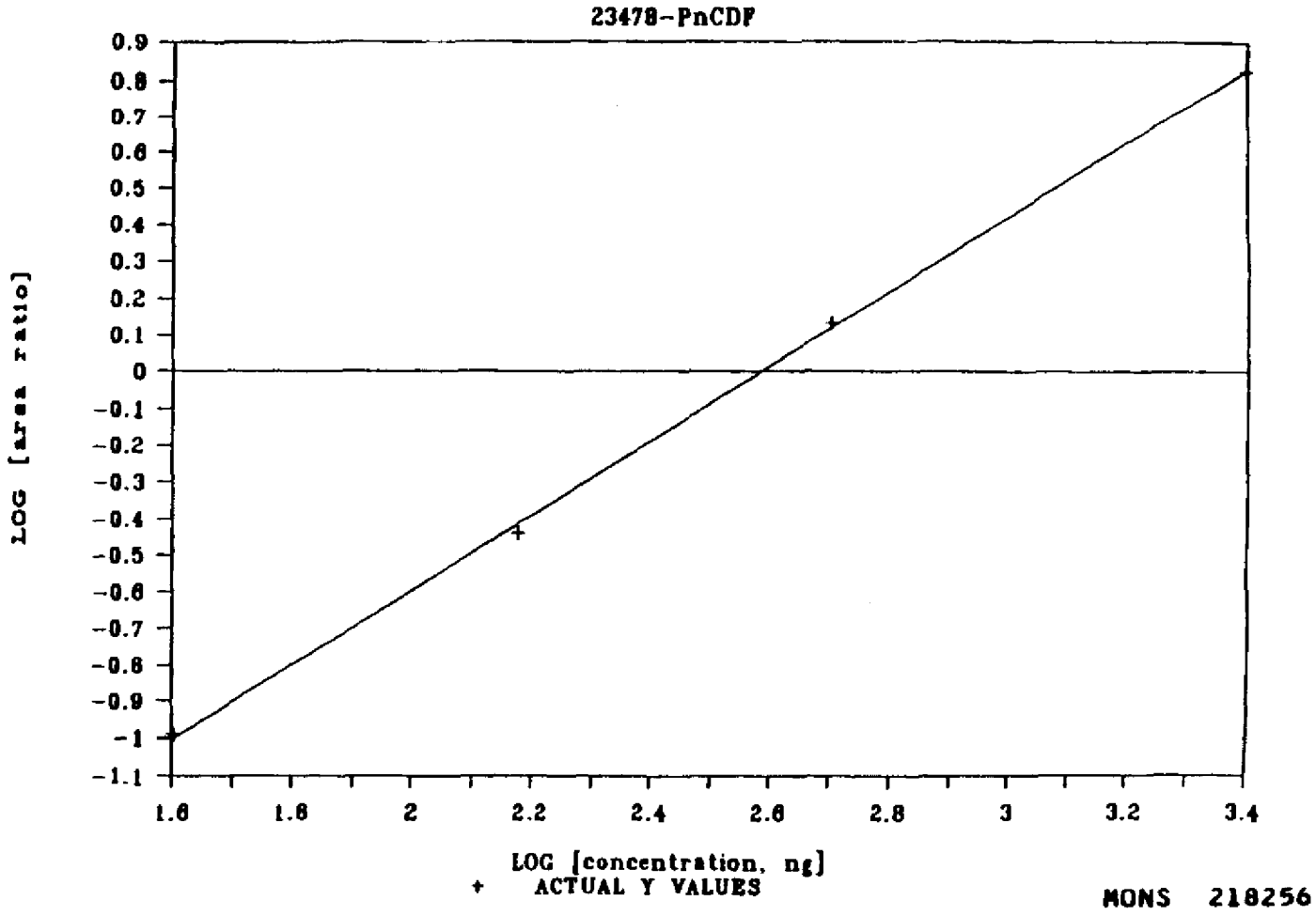
MONS 218254

CALCULATED SP-2331 CALIBRATION CURVE

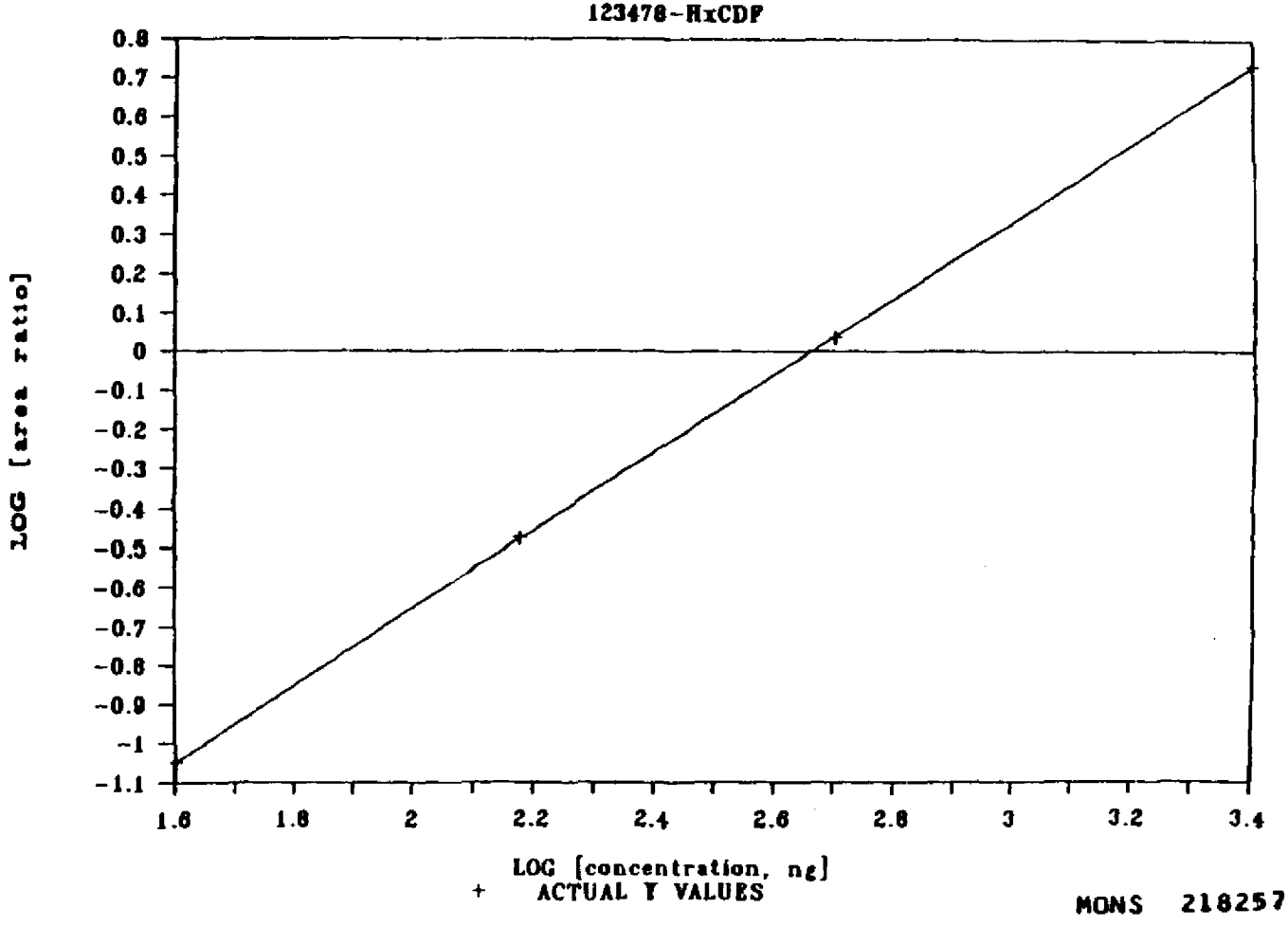


MONS 218255

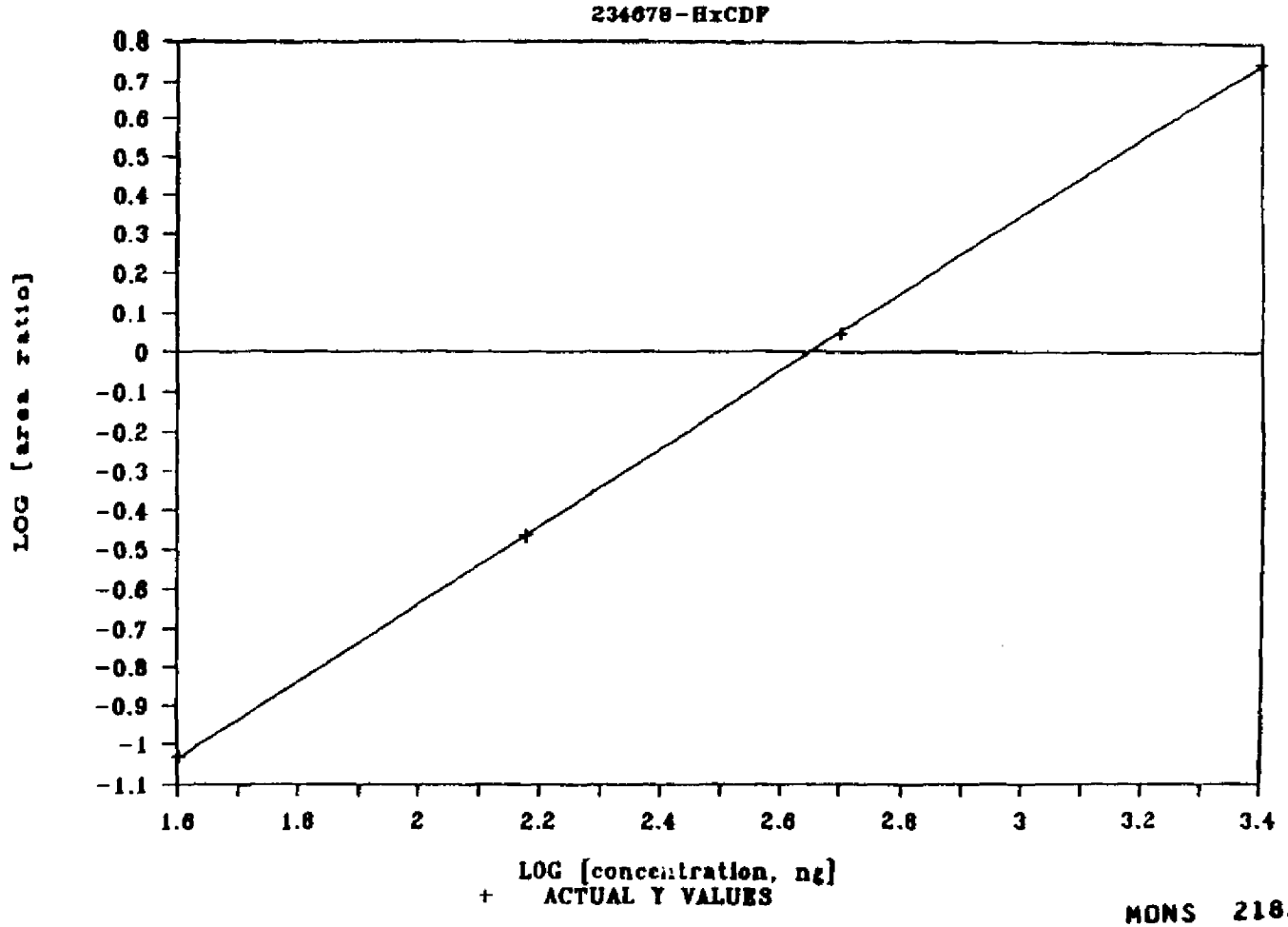
CALCULATED SP-2331 CALIBRATION CURVE



CALCULATED SP-2331 CALIBRATION CURVE

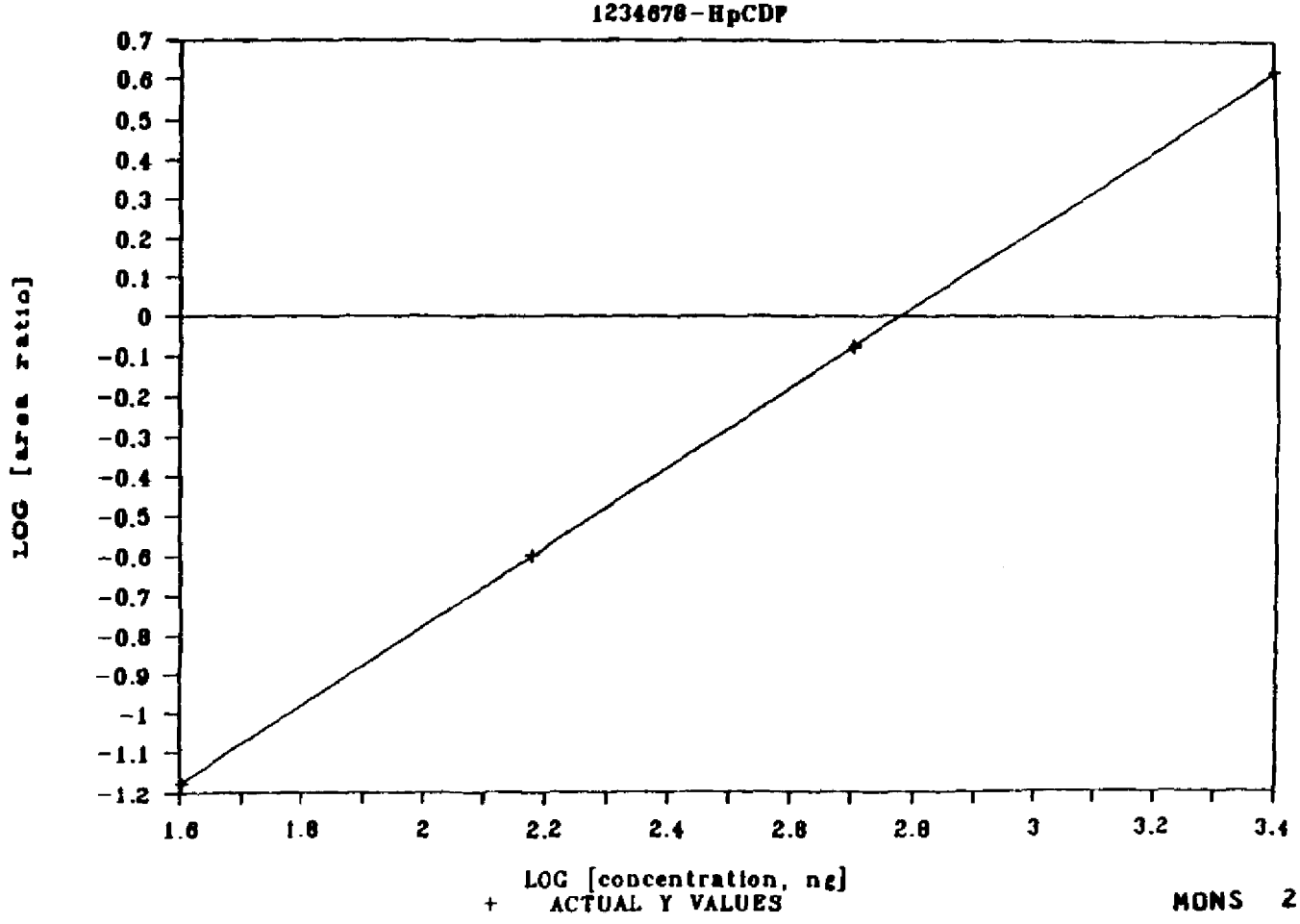


CALCULATED SP-2331 CALIBRATION CURVE

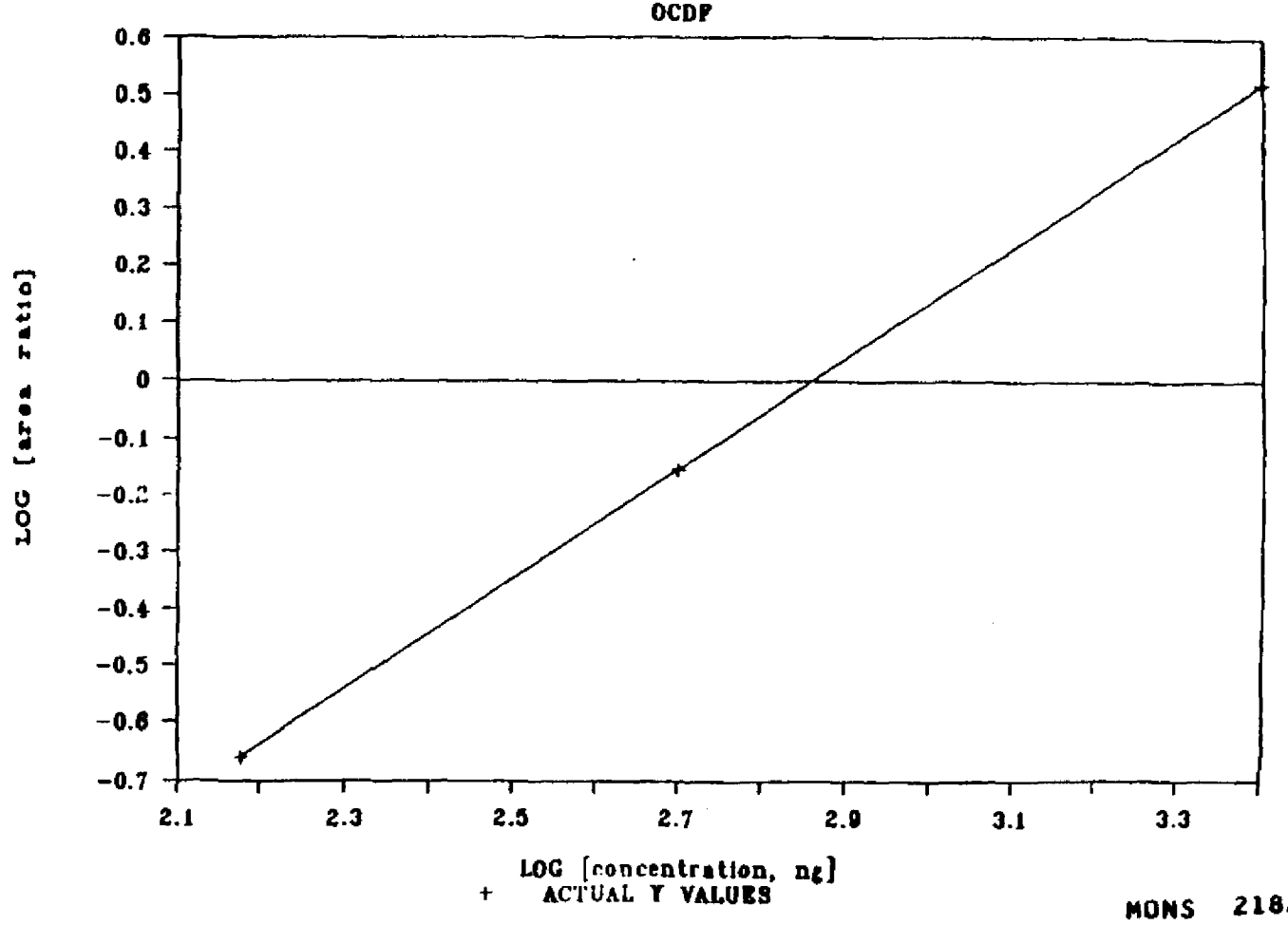


MONS 218258

CALCULATED SP-2331 CALIBRATION CURVE



CALCULATED SP-2331 CALIBRATION CURVE



Appendix G

EXTRACTION BLANK RESULTS

The following pages contain the results of the blank extractions performed during the course of this program.

MONS 218261

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN BLANK ANALYSIS RESULTS

SAMPLE: A509049
SOLVENT BLANK

PCDF/PCDD	DET.L
TOTAL TCDF	<3
TOTAL PnCDF	<3
TOTAL HxCDF	<3
TOTAL HpCDF	<3
*OCDF	<10
TOTAL TCDD	<3
OCDD	<10
COMPOUNDS	
*2378-TCDD	<3
*[2378-TCDF]	<3
[2348-TCDF]	
*([12378-PnCDF]	<3
[12348-PnCDF])	
*23478-PnCDF	<3
*123478-HxCDF	<3
123479-HxCDF	<3
[23789-HxCDF	<3
123678-HxCDF	<3
*234678-HxCDF	<3
*1234678-HpCDF	<3

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[],()=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF ng/g.

MONS 218262

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN BLANK ANALYSIS RESULTS

SAMPLE: AS10015-C2
SOLVENT BLANK

PCDF/PCDD	DET.?
TOTAL TCDF	10
TOTAL PnCDF	<3
TOTAL HxCDF	<3
TOTAL HpCDF	8
*OCDF	<10
TOTAL TCDD	<3
OCDD	<10
COMPOUNDS	
*2378-TCDD	<3
*[2378-TCDF]	7
[2348-TCDF]	
*([12378-PnCDF]	<3
[12348-PnCDF]	
*23478-PnCDF	<3
*123478-HxCDF	7
123479-HxCDF	<3
123789-HxCDF	<3
123678-HxCDF	<3
*234678-HxCDF	<3
*1234678-HpCDF	5

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[],()=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218263

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN BLANK ANALYSIS RESULTS

SAMPLE: AS10013-02
SOLVENT BLANK

PCDF/PCDD	DET.
TOTAL TCDF	17
TOTAL PnCDF	13
TOTAL HxCDF	13
TOTAL HpCDF	<3
*OCDF	<10
TOTAL TCDD	<3
OCDD	<10
COMPOUNDS	
*2378-TCDD	<3
*(2378-TCDF] [2348-TCDF]	7
*(12378-PnCDF] [12348-PnCDF]	7
*23478-PnCDF	7
*123478-HxCDF	8
123479-HxCDF	<3
123789-HxCDF	<3
123678-HxCDF	<3
*234679-HxCDF	<3
*1234678-HpCDF	<3

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(),,=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/S.

MONS 218264

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN BLANK ANALYSIS RESULTS

SAMPLE: A510915-E2
SOLVENT BLANK

PCDF/PCDD	DET.L
TOTAL TCDF	3
TOTAL PnCDF	<3
TOTAL HxCDF	<3
TOTAL HpCDF	<3
*OCDF	<10
TOTAL TCDD	<3
OCDD	<10
COMPOUNDS	
*2378-TCDD	<3
*(2378-TCDF) (2348-TCDF)	3
*((12378-PnCDF) (12348-PnCDF)	<3
*23478-PnCDF	<3
*123478-HxCDF	<3
123479-HxCDF	<3
123789-HxCDF	<3
123678-HxCDF	<3
*234678-HxCDF	<3
*1234678-HpCDF	<3

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(),(]=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218265

POLYCHLOROBIBENZO-p-DIOXIN AND POLYCHLOROBIBENZOFURAN BLANK ANALYSIS RESULTS

SAMPLE: AS10015-F2
SOLVENT BLANK

PCDF/PCDD	DET.L
TOTAL TCDF	<3
TOTAL PnCDF	<3
TOTAL HxCDF	<3
TOTAL HpCDF	<3
*OCDF	<10
TOTAL TCDD	<3
OCDD	<10
COMPOUNDS	
*2378-TCDD	<3
*(2378-TCDF)	<3
(2348-TCDF)	
*((12378-PnCDF)	<3
(12348-PnCDF)	
*23478-PnCDF	<3
*123478-HxCDF	<3
123479-HxCDF	<3
123789-HxCDF	<3
123678-HxCDF	<3
*234678-HxCDF	<3
*1234678-HpCDF	<3

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(),()=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218266

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN BLANK ANALYSIS RESULTS

SAMPLE: AS10015-C3
SOLVENT BLANK

PCDF/PCDD	DET.]
TOTAL TCDF	<3
TOTAL PnCDF	<3
TOTAL HxCDF	<3
TOTAL HpCDF	<3
*OCDF	<10
TOTAL TCDD	<3
OCDD	<10
COMPOUNDS	
*2378-TCDD	<3
*(2378-TCDF)	<3
[2349-TCDF]	
*(12378-PnCDF)	<3
(12348-PnCDF)	
*23478-PnCDF	<3
*123478-HxCDF	<3
123479-HxCDF	<3
[23789-HxCDF	<3
[23678-HxCDF	<3
*234678-HxCDF	<3
*1234678-HpCDF	<3

* PRIMARY TIEPS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(),()=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218267

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN BLANK ANALYSIS RESULTS

SAMPLE: AS10015-C3
SOLVENT BLANK

PCDF/PCDD	DET. L
TOTAL TCDF	<3
TOTAL PnCDF	<3
TOTAL HxCDF	<3
TOTAL HpCDF	<3
+OCDF	<10
TOTAL TCDD	<3
OCDD	<10
COMPOUNDS	
+2378-TCDD	<3
+(2378-TCDF)	<3
[2348-TCDF]	
+(12378-PnCDF)	<3
[12348-PnCDF]	
+23478-PnCDF	<3
+123478-HxCDF	<3
123479-HxCDF	<3
123789-HxCDF	<3
123678-HxCDF	<3
+234678-HxCDF	<3
+1234678-HpCDF	<3

+ PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[],()=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218268

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN BLANK ANALYSIS RESULTS

SAMPLE: A510015-03
SOLVENT BLANK

PCDF/PCDD	DET.[1]
TOTAL TCDF	<3
TOTAL PnCDF	<3
TOTAL HxCDF	<3
TOTAL HpCDF	<3
*OCDF	<10
TOTAL TCDD	<3
OCDD	<10
COMPOUNDS	
*2378-TCDD	<3
*(2378-TCDF] [2348-TCDF)	<3
*(12378-PnCDF) (12348-PnCDF)	<3
*23478-PnCDF	<3
*123478-HxCDF	<3
123479-HxCDF	<3
123789-HxCDF	<3
123678-HxCDF	<3
*234678-HxCDF	<3
*1234678-HpCDF	<3

* PRIMARY ITEMS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[],()=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218269

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN BLANK ANALYSIS RESULTS

SAMPLE: AS10013-EJ
SOLVENT BLANK

PCDF/PCDD	DET.L
TOTAL TCDF	<3
TOTAL PnCDF	<3
TOTAL HxCDF	<3
TOTAL HpCDF	<3
*OCDF	<10
TOTAL TCDD	<3
OCDD	<10
COMPOUNDS	
*2378-TCDD	<3
*[2378-TCDF]	<3
[2348-TCDF]	
*(12378-PnCDF)	<3
(12348-PnCDF)	
*23478-PnCDF	<3
*123478-HxCDF	<3
123479-HxCDF	<3
123789-HxCDF	<3
123678-HxCDF	<3
*234678-HxCDF	<3
*1234678-HpCDF	<3

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[],()=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218270

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN BLANK ANALYSIS RESULTS

SAMPLE: A510015-F3
SOLVENT BLANK

PCDF/PCDD	DET.1
TOTAL TCDF	<3
TOTAL PnCDF	<3
TOTAL HxCDF	<3
TOTAL HpCDF	<3
*OCDF	<10
TOTAL TCDD	<3
OCDD	<10
COMPOUNDS	
*2378-TCDD	<3
*[2378-TCDF]	<3
[2348-TCDF]	
*[12378-PnCDF]	<3
[12348-PnCDF]	
*23478-PnCDF	<3
*123478-HxCDF	<3
123479-HxCDF	<3
122789-HxCDF	<3
123678-HxCDF	<3
*234678-HxCDF	<3
*1234678-HpCDF	<3

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[],{}=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218271

Appendix H

BLANK BASELINE LIQUID RESULTS

The following peges contain the results of extraction and analysis of the blank (not-spiked) Baseline Liquids.

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN BLANK ANALYSIS RESULTS

SAMPLE: E729-54-02
MINERAL OIL BLANK

PCDF/PCDD	DET.I
TOTAL TCDF	<3
TOTAL PnCDF	<3
TOTAL HxCDF	<3
TOTAL HpCDF	<3
*OCDF	<10
TOTAL TCDD	<3
OCDD	<10
COMPOUNDS	
*2378-TCDD	<10
*[2378-TCDF]	<10
[2348-TCDF]	
*[12378-PnCDF]	<10
[12348-PnCDF]	
*23478-PnCDF	<10
*123478-HxCDF	<10
123479-HxCDF	<10
[23789-HxCDF	<10
123678-HxCDF	<10
*234678-HxCDF	<10
*1234678-HpCDF	<10

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[],()=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218273

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN BLANK ANALYSIS RESULTS

SAMPLE: E729-54-04
ARDCLOR 1016 BLANK

PCDF/PCDD	DET.I
TOTAL TCDF	<3
TOTAL PnCDF	<3
TOTAL HxCDF	11
TOTAL HpCDF	27
+OCDF	32
TOTAL TCDD	<3
OCDD	<10
COMPOUNDS	
+2378-TCDD	<3
+[2378-TCDF] (2348-TCDF)	<3
+(12378-PnCDF) (12348-PnCDF)	<3
+23478-PnCDF	<3
+123478-HxCDF	11
123479-HxCDF	<3
123789-HxCDF	<3
123678-HxCDF	<3
+234678-HxCDF	<3
+1234678-HpCDF	12

+ PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[],()=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218274

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN BLANK ANALYSIS RESULTS

SAMPLE: E729-S4-03
AROCLOR 1260 BLANK

PCDF/PCDD	DET.L
TOTAL TCDF	301
TOTAL PnCDF	1000
TOTAL HxCDF	1263
TOTAL HpCDF	1981
*OCDF	3541
TOTAL TCDD	<3
OCDD	<10
COMPOUNDS	
*2378-TCDD	<3
*[2378-TCDF]	19
[2348-TCDF]	
*12378-PnCDF	70
12348-PnCDF	149
*23478-PnCDF	178
*123478-HxCDF	609
123479-HxCDF	33
123789-HxCDF	36
[23678-HxCDF	40
*234678-HxCDF	67
*1234678-HpCDF	625

* PRIMARY TISSUE OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[]=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218275

Appendix I

BASELINE LIQUID RESULTS

The following pages contain the analytical data from the extraction and analysis of the Baseline Liquids in the Round Robin Analysis. For each sample, the average results of three extractions are presented, followed by isomer resolution results, and then the results of triplicate determination for each extract.

MONS 218276

AVERAGE POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE EXTRACTIONS

SAMPLE: E729-55-01

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	178	195	195	189	9	2
TOTAL PnCDF	290	307	297	298	8	3
TOTAL HxCDF	496	481	474	480	6	1
TOTAL HpCDF	412	424	375	407	29	7
OCDF	16	11	NA	14	NA	NA
TOTAL TCDD	25	25	23	24	1	4
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	25	25	23	24	1	4
*[2378-TCDF]	178	195	185	183	9	2
[2348-TCDF]						
*12378-PnCDF	146	137	140	141	5	3
12348-PnCDF	<3	<3	<3	<3	NA	NA
*23478-PnCDF	144	163	157	155	10	6
*123478-HxCDF	263	235	258	259	6	2
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	51	49	43	48	4	9
*234678-HxCDF	172	177	173	174	3	2
*1234678-HpCDF	412	424	375	407	29	7

* PRIMARY TIERS OF DATA. I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[]=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218277

POLYCHLORODIBENZOFURAN ISOMER SEPARATION RESULTS

EXAMPLE: E729-S3-01

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	178	185	185	183	4	2
TOTAL PnCDF	290	307	297	298	8	3
TOTAL HxCDF	484	481	474	480	6	1
TOTAL HpCDF	412	434	375	407	29	7
OCDF	16	11	NA	14	NA	NA
TOTAL TCDD	25	25	23	24	1	4
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	25	25	23	24	1	4
*(2378-TCDF)	101	130	107	113	15	14
(2348-TCDF)	77	70	78	75	4	6
*(12378-PnCDF)	146	137	140	141	5	3
(12348-PnCDF)	<3	<3	<3	<3	NA	NA
*23478-PnCDF	144	163	157	155	10	6
*123478-HxCDF	263	255	258	259	4	2
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	51	49	47	48	4	9
*234678-HxCDF	172	177	173	174	3	2
*1234678-HpCDF	412	434	375	407	29	7

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(I,J)=PAIRS RESOLVED BY CHROMATOGRAPHY.

SINGLE RESOLUTION OF ISOMER PAIRS SHOWN WITH THE AVERAGE VALUE FOR EACH EXTRACTION. ALL DATA IN UNITS OF NG/G.

MONS 218278

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: E729-S5-01
EXTRACT: I

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	168	180	177	178	10	6
TOTAL PnCDF	285	303	303	299	22	8
TOTAL HxCDF	493	483	482	486	6	1
TOTAL HpCDF	459	386	389	412	40	10
OCDF	19	<19	13	16	NA	NA
TOTAL TCDD	24	25	25	25	1	2
OCDD	<19	<19	<19	<19	NA	NA
COMPOUNDS						
*2378-TCDD	24	25	25	25	1	2
*[2378-TCDF]	168	180	177	178	10	6
[2348-TCDF]						
*[12378-PnCDF]	144	147	148	146	2	1
[12348-PnCDF]						
*23478-PnCDF	121	156	155	144	20	14
*123478-HxCDF	265	263	259	263	3	1
123479-HxCDF	3	<3	<3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	52	49	53	51	2	4
*234678-HxCDF	175	169	170	172	4	2
*1234678-HpCDF	458	388	389	412	40	10

* =PRIMARY TIERS OF DATA. I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[ ]=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/S.

MONS 218279

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: E729-53-01
EXTRACT: 2

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	199	181	176	185	12	7
TOTAL PnCDF	309	317	295	307	11	4
TOTAL HxCDF	477	486	479	481	5	1
TOTAL HpCDF	411	435	455	434	22	5
*OCDF	11	<10	<10	11	NA	NA
TOTAL TCDD	25	26	24	25	1	4
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	25	26	24	25	1	4
*[2378-TCDF]	199	191	176	195	12	7
[2348-TCDF]						
*[12378-PnCDF]	142	136	134	137	4	3
[12348-PnCDF]						
*23478-PnCDF	167	161	161	163	3	2
*123478-HxCDF	256	260	248	255	6	2
123479-HxCDF	3	<3	<3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	47	47	52	49	3	5
*234678-HxCDF	174	179	179	177	3	2
*1234678-HpCDF	411	435	455	434	22	5

* PRIMARY TIERS OF DATA. I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[X,X]=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218280

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: E729-53-01
EXTRACT: 3

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	185	189	182	185	4	2
TOTAL PnCDF	298	292	260	283	55	21
TOTAL HxCDF	465	480	477	474	9	2
TOTAL HpCDF	366	384	376	375	9	2
*OCDF	<10	<10	<10	<10	NA	NA
TOTAL TCDD	24	22	22	23	1	4
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	24	22	22	23	1	4
*(2378-TCDF) (2348-TCDF)	185	189	182	185	4	2
*(12378-PnCDF) (12348-PnCDF)	140	139	141	140	1	1
*23478-PnCDF	159	152	159	157	3	2
*123478-HxCDF	247	265	262	258	10	4
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	47	41	41	43	3	8
*234678-HxCDF	170	174	174	173	2	1
*1234678-HpCDF	366	384	376	375	9	2

* PRIMARY PEAKS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(1,2)=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218281

AVERAGE POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE EXTRACTIONS

SAMPLE: E729-53-02

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	63	60	60	61	2	3
TOTAL PnCDF	301	310	304	305	5	2
TOTAL HxCDF	190	196	195	194	3	2
TOTAL HpCDF	549	600	525	558	38	7
OCDF	127	123	128	126	3	2
TOTAL TCDD	51	51	50	50	1	1
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	51	51	50	50	1	1
*[2378-TCDF] [2348-TCDF]	63	60	60	61	2	3
*12378-PnCDF	157	153	156	155	2	1
12348-PnCDF	<3	<3	<3	<3	NA	NA
*23478-PnCDF	144	157	148	150	7	5
*123478-HxCDF	<3	<3	<3	<3	NA	NA
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	58	57	60	58	2	3
*234678-HxCDF	133	139	135	136	3	3
*1234678-HpCDF	549	600	525	558	38	7

* PRIMARY ITEMS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[]=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218282

POLYCHLORODIBENZOFURAN ISOMER SEPARATION RESULTS

SAMPLE: E729-SS-02

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	63	60	60	61	2	3
TOTAL PnCDF	301	310	304	305	5	2
TOTAL HxCDF	190	196	195	194	3	2
TOTAL HpCDF	549	600	525	558	38	7
OCDF	127	123	128	126	3	2
TOTAL TCDD	51	51	50	50	1	1
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	51	51	50	50	1	1
*(2378-TCDF)	63	60	60	61	2	3
(2348-TCDF)	<3	<3	<3	<3	NA	NA
*(12378-PnCDF)	157	153	156	155	2	1
(12348-PnCDF)	<3	<3	<3	<3	NA	NA
*23478-PnCDF	144	157	148	150	7	5
*123478-HxCDF	<3	<3	<3	<3	NA	NA
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	58	57	60	58	2	3
*234678-HxCDF	133	139	135	136	3	3
*1234678-HpCDF	549	600	525	558	38	7

* PRIMARY TIEPS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(1,2)=PAIRS RESOLVED BY CHROMATOGRAPHY.

SINGLE RESOLUTION OF ISOMER PAIRS SHOWN WITH THE AVERAGE VALUE FOR EACH EXTRACTION. ALL DATA IN UNITS OF NG/G.

MONS 218283

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: E729-53-02
EXTRACT: 1

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	61	64	63	63	2	2
TOTAL PnCDF	298	303	301	301	3	1
TOTAL HxCDF	194	189	188	190	3	2
TOTAL HpCDF	549	539	559	549	17	3
*OCDF	125	126	129	127	2	2
TOTAL TCDD	52	49	51	51	2	3
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	52	49	51	51	2	3
*(12378-TCDF)	61	64	63	63	2	2
(2348-TCDF)						
*(12378-PnCDF)	155	159	157	157	2	1
(12348-PnCDF)						
*23478-PnCDF	142	144	144	144	1	0
*123478-HxCDF	<3	<3	<3	<3	NA	NA
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	61	57	55	58	3	5
*234678-HxCDF	133	132	133	133	1	0
*1234678-HpCDF	549	539	559	549	17	3

* PRIMARY PIECE OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
( ).( )=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218284

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: E729-SC-02
EXTRACT: C

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	62	60	57	60	3	4
TOTAL PnCDF	310	305	306	310	0	3
TOTAL HxCDF	197	197	194	196	2	1
TOTAL HpCDF	577	596	628	600	26	4
*OCDF	109	122	116	115	5	4
TOTAL TCDD	50	52	50	51	1	2
OCDD	19	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	50	52	50	51	1	2
*(2378-TCDF)	62	60	57	60	3	4
(2348-TCDF)						
*(12378-PnCDF)	157	151	152	153	3	2
(12348-PnCDF)						
*23478-PnCDF	153	154	154	157	5	3
*123478-HxCDF	<3	<3	<3	<3	NA	NA
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	57	58	55	57	2	3
*234678-HxCDF	140	139	139	139	1	0
*1234678-HpCDF	577	596	628	600	26	4

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(),()=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218285

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: E729-53-02
EXTRACT: 3

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	60	60	61	60	1	1
TOTAL PnCDF	306	300	306	304	3	1
TOTAL HxCDF	201	184	199	195	9	5
TOTAL HpCDF	561	496	519	525	33	6
*OCDF	135	123	136	128	7	5
TOTAL TCDD	50	51	48	50	2	3
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	50	51	48	50	2	3
*(2378-TCDF)	60	60	61	60	1	1
[2346-TCDF]						
*(12378-PnCDF)	156	154	157	156	2	1
(12348-PnCDF)						
*23478-PnCDF	150	146	149	148	2	1
*123478-HxCDF	<3	<3	<3	<3	NA	NA
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	64	55	61	60	5	8
*234678-HxCDF	137	129	138	135	5	4
*1234678-HpCDF	561	496	519	525	33	6

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[],()=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218286

AVERAGE POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE EXTRACTIONS

SAMPLE: E729-53-03

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	1480	1473	1573	1509	56	4
TOTAL PnCDF	1412	1265	1353	1343	74	6
TOTAL HxCDF	2483	2307	2220	2363	133	6
TOTAL HpCDF	2424	2246	2085	2252	169	8
*OCDF	3913	3662	4189	3921	264	7
TOTAL TCDD	99	105	94	99	5	5
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	99	105	94	99	5	5
*(2378-TCDF) (2348-TCDF)	1154	1104	1149	1136	28	2
*12378-PnCDF	183	173	182	180	5	3
12348-PnCDF	131	123	143	132	10	8
*23478-PnCDF	260	268	287	278	10	3
*123478-HxCDF	1137	1137	1142	1139	3	0
123479-HxCDF	38	31	29	33	5	15
123789-HxCDF	33	34	38	35	3	8
123678-HxCDF	305	293	298	299	6	2
*234678-HxCDF	321	308	312	327	18	6
*1234678-HpCDF	774	791	740	768	26	3

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
;;=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218287

POLYCHLORODIBENZOFURAN ISOMER SEPARATION RESULTS

SAMPLE: E729-53-03

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	1480	1473	1573	1509	56	4
TOTAL PeCDF	1412	1265	1353	1343	74	6
TOTAL HxCDF	2403	2287	2220	2303	123	6
TOTAL HpCDF	2424	2246	2085	2252	169	8
*OCDF	3913	3662	4189	3921	264	7
TOTAL TCDD	99	105	94	99	5	5
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	99	105	94	99	5	5
*(2378-TCDF)	914	854	874	881	31	3
(2348-TCDF)	117	178	154	150	31	21
*(12378-PeCDF)	172	186	192	183	10	6
(12348-PeCDF)	128	137	129	131	5	4
*23478-PeCDF	280	260	287	276	10	3
*123478-HxCDF	1137	1137	1142	1139	3	0
123479-HxCDF	30	31	39	33	5	15
*123789-HxCDF	33	34	38	35	3	8
123678-HxCDF	305	293	298	299	6	2
*234678-HxCDF	321	340	312	327	10	6
*1234678-HpCDF	774	791	740	768	26	3

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(),()=PAIRS RESOLVED BY CHROMATOGRAPHY.

SINGLE RESOLUTION OF ISOMER PAIRS SHOWN WITH THE AVERAGE VALUE FOR EACH EXTRACTION. ALL DATA IN UNITS OF NG/G.

MONS 218288

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: E729-53-03
EXTRACT: 1

PCDF/PCDD		DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
	TOTAL TCDF	1593	1416	1431	1480	98	7
	TOTAL PnCDF	1605	1310	1322	1412	167	12
	TOTAL HxCDF	2348	2795	2306	2483	271	11
	TOTAL HpCDF	2492	2416	2365	2424	65	3
	*OCDF	4029	3910	3801	3913	114	3
	TOTAL TCDD	100	100	97	99	2	2
	OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS							
	*2378-TCDD	100	100	97	99	2	2
	*[2378-TCDF]	1141	1157	1165	1154	12	1
	[2348-TCDF]						
	*12378-PnCDF	172	186	192	183	10	6
	12348-PnCDF	128	137	129	131	5	4
	*23478-PnCDF	281	282	278	280	2	1
	*123478-HxCDF	1113	1156	1142	1137	22	2
	123479-HxCDF	29	30	32	30	2	5
	123789-HxCDF	31	34	33	33	2	5
	123678-HxCDF	323	294	298	305	16	5
	*234678-HxCDF	317	324	323	321	4	1
	*1234678-HpCDF	781	780	762	774	11	1

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[]=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218289

POLYCHLORODIPHENYL-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: E729-53-03
EXTRACT: 2

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	1407	1538	1474	1473	66	4
TOTAL PnCDF	1267	1398	1329	1265	135	11
TOTAL HxCDF	1335	2400	2425	2053	622	30
TOTAL HpCDF	2095	2253	2389	2246	147	7
*OCDF	3772	3806	3407	3662	221	6
TOTAL TCDD	101	102	111	105	6	5
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	101	102	111	105	6	5
*(2378-TCDF) [2348-TCDF]	1150	1093	1068	1104	42	4
*12378-PnCDF	183	179	158	173	13	8
12348-PnCDF	126	128	115	123	7	6
*23478-PnCDF	279	272	252	268	14	5
*123478-HxCDF	1126	1168	1117	1137	27	2
123479-HxCDF	31	27	34	31	4	11
123789-HxCDF	36	33	33	34	2	5
123678-HxCDF	274	280	324	293	27	9
*234678-HxCDF	320	393	330	348	40	11
*1234678-HpCDF	751	787	836	791	43	5

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[]=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218290

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: E729-53-03
EXTRACT: 3

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	1593	1575	1552	1573	21	1
TOTAL PnCDF	1431	1311	1316	1353	68	5
TOTAL HxCDF	2244	2171	2244	2220	42	2
TOTAL HpCDF	2254	1976	2026	2085	149	7
OCDF	4130	3996	4441	4189	228	5
TOTAL TCDD	96	93	94	94	2	2
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	96	93	94	94	2	2
*[2378-TCDF]	1154	1121	1171	1149	25	2
[2348-TCDF]						
*12378-PnCDF	171	192	193	182	13	6
12348-PnCDF	162	139	128	143	17	12
*23478-PnCDF	296	282	282	287	8	3
*123478-HxCDF	1179	1099	1148	1142	40	4
123479-HxCDF	55	45	78	59	5	13
123789-HxCDF	39	38	39	38	1	2
123678-HxCDF	302	304	298	298	6	2
*234678-HxCDF	317	305	315	312	6	2
*1234678-HpCDF	786	704	729	740	42	6

* PRIMARY TIERS OF DATA. I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[ ]=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218291

Appendix J

IN-SERVICE LIQUID RESULTS

The following pages contain the analytical data from the extraction and analysis of the In-Service Liquids in the Round Robin Analysis. For each sample, the average results of three extractions are presented, followed by isomer resolution results, and then the results of triplicate determinations for each extract.

MONS 218292

AVERAGE POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE EXTRACTIONS

SAMPLE: 41159-11-11

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	144	169	168	160	14	9
TOTAL PnCDF	2031	2045	2084	2053	27	1
TOTAL HxCDF	6976	7065	7125	7055	75	1
TOTAL HpCDF	10143	11080	10959	10727	510	5
*OCDF	25959	10346	28138	21814	9949	46
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF) (2348-TCDF)	36	38	39	38	2	4
*(12378-PnCDF) (12348-PnCDF)	1007	1014	1018	1013	6	1
*23478-PnCDF	107	107	113	109	3	3
*123478-HxCDF	4801	4666	4798	4755	77	2
123479-HxCDF	127	114	118	119	7	6
123789-HxCDF	260	265	269	265	4	2
123678-HxCDF	320	310	305	312	7	2
*234678-HxCDF	196	200	200	199	2	1
*1234678-HpCDF	3827	4185	4172	4061	203	5

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(1,2)=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218293

POLYCHLORODIBENZOFURAN ISOMER SEPARATION RESULTS

SAMPLE: 41139-11-11

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	144	169	168	160	14	9
TOTAL PnCDF	2031	2045	2084	2053	27	1
TOTAL HxCDF	6976	7065	7125	7055	75	1
TOTAL HpCDF	10143	11080	10959	10727	530	5
*OCDF	26959	10346	28138	21814	9949	46
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF)	10	10	8	9	1	12
(2348-TCDF)	18	17	17	17	1	3
*(12378-PnCDF)	1007	1014	1018	1013	6	1
(12348-PnCDF)						
*23478-PnCDF	107	107	113	109	3	3
*123478-HxCDF	4801	4666	4798	4755	77	2
123479-HxCDF	127	114	118	119	7	6
123769-HxCDF	260	265	269	265	4	2
123678-HxCDF	320	310	305	312	7	2
*234678-HxCDF	196	200	200	199	2	1
*1234678-HpCDF	3827	4185	4172	4061	203	5

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(:)=PAIR RESOLVED BY CHROMATOGRAPHY.
(,)=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

SINGLE RESOLUTION OF ISOMER PAIRS SHOWN WITH THE AVERAGE VALUE FOR EACH EXTRACTION. ALL DATA IN UNITS OF NG/G.

MONS 218294

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: [illegible]
EXTRACT: 1

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	147	162	123	144	20	14
TOTAL PnCDF	2206	2217	1670	2031	313	15
TOTAL HxCDF	7143	7709	6477	6976	829	12
TOTAL HpCDF	11271	11208	8000	10143	1856	18
*OCDF	29720	28259	22798	26959	3667	14
TOTAL TCDD	<3	3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	3	<3	<3	<3	NA	NA
*(2378-TCDF) (2748-TCDF)	38	39	31	36	5	10
*(12378-PnCDF) (12348-PnCDF)	1018	998	1106	1007	104	10
*23478-PnCDF	107	116	69	107	25	32
*(123478-HxCDF	4751	5197	4454	4801	374	8
123479-HxCDF	103	149	108	127	21	16
(123789-HxCDF	278	275	227	260	29	11
(12678-HxCDF	334	321	294	320	22	7
*(234678-HxCDF	207	219	182	196	30	15
*(1234678-HpCDF	4221	4210	3060	3827	673	18

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
([illegible]=PEAKS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218295

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41139-11-11
EXTRACT: 2

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	193	179	146	169	20	12
TOTAL PnCDF	2038	1959	2078	2045	41	2.
TOTAL HxCDF	7115	7158	6921	7065	126	2
TOTAL HpCDF	11075	10990	11175	11080	93	1
*OCDF	10192	11292	9554	10346	879	8
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF)	38	38	38	38	0	0
(2348-TCDF)						
*(12378-PnCDF)	1005	1000	1038	1014	21	2
(12348-PnCDF)						
*23478-PnCDF	111	103	107	107	4	4
*123478-HxCDF	4759	4662	4638	4686	69	1
123479-HxCDF	123	118	90	114	22	19
123789-HxCDF	278	255	263	265	12	4
123678-HxCDF	253	280	297	310	24	21
*234678-HxCDF	209	193	199	200	8	4
*1234678-HpCDF	4165	4171	4219	4185	29	1

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[illegible]=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218296

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41159-11-11
EXTRACT: 3

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	156	173	175	168	10	6
TOTAL PnCDF	2080	2087	2084	2084	4	0
TOTAL HxCDF	7276	7041	7058	7125	131	2
TOTAL HpCDF	11581	10545	10751	10959	548	5
*OCDF	28338	29448	26629	28138	1420	5
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF) (2348-TCDF)	38	40	40	39	1	3
*(12378-PnCDF) (12348-PnCDF)	998	1014	1043	1018	23	2
*23478-PnCDF	110	112	116	113	3	3
*123478-HxCDF	4893	4748	4754	4798	82	2
123479-HxCDF	120	114	120	118	3	3
123789-HxCDF	274	261	271	269	7	3
123678-HxCDF	310	303	303	305	4	1
*234678-HxCDF	199	200	201	200	1	1
*1234678-HpCDF	4347	4015	4153	4172	167	4

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(),()=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218297

AVERAGE POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE EXTRACTIONS

SAMPLE: 41159-11-12

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	67	100	107	91	21	23
TOTAL PnCDF	312	272	314	299	23	8
TOTAL HxCDF	998	879	872	916	71	8
TOTAL HpCDF	2392	2352	2247	2330	75	3
+OCDF	6690	5295	5740	5904	710	12
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
+2378-TCDD	<3	<3	<3	<3	NA	NA
+(2378-TCDF)	27	32	35	31	4	13
(2348-TCDF)						
+(12378-PnCDF)	119	101	105*	108	10	9
(12348-PnCDF)						
+23478-PnCDF	36	36	39	37	2	5
+123478-HxCDF	662	514	537	571	90	14
123479-HxCDF	10	16	16	14	4	27
123789-HxCDF	37	39	37	38	1	3
123678-HxCDF	37	45	29	37	8	21
+234678-HxCDF	33	36	39	32	8	26
+1234678-HpCDF	582	671	650	668	16	2

+ PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(1,2,3,4)=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.
* PAIR RESOLVED IN THIS EXTRACT. 12378-PnCDF=16 ng/g, 12348-PnCDF=87 ng/g.

ALL DATA IN UNITS OF NG/G.

MONS 218298

POLYCHLORODIBENZOFURAN ISOMER SEPARATION RESULTS

SAMPLE: 41159-11-12

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	97	140	107	91	21	23
TOTAL PnCDF	312	272	314	299	23	8
TOTAL HxCDF	998	879	872	916	71	8
TOTAL HpCDF	2292	2352	2247	2330	75	3
OCDF	6690	5203	5740	5964	718	12
TOTAL TCDD	43	43	43	43	NA	NA
OCDD	210	210	210	210	NA	NA
COMPOUNDS						
*2378-TCDD	43	43	3	3	NA	NA
*(2378-TCDF)	19	19	18	18	1	7
(2340-TCDF)	8	8	9	9	0	0
*(12378-PnCDF)	119	101	14	79	55	70
(12348-PnCDF)			97			
*23478-PnCDF	36	36	39	37	2	5
*123478-HxCDF	662	514	537	571	80	14
(23479-HxCDF	16	16	15	14	4	27
(23789-HxCDF	37	29	37	28	1	3
123678-HxCDF	37	45	29	37	8	21
*23467B-HxCDF	23	36	29	32	8	26
*1234678-HpCDF	652	671	650	668	10	2

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(:=PAIR RESOLVED BY CHROMATOGRAPHY.
(:=PAIR NOT RESOLVED CONSISTENTLY BY CHROMATOGRAPHY.

SINGLE RESOLUTION OF ISOMER PAIRS SHOWN WITH THE AVERAGE VALUE FOR EACH EXTRACTION. ALL DATA IN UNITS OF NG G.

MONS 218299

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41159-(1-12
EXTRACT: 1

PCDF/PCDD		DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
	TOTAL TCDF	72	66	63	67	5	7
	TOTAL PnCDF	322	340	265	312	42	14
	TOTAL HxCDF	987	1098	919	998	85	9
	TOTAL HpCDF	2529	2580	2068	2392	333	14
	*OCDF	6110	7027	6326	6490	810	12
	TOTAL TCDD	<7	<3	<3	<3	NA	NA
	OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS							
	*2378-TCDD	<3	<3	<3	<3	NA	NA
	*(2378-TCDF)	26	30	24	27	3	11
	(2348-TCDF)						
	*((2378-PnCDF)	102	141	115	119	20	17
	(12348-PnCDF)						
	*23478-PnCDF	29	42	27	36	8	22
	*123478-HxCDF	643	755	669	662	65	10
	123479-HxCDF	6	13	8	10	3	30
	*123789-HxCDF	37	34	29	37	3	7
	123678-HxCDF	37	40	34	37	3	8
	*234678-HxCDF	23	28	18	23	5	22
	*1234678-HpCDF	708	745	565	603	101	15

* PRIMARY TYPES OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(),()=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218300

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 4115P-11-12
EXTRACT: 2

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	103	97	99	100	3	3
TOTAL PnCDF	271	266	280	272	7	3
TOTAL HxCDF	855	845	926	879	50	6
TOTAL HpCDF	2557	2274	2483	2552	125	5
*OCDF	5429	5226	5193	5283	120	2
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF)	33	33	30	32	2	5
12348-TCDF)						
*(12378-PnCDF)	96	102	105	101	5	5
(12348-PnCDF)						
*23478-PnCDF	35	37	35	36	1	3
*123478-HxCDF	511	514	518	514	4	1
123479-HxCDF	17	16	16	16	1	4
123789-HxCDF	38	37	41	39	2	5
123678-HxCDF	40	45	41	45	4	9
*234678-HxCDF	35	37	35	36	1	3
*1234678-HpCDF	e72	e74	768	e71	37	5

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(1,2)=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218301

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41159-11-12
EXTRACT: 3

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	105	89	128	107	20	18
TOTAL PnCDF	322	310	309	314	7	2
TOTAL HxCDF	960	921	834	872	45	5
TOTAL HpCDF	2244	2344	2154	2247	95	4
*OCDF	5764	5959	5496	5740	232	4
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*[2378-TCDF] [2368-TCDF]	32	31	44	35	6	16
*12378-PnCDF	18	18	13	16	3	18
12348-PnCDF	85	89	87	87	2	2
*23478-PnCDF	37	40	41	39	2	5
*[123478-HxCDF	514	545	531	537	26	5
[123479-HxCDF	16	16	17	16	1	4
123689-HxCDF	37	38	37	37	1	2
123678-HxCDF	34	34	20	29	8	28
*234678-HxCDF	37	41	38	39	2	5
*1234678-HpCDF	656	675	615	650	32	5

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[]=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218302

AVERAGE POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE EXTRACTIONS

SAMPLE: 41159-11-15

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	863	804	875	847	37	4
TOTAL PnCDF	266	319	330	305	34	11
TOTAL HxCDF	16	51	44	37	19	50
TOTAL HpCDF	<3	17	<3	NA	NA	NA
*OCDF	17	26	27	23	6	25
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF)	232	215	268	245	27	11
(2348-TCDF)						
*(42378-PnCDF)	5	6	6	6	1	15
(12348-PnCDF)	11	10	14	12	2	17
*23478-PnCDF	60	57	61	59	2	3
*123478-HxCDF	17	22	22	20	3	12
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	<3	6	<3	NA	NA	NA
123678-HxCDF	<3	6	<3	NA	NA	NA
*234678-HxCDF	<3	11	11	NA	NA	NA
*1234678-HpCDF	<3	9	<3	NA	NA	NA

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(),()=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218303

POLYCHLORODIBENZOFURAN ISOMER SEPARATION RESULTS

SAMPLE: 41159-11-13

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	863	804	873	847	37	4
TOTAL PnCDF	266	319	330	305	34	11
TOTAL HxCDF	16	51	44	37	19	50
TOTAL HpCDF	<3	17	<3	NA	NA	NA
*OCDF	17	26	27	23	6	25
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF)	21	27	17	22	5	23
(2348-TCDF)	66	65	74	68	6	9
*(12378-PnCDF)	5	5	6	6	1	15
(12348-PnCDF)	11	10	14	12	2	17
*23478-PnCDF	60	57	61	59	2	3
*123478-HxCDF	17	22	22	20	3	12
127478-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	<3	6	<3	NA	NA	NA
123678-HxCDF	3	6	<3	NA	NA	NA
*234678-HxCDF	<3	11	11	NA	NA	NA
*1234678-HpCDF	<3	8	<3	NA	NA	NA

* PRIMARY TYPES OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(),(),=PAIRS RESOLVED BY CHROMATOGRAPHY.

SINGLE RESOLUTION OF ISOMER PAIRS SHOWN WITH THE AVERAGE VALUE FOR EACH EXTRACTION. ALL DATA IN UNITS OF NG/G.

MONS 218304

POLYCHLORODIBENZO-P-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41159-11-13
EXTRACT: 1

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	812	975	902	863	57	11
TOTAL PnCDF	259	320	240	266	46	17
TOTAL HxCDF	29	15	13	16	4	23
TOTAL HpCDF	<3	<3	<3	<3	NA	NA
OCDF	23	17	10	17	7	39
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<19	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*[2378-TCDF]	227	277	251	252	25	10
[2348-TCDF]						
12378-PnCDF	4	4	6	5	1	25
12348-PnCDF*	11	12	11	11	1	5
*23478-PnCDF	60	66	53	60	7	11
*123478-HxCDF	19	17	16	17	2	9
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	3	<3	<3	<3	NA	NA
123678-HxCDF	3	<3	<3	<3	NA	NA
*234678-HxCDF	3	<3	<3	<3	NA	NA
*1234678-HpCDF	<3	<3	<3	<3	NA	NA

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
* CALCULATED FROM 40 ng/ml AND 150 ng/ml RESPONSE FACTORS ONLY.
[]=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218305

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41[illegible]-11-1C
EXTRACT: 2

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	702	827	883	804	93	12
TOTAL PnCDF	337	298	323	319	21	7
TOTAL HxCDF	62	48	44	51	9	18
TOTAL HpCDF	17	16	17	17	1	3
OCDF	27	25	27	26	1	4
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF]	204	214	228	215	12	6
[2248-TCDF]						
*12378-PnCDF	7	4	6	6	2	27
12348-PnCDF	9	10	10	10	1	6
*23478-PnCDF	57	56	58	57	1	2
*123478-HxCDF	22	21	22	22	1	3
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	6	5	5	6	2	24
123678-HxCDF	5	6	5	5	1	19
*234678-HxCDF	12	10	12	11	1	10
*1234678-HpCDF	7	9	8	8	1	8

* PRIMARY TIERS OF DATA. (I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[]=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218306

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41139-11-13
EXTRACT: 3

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	790	997	831	873	119	13
TOTAL PnCDF	307	324	359	330	27	8
TOTAL HxCDF	48	31	54	44	12	27
TOTAL HpCDF	<3	<3	10	NA	NA	NA
*OCDF	26	28	28	27	1	4
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF]	271	271	261	268	6	2
[2348-TCDF]						
*12378-PnCDF	7	7	5	6	1	19
12348-PnCDF	13	13	15	14	1	8
*23478-PnCDF	59	61	62	61	2	3
*123478-HxCDF	20	21	24	22	2	10
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	4	5	<3	NA	NA	NA
123678-HxCDF	4	<3	<3	NA	NA	NA
*234678-HxCDF	11	8	13	11	3	24
*1234678-HpCDF	<3	<3	10	NA	NA	NA

* PRIMARY TIERS OF DATA. I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[]=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218307

AVERAGE POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE EXTRACTIONS

SAMPLE: 41139-11-14

PCDF/PCDD		EXTR.1	EXTR.2	EXTR.3	AVE.	S.D.	C.V.
	TOTAL TCDF	860	866	899	892	17	2
	TOTAL PnCDF	10139	8878	9154	9584	666	7
	TOTAL HxCDF	9823	9563	9912	9733	147	2
	TOTAL HpCDF	2093	2022	2119	2048	56	3
	OCDF	192	193	218	201	15	7
	TOTAL TCDD	<3	<3	<3	<3	NA	NA
	OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS							
	*2378-TCDD	<3	<3	<3	<3	NA	NA
	*(2378-TCDF)	331	342	341	338	6	2
	(2348-TCDF)						
	*12378-PnCDF	1029	668	698	862	161	19
	(2348-PnCDF	396	352	288	345	54	16
	*23478-PnCDF	1555	1654	1729	1646	87	5
	*(123478-HxCDF	3926	3761	4051	3913	145	4
	123479-HxCDF	295	282	300	293	11	4
	(23789-HxCDF	1033	1078	1311	1175	85	7
	123678-HxCDF	841	753	692	762	75	10
	*234678-HxCDF	947	954	963	921	59	6
	*(1234678-HpCDF	652	680	701	678	25	4

* PRIMARY ITEMS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
()=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218308

POLYCHLORODIBENZOFURAN ISOMER SEPARATION RESULTS

SAMPLE: 41159-IE-14

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	880	866	899	882	17	2
TOTAL PnCDF	10139	8878	9134	9384	666	7
TOTAL HxCDF	9823	9563	9612	9733	147	2
TOTAL HpCDF	2001	2032	2110	2048	56	3
OCDF	192	193	216	201	15	7
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF)	150	139	128	139	11	8
(2348-TCDF)	56	90	97	74	23	45
*(12378-PnCDF)	1020	858	899	962	161	19
(12348-PnCDF)						
*23478-PnCDF	1555	1654	1729	1646	87	5
*123478-HxCDF	3926	3761	4051	3913	145	4
123479-HxCDF	295	282	303	293	11	4
123789-HxCDF	1235	1078	1211	1175	85	7
123678-HxCDF	841	753	692	762	75	10
*234678-HxCDF	947	954	963	921	59	6
*1234679-HpCDF	652	680	701	678	25	4

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(=PAIR RESOLVED BY CHROMATOGRAPHY. INTERFERENCE NOTED.
[=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

SINGLE RESOLUTION OF ISOMER PAIRS SHOWN WITH THE AVERAGE VALUE FOR EACH EXTRACTION. ALL DATA IN UNITS OF NG/G.

MONS 218309

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41159-11-14
EXTRACT: I

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	818	986	835	880	92	11
TOTAL PnCDF	13100	9983	7333	10139	2887	29
TOTAL HxCDF	9436	11187	8846	9823	1218	12
TOTAL HpCDF	2010	2384	1609	2001	388	19
OCDF	200	194	183	192	9	4
TOTAL TCDD	<3	<3	<3	<3	NA	NA
PCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF]	308	368	318	331	32	10
[2348-TCDF]						
*12378-PnCDF	1015	1149	896	1020	127	12
[2348-PnCDF	420	479	290	396	97	24
*23478-PnCDF	1803	1659	1204	1555	313	20
*123479-HxCDF	3849	4317	3613	3926	358	9
123479-HxCDF	278	363	243	295	62	21
[23789-HxCDF	1166	1432	1107	1235	173	14
123678-HxCDF	822	912	789	841	64	8
*234678-HxCDF	903	1068	871	947	106	11
*1234678-HpCDF	671	774	510	652	133	20

* PRIMARY TIERS OF DATA, T.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[]=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218310

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41159-11-14
EXTRACT: 2

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	845	851	901	866	31	4
TOTAL PnCDF	9050	8561	9024	8878	275	3
TOTAL HxCDF	9844	9961	8884	9563	591	6
TOTAL HpCDF	2218	2213	1606	2032	370	18
*OCDF	174	186	219	193	23	12
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*[2378-TCDF]	350	336	341	342	7	2
[2348-TCDF]						
*12378-PnCDF	930	926	691	868	157	18
12348-PnCDF	359	411	296	352	63	18
*23478-PnCDF	1674	1622	1657	1654	21	1
*123478-HxCDF	4128	4124	3032	3761	632	17
123479-HxCDF	294	303	249	282	29	10
123789-HxCDF	1244	1246	743	1078	290	27
123678-HxCDF	668	714	878	753	110	15
*234678-HxCDF	979	992	592	854	227	27
*1234678-HpCDF	729	759	542	680	120	18

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[]=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218311

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41159-11-14
EXTRACT: 3

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	915	879	904	899	18	2
TOTAL PnCDF	9159	9252	9010	9154	113	1
TOTAL HxCDF	9817	9905	9713	9812	96	1
TOTAL HpCDF	2151	2126	2063	2110	41	2
*OCDF	197	196	260	218	37	17
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*[2378-TCDF]	345	337	341	341	4	1
[2048-TCDF]						
*12378-PnCDF	662	679	754	698	49	7
12348-PnCDF	290	277	297	288	10	4
*23478-PnCDF	1712	1766	1706	1728	33	2
*[123478-HxCDF	4067	4136	3899	4051	144	4
123479-HxCDF	298	310	301	303	6	2
[123789-HxCDF	1220	1218	1193	1211	16	1
123678-HxCDF	662	599	814	692	111	16
*234678-HxCDF	936	972	949	953	20	2
*[1234678-HpCDF	707	705	691	701	9	1

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[]=PAIR NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218312

AVERAGE POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE EXTRACTIONS

SAMPLE: 41159-11-15

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	<3	<3	<3	<3	NA	NA
TOTAL PnCDF	<3	<3	<3	<3	NA	NA
TOTAL HxCDF	23	10	22	18	7	38
TOTAL HpCDF	24	32	50	39	19	27
*OCDF	107	58	97	87	26	30
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF)	<3	<3	<3	<3	NA	NA
(2348-TCDF)						
*(12378-PnCDF)	<3	<3	<3	<3	NA	NA
(12348-PnCDF)						
*23478-PnCDF	<3	<3	<3	<3	NA	NA
*123478-HxCDF	22	10	21	18	7	37
123479-HxCDF	<3	<3	3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	<3	<3	<3	<3	NA	NA
*234678-HxCDF	<3	<3	<3	<3	NA	NA
*1234678-HpCDF	24	15	24	21	5	25

* PRIMARY TIERS OF DATA. I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(),,,=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218313

POLYCHLORODIBENZOFURAN ISOMER SEPARATION RESULTS

SAMPLE: 41159-11-15

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	<3	<3	<3	<3	NA	NA
TOTAL PnCDF	<3	<3	<3	<3	NA	NA
TOTAL HxCDF	23	10	22	18	7	38
TOTAL HpCDF	34	32	50	39	10	27
*OCDF	107	58	97	87	26	30
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF]	<3	<3	<3	<3	NA	NA
[2248-TCDF]						
*(12378-PnCDF)	<3	<3	<3	<3	NA	NA
(12348-PnCDF)						
*23478-PnCDF	<3	<3	<3	<3	NA	NA
*123478-HxCDF	22	10	21	18	7	37
123479-HxCDF	<3	<3	<3	<3	NA	NA
[23789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	<3	<3	<3	<3	NA	NA
*234678-HxCDF	<3	<3	<3	<3	NA	NA
*1234678-HpCDF	24	15	24	21	5	25

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[]...=PAIRS NOT RESOLVED BY CHROMATOGRAPHY, NO SEPARATION ATTEMPTED AT THIS CONCENTRATION LEVEL.

SINGLE RESOLUTION OF ISOMER PAIRS SHOWN WITH THE AVERAGE VALUE FOR EACH EXTRACTION. ALL DATA IN UNITS OF NG/G.

MONS 218314

POLYCHLORODIBENZO-5-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41159-11-15
EXTRACT: 1

PCDF/PCDD	DET.1*	DET.2	DET.3	AVE.	S.D.	C.V.
TOTAL TCDF	<50	<3	<3	NA	NA	NA
TOTAL PnCDF	<50	<3	<3	NA	NA	NA
TOTAL HxCDF	34	14	21	23	10	44
TOTAL HpCDF	47	33	21	34	13	39
*OCDF	106	103	113	107	5	5
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF)	<20	<3	<3	NA	NA	NA
(2348-TCDF)						
*(12378-PnCDF)	<20	<3	3	NA	NA	NA
(12348-PnCDF)						
*23478-PnCDF	<20	<3	<3	NA	NA	NA
*123478-HxCDF	30	14[2]	21[2]	22	8	36
123479-HxCDF	<20	<3	3	NA	NA	NA
123789-HxCDF	<20	3	<3	NA	NA	NA
123678-HxCDF	<20	<3	<3	NA	NA	NA
*234678-HxCDF	<20	<3	<3	NA	NA	NA
*1234678-HpCDF	30	21[2]	21[2]	24	5	21

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(3,4)+PAIRS NOT RESOLVED BY CHROMATOGRAPHY.
* DETERMINATION MOST LIKELY CONTAMINATED BY CARRY-OVER.
[2] NO SECOND CONFIRMING ION DETECTED.

ALL DATA IN UNITS OF NG/G.

MONS 218315

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41159-11-15
EXTRACT: 1

PCDF.PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	<3	<3	<3	<3	NA	NA
TOTAL PnCDF	<3	<3	<3	<3	NA	NA
TOTAL HxCDF	10	11	10	10	1	6
TOTAL HpCDF	24	24	27	22	4	13
+OCDF	58	59	56	58	2	3
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
+2378-TCDD	<3	<3	<3	<3	NA	NA
+(2378-TCDF]	<3	<3	<3	<3	NA	NA
[2348-TCDF]						
+([2378-PnCDF)	<3	<3	<3	<3	NA	NA
(12348-PnCDF)						
+23478-PnCDF	<3	<3	<3	<3	NA	NA
+[123478-HxCDF	10	11	10*	10	1	6
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	<3	<3	<3	<3	NA	NA
+234678-HxCDF	<3	<3	<3	<3	NA	NA
+[1234a78-HpCDF'	14	15	16	15	1	7

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[1,2,3,4,6,7,8-HpCDF] NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218316

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 4113°-11-15
EXTRACT: 3

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	<3	<3	<3	<3	NA	NA
TOTAL PnCDF	<3	<3	<3	<3	NA	NA
TOTAL HxCDF	20	20	18	22	5	24
TOTAL HpCDF	45	51	55	50	5	10
*OCDF	92	100	100	97	5	5
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF]	<3	<3	<3	<3	NA	NA
[2348-TCDF]						
*(12378-PnCDF)	<3	<3	<3	<3	NA	NA
(12348-PnCDF)						
*23478-PnCDF	<3	<3	<3	<3	NA	NA
*123478-HxCDF	20	16	18	21	6	31
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	<3	<3	<3	<3	NA	NA
*234678-HxCDF	<3	<3	<3	<3	NA	NA
*1234678-HpCDF	22	24	27	24	3	10

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[3,7]=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218317

AVERAGE POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE EXTRACTIONS

SAMPLE: 41159-11-15

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	<3	<3	NA	<3	NA	NA
TOTAL PeCDF	<3	<3	<3	<3	NA	NA
TOTAL HxCDF	14	<3	NA	NA	NA	NA
TOTAL HpCDF	28	<3	<3	NA	NA	NA
+OCDF	94	28	<10	NA	NA	NA
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	12	<10	<10	NA	NA
COMPOUNDS						
+2378-TCDD	<3	<3	<3	<3	NA	NA
+(2378-TCDF) [2348-TCDF]	<3	<3	NA	NA	NA	NA
+(12378-PeCDF) (12348-PeCDF)	<3	<3	<3	<3	NA	NA
+23478-PeCDF	<3	<3	<3	<3	NA	NA
+123478-HxCDF	14	<3	6	NA	NA	NA
[123479-HxCDF	<3	<3	<3	<3	NA	NA
[123789-HxCDF	<3	<3	<3	<3	NA	NA
[123678-HxCDF	<3	<3	<3	<3	NA	NA
+234678-HxCDF	<3	<3	<3	<3	NA	NA
+1234678-HpCDF	11	<3	<3	NA	NA	NA

+ PRIMARY TIERS OF DATA, I.E., ORTA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[X,X]=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218318

POLYCHLORODIBENZOFURAN ISOMER SEPARATION RESULTS

SAMPLE: 41159-11-16

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	<3	<3	NA	<3	NA	NA
TOTAL PnCDF	<3	<3	<3	<3	NA	NA
TOTAL HxCDF	14	<7	NA	NA	NA	NA
TOTAL HpCDF	28	<3	<3	NA	NA	NA
OCDF	94	28	<10	NA	NA	NA
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	12	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF]	<3	<3	NA	NA	NA	NA
(2346-TCDF)						
*(12378-PnCDF)	<3	<3	<3	<3	NA	NA
(12348-PnCDF)						
*23478-PnCDF	<3	<3	<3	<3	NA	NA
*123478-HxCDF	14	<3	6	NA	NA	NA
123479-HxCDF	<3	<7	<3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	<3	<7	<3	<3	NA	NA
*234678-HxCDF	<3	<3	<3	<3	NA	NA
*1234678-HpCDF	11	<3	<3	NA	NA	NA

* PRIMARY PEAKS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(): PAIRS NOT RESOLVED BY CHROMATOGRAPHY. NO SEPARATION ATTEMPTED AT THIS CONCENTRATION LEVEL.

SINGLE RESOLUTION OF ISOMER PAIRS SHOWN WITH THE AVERAGE VALUE FOR EACH EXTRACTION. ALL DATA IN UNITS OF NG/G.

MONS 218319

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 43159-11-15
EXTRACT: 1

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	<3	<3	<3	3	NA	NA
TOTAL PnCDF	<3	<3	<3	<3	NA	NA
TOTAL HxCDF	21	9*	11*	14	6	47
TOTAL HpCDF	50	11*	24*	28	20	70
+OCDF	121	44	107	94	45	48
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	19	<10	<10	<10	NA	NA
COMPOUNDS						
+2378-TCDD	3	<3	<3	<3	NA	NA
+(12378-TCDF) (2348-TCDF)	3	<3	<3	<3	NA	NA
+(11378-PnCDF) (12348-PnCDF)	3	<3	<3	<3	NA	NA
+23478-PnCDF	<3	<3	<3	<3	NA	NA
+123478-HxCDF	21	9*	11*	14	6	47
123479-HxCDF	<3	<3	3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	<3	<3	<3	<3	NA	NA
+234678-HxCDF	3	3	3	3	NA	NA
+1234678-HpCDF	19	8*	11*	11	7	57

+ PRIMARY PEAKS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[illegible]=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.
* NO SECOND CONFIRMING ION DETECTED.

ALL DATA IN UNITS OF NG/S.

MONS 218320

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41159-11-1
EXTRACT: T

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	<3	<3	<3	<3	NA	NA
TOTAL PnCDF	<3	<3	<3	<3	NA	NA
TOTAL HxCDF	<3	<3	<3	<3	NA	NA
TOTAL HpCDF	<3	<3	<3	<3	NA	NA
OCDF	22	26	27	28	3	27
TOTAL TCDD	<2	<2	<2	<2	NA	NA
OCDD	11	13	12	12	1	9
COMPOUNDS						
2378-TCDD	2	2	<2	<2	NA	NA
*(2378-TCDF)	<3	<3	<3	<3	NA	NA
(2348-TCDF)						
*(12378-PnCDF)	<2	<2	<2	<2	NA	NA
(12348-PnCDF)						
*23478-PnCDF	3	<3	<3	<3	NA	NA
*123478-HxCDF	2	<3	<3	<3	NA	NA
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	3	<3	<3	<3	NA	NA
123678-HxCDF	<3	<3	<3	<3	NA	NA
*234678-HxCDF	2	<2	<2	<2	NA	NA
*1234678-HpCDF	3	<3	<3	<3	NA	NA

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(I,J)=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218321

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41159-[]-16
EXTRACT: 3

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	<3	7	<3	NA	NA	NA
TOTAL PnCDF	<3	<3	<3	<3	NA	NA
TOTAL HxCDF	8	<3	<3	NA	NA	NA
TOTAL HpCDF	<3	<3	<3	<3	NA	NA
+OCDF	<10	<10	<10	<10	NA	NA
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
+2378-TCDD	<3	<3	<3	<3	NA	NA
+(2378-TCDF)	<3	7	<3	NA	NA	NA
(2348-TCDF)						
+((2378-PnCDF)	<3	<3	<3	<3	NA	NA
(12348-PnCDF)						
+23478-PnCDF	<3	<3	<3	<3	NA	NA
+123478-HxCDF	8	5	5	5	2	29
123479-HxCDF	<3	<3	<3	<3	NA	NA
123789-HxCDF	<3	<3	<3	<3	NA	NA
123678-HxCDF	<3	<3	<3	<3	NA	NA
+234678-HxCDF	<3	<3	<3	<3	NA	NA
+1234678-HpCDF	<3	<3	<3	<3	NA	NA

+ PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED
WITHIN T-E PROGRAM.
(),[]=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218322

AVERAGE POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE EXTRACTIONS

SAMPLE: 41159-11-17

PCDF/PCDD	EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
TOTAL TCDF	501	422	514	479	50	10
TOTAL PnCDF	1328	1177	1239	1248	76	6
TOTAL HxCDF	2291	2323	2365	2313	58	2
TOTAL HpCDF	2178	2567	2300	2350	220	9
OCDF	4981	599	4334	3205	2446	76
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
2378-TCDD	<3	<3	<3	<3	NA	NA
*(2378-TCDF)	190	171	184	182	10	5
(2348-TCDF)						
*(12379-PnCDF)	225	267	223*	305	33	11
(12348-PnCDF)						
*23478-PnCDF	203	197	190	196	7	3
*123478-HxCDF	1349	1330	1356	1345	13	1
123479-HxCDF	58	56	64	63	4	7
123789-HxCDF	60	67	58	65	5	7
123678-HxCDF	57	93	69	70	13	18
*234678-HxCDF	105	109	109	108	3	2
*1234678-HpCDF	1032	1230	1152	1138	100	9

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[illegible] ISOMERS NOT RESOLVED BY CHROMATOGRAPHY.
PAIR RESOLVED IN THIS EXTRACT. 12378-PnCDF=52 ng/g, 12348-PnCDF=271 ng/g.

ALL DATA IN UNITS OF NG/G.

MONS 218323

POLYCHLORODIBENZOFURAN ISOMER SEPARATION RESULTS

SAMPLE: 41159-11-17

PCDF/PCDD		EXTR.1	EXTR.2	EXTR.3	AVG.	S.D.	C.V.
	TOTAL TCDF	591	422	514	479	50	10
	TOTAL PnCDF	1320	1177	1239	1248	76	6
	TOTAL HxCDF	2251	2329	2345	2313	50	2
	TOTAL HpCDF	2128	2567	2280	2358	220	9
	*OCDF	4881	198	4334	3205	2446	76
	TOTAL TCDD	<3	<3	<3	<3	NA	NA
	OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS							
	*2378-TCDD	<3	<3	<3	<3	NA	NA
	*(2378-TCDF)	104	108	115	109	6	5
	(2348-TCDF)	37	36	31	35	3	9
	(12378-PnCDF)	55	52	48	52	4	7
	(12348-PnCDF)	260	261	271	271	18	7
	*23478-PnCDF	203	197	190	196	7	3
	*123478-HxCDF	1349	1330	1356	1345	13	1
	123479-HxCDF	58	66	54	63	6	7
	123789-HxCDF	60	67	60	65	5	7
	123678-HxCDF	57	83	69	70	15	18
	*234678-HxCDF	105	109	109	108	3	2
	*1234678-HpCDF	1032	1230	1152	1138	100	9

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[illegible] PAIRS RESOLVED BY CHROMATOGRAPHY.

SINGLE RESOLUTION OF ISOMER PAIRS SHOWN WITH THE AVERAGE VALUE FOR EACH EXTRACTION. ALL DATA IN UNITS OF NG/G.

MONS 218324

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41159-11-17
EXTRACT: 1

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	458	509	557	501	40	12
TOTAL PnCDF	1280	1508	1197	1328	161	12
TOTAL HxCDF	2242	2462	2049	2251	207	9
TOTAL HpCDF	2277	2048	1758	2128	322	15
OCDF	4857	5492	4295	4881	599	12
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*[2378-TCDF]	183	189	218	190	20	14
[2348-TCDF]						
*[12378-PnCDF]	300	358	307	325	37	12
[12348-PnCDF]						
*23478-PnCDF	297	245	156	393	45	22
*123478-HxCDF	1362	1481	1294	1349	129	10
123479-HxCDF	54	64	53	58	7	13
*123789-HxCDF	55	63	58	60	3	5
123678-HxCDF	52	57	53	57	5	8
*234678-HxCDF	99	111	104	105	6	6
*1234678-HpCDF	2094	1145	957	1922	154	15

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[X,Y]=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218325

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41159-11-17
EXTRACT: 2

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	462	383	419	421	40	9
TOTAL PnCDF	1228	1116	1080	1177	131	11
TOTAL HxCDF	2258	2362	2310	2323	20	1
TOTAL HpCDF	2411	2609	2680	2567	139	5
*OCDF	414	395	386	398	14	4
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
*2378-TCDD	<3	<3	<3	<3	NA	NA
*[2378-TCDF] [2348-TCDF]	184	164	166	171	11	6
*(12378-PnCDF) (12348-PnCDF)	316	218	266	267	49	18
*23478-PnCDF	204	186	201	197	10	5
*123478-HxCDF	1316	1247	1328	1330	16	1
123479-HxCDF	68	60	69	66	5	9
123789-HxCDF	56	69	67	67	2	2
123678-HxCDF	100	63	85	83	19	23
*234678-HxCDF	110	107	110	109	2	2
*1234678-HpCDF	1166	1242	1283	1230	59	5

* PRIMARY TIERS OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
[1,2]=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/G.

MONS 218326

POLYCHLORODIBENZO-p-DIOXIN AND POLYCHLORODIBENZOFURAN ANALYSIS RESULTS FOR TRIPLICATE DETERMINATIONS

SAMPLE: 41159-11-17
EXTRACT: 3

PCDF/PCDD	DET.1	DET.2	DET.3	AVG.	S.D.	C.V.
TOTAL TCDF	513	512	518	514	3	1
TOTAL PnCDF	1263	1119	1334	1239	110	9
TOTAL HxCDF	2360	2374	2360	2365	8	0
TOTAL HpCDF	2412	2362	2365	2380	28	1
+OCDF	4412	4354	4256	4334	90	2
TOTAL TCDD	<3	<3	<3	<3	NA	NA
OCDD	<10	<10	<10	<10	NA	NA
COMPOUNDS						
+2378-TCDD	<3	<3	<3	<3	NA	NA
+(2378-TCDF)	182	194	196	194	2	1
(2348-TCDF)						
+(12378-PnCDF)	55	52	48	52	4	7
(12348-PnCDF)	250	261	291	271	19	7
+23478-PnCDF	187	182	200	190	9	5
+123478-HxCDF	1340	1368	1361	1356	15	1
123479-HxCDF	66	64	63	64	2	3
123789-HxCDF	64	68	73	68	5	7
123675-HxCDF	70	75	63	69	6	9
+234678-HxCDF	109	103	115	109	6	6
+1234678-HpCDF	1173	1143	1141	1152	18	2

+ PRIMARY TYPES OF DATA, I.E., DATA FOR WHICH STANDARDS WERE SUPPLIED WITHIN THE PROGRAM.
(1,2)=PAIRS NOT RESOLVED BY CHROMATOGRAPHY.

ALL DATA IN UNITS OF NG/S.