



**ENVIRONMENTAL LABORATORIES, INC.**

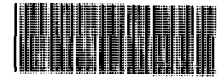
142 TEMPLE STREET  
NEW HAVEN, CT 06510  
(203) 789-1260  
FAX (203) 789-8261

**Superfund Records Center**

SITE: Raymark

BREAK: 19.4

OTHER: 464962



SDMS DocID

**464962**

February 8, 1993

Mr. Robert F. Moody  
Raymark Industries, Inc.  
75 East Main Street  
Stratford, Connecticut 06497

RE: PCB Samples  
Northern Fence Line  
ELI 91-545-14

Dear Mr. Moody:

Environmental Laboratories, Inc. (ELI) is pleased to present the results of the sampling collected on October 15, 1992. The goal of the sampling program was to confirm the EPA's sampling program conducted September 24, 1992.

The samples were collected from the stained areas west of the small building located along the northern property line. This location was previously remediated as the result of a capacitor oil spill. Three stained areas were sampled and analyzed for PCB's (see attached sketch).

The results indicate elevated levels of PCB-1242 were detected at Stained Area 3 at 9.420 ppm. PCB-1248 and 1268 were detected at Stained Area 2 at 65.4 and 1.15 ppm, respectively. PCB-1268 was detected at Stained Area 1 at 1.52 ppm.

These results indicate that PCB-1242, 1248 and 1268 were detected. The PCB detected represent two groups of PCB used and manufactured in the United States since 1970. The PCB-1242 and 1248 represent the largest group of PCB manufactured. This group includes PCB-1016, 1221, 1232, 1242, 1248, 1254 and 1260 and represents 98 percent of the PCB's sold in the United States. PCB-1268 and 1262 only represented 2 percent of the PCB's sold. The identification of PCB-1268 may represent an additive to another PCB oil.

ELI has also reviewed the analytical data for the Raybestos Memorial Ballfield. The results indicated elevated levels of PCB-1262 and 1268 (i.e. non-detected to 7,100 ppm PCB-1268). The PCB 1262 and 1268 appears to be the only PCB detected at the ballfield.



Mr. Robert F. Moody  
Raymark Industries, Inc.

- 2 -

February 8, 1993  
ELI 91-545-14

ELI has also reviewed the degradation pathway of PCB's. PCB's can be divided into three biodegradational groups:

They area:

- PCB-1221 and 1232 - biodegrade relatively rapidly
- PCB-1061 and 1242 - biodegrade slowly
- PCB-1248, 1254 and 1260 - are resistant to biodegradation
- PCB-1268 was not defined in the study reviewed

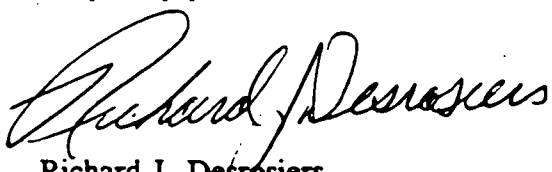
The results of our analysis indicate that the remediation effort previously completed did not remove all the soil stains and resultant PCB contamination or the staining occurred after the removal.

ELI recommends that the soil stains be excavated as part of a remediation effort. The stains appear isolated, however, no tests were conducted on the non-stained areas. The staining did not appear to be deep and the remediation may only require the excavation of the surface soil.

ELI recommends that additional tests be conducted after the remediation effort is complete. This will include all stained areas to a depth where staining does not occur.

We trust this information is sufficient for your use at this time. If you have any questions, please do not hesitate to contact us.

Very truly yours,



Richard J. Desrosiers  
Project Manager/Senior Hydrogeologist

RJD/jd  
026RL

cc: Mr. Mark Zimmerman, Esq. - Updike, Kelly and Spellacy, P.C.  
Mr. Robert G. Wasp, P.E. - Environmental Laboratories, Inc.

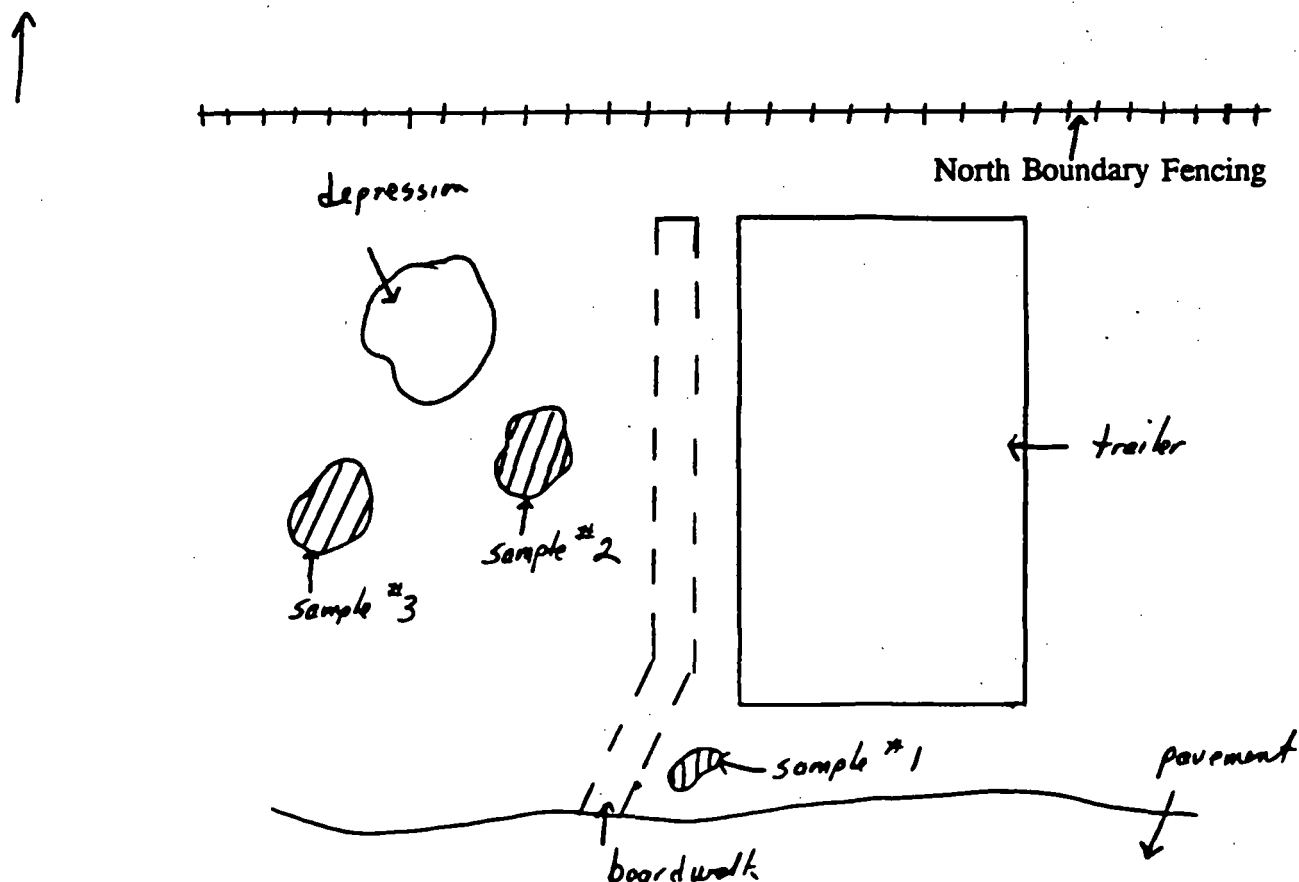
# PCB SAMPLE PROCEDURE

FIGURE 2

## SKETCH MAP - NOT TO SCALE

N

Sample Date: October 15, 1992



### SAMPLE LOCATIONS

- #1 - 1/2' diameter stained area.
- #2 - Reported transformer location.
- #3 - 1' diameter stained area.

DTW/gdp  
021RL/2



**NAME/ADDRESS:** Mr. Robert F. Moody  
Raymark Industries, Inc.  
75 East Main Street  
Stratford, CT 06497

**ELI PROJECT NUMBER:** 91-545-10

**ELI SAMPLE NUMBER(S):** 92-3017 - 92-3019

**COLLECTED BY:** ELI/DW

**DATE COLLECTED:** 10/15/92

**DATE RECEIVED:** 10/16/92

**COMPLETION DATE:** 12/2/92

**REPORT CERTIFIED BY:**

ELAINE A. CARINO  
LABORATORY DIRECTOR

92DECWK1.2

4





**RESULTS OF ANALYSIS:**Extraction Date: 11/18/92  
Analysis Date: 11/30-12/01/92**SAMPLE DESCRIPTION:****ELI SAMPLE NUMBER:**  
**PARAMETER****Stained\***  
**Soil #1**  
92-3017**Stained\*\***  
**Soil #2**  
92-3018**Stained\*\*\***  
**Soil #3**  
92-3019**Detection**  
**Limit****UNITS**

PCB-1016	ND	ND	ND	0.1	ppm
PCB-1221	ND	ND	ND	1.0	ppm
PCB-1232	ND	ND	ND	0.5	ppm
PCB-1242	ND	ND	9,420	0.1	ppm
PCB-1248	ND	65.4	ND	0.1	ppm
PCB-1254	ND	ND	ND	0.1	ppm
PCB-1260	ND	ND	ND	0.1	ppm
PCB-1268	1.52	1.15	ND	0.1	ppm

\* Detection Limit = 10x

\*\* Detection Limit = 50x

\*\*\* Detection Limit = 10,000x

ND = None Detected

BDL = Below Detection Limit





Mr. Robert F. Moody  
Raymark Industries, Inc.  
75 East Main St.  
Stratford, Ct. 06497

ELI PROJECT NO: 90-601-10  
COLLECTED BY: Client  
DATE RECEIVED: 11/5/90  
COMPLETION DATE: 11/7/90  
SAMPLE LOCATION: Stratford, Ct.

REPORT CERTIFIED BY:

*James P. McEntley*  
*for*  
*Elaine A. Carino*  
ELAINE A. CARINO  
LABORATORY DIRECTOR

**ENVIRONMENTAL LABORATORIES, INC.**

142 TEMPLE STREET  
NEW HAVEN, CT 06510  
(203) 789-1260  
FAX (203) 789-8261

November 30, 1990

Mr. Robert F. Moody  
Raymark Industries, Inc.  
75 East Main Street  
Stratford, CT 06497

Dear Mr. Moody:

Please find enclosed the analytical results that are in reference to ELI's contract #90-601-10.

If you should have any questions, please feel free to contact us at 371-0405.

Sincerely,



Marybeth C. Sawyer  
Administrative Assistant

Enclosure

12/3/90

Three Transformers located  
parallel to East Main St  
in the area between Bldg 6  
& East Main St.

**RECEIVED**

DEC 3 1990

RAYMARK INDUSTRIES, INC.

Laboratory:

The Cook Industrial Building 26-28 Beaver Street Ansonia, CT 06401 (203) 735-1808 FAX (203) 735-6590  
A MEMBER OF THE GIAVARA GROUP • BRIDGEPORT • NEW HAVEN • HARTFORD • ANSONIA • SAN JUAN, P.R.

**RESULTS OF ANALYSIS:** Transformer Oil

SAMPLE DESCRIPTION	Left Transformer	Center Transformer	Right Transformer	
ELI SAMPLE NUMBER:	90-2483	90-2484	90-2485	
PARAMETER				UNITS
PCB's	5.1	2.9	4.5	ppm

ND = None Detected

5041A/7

Mr. Robert Moody  
Raymark Industries  
75 East Main Street  
Stratford, CT 06497

ELI Project No.: 91-630-10

Collected By: ELI/J.M.

Date Received: 8-26-91

Sample Location: Raymark Industries  
75 East Main Street  
Stratford, CT 06497

Completion Date: 8-27-91

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REPORT CERTIFIED BY:

Elaine A. Carino (F.M.)  
ELAINE A. CARINO  
LABORATORY DIRECTOR

ENVIRONMENTAL LABORATORIES, INC.

**RESULTS OF ANALYSIS:**

**SAMPLE DESCRIPTION:**

TRANSFORMER OIL  
Building No.19  
West Side Trans

**ELI SAMPLE NUMBER:**

91-2667

**PARAMETER**

**UNITS**

PCB's

12.8

ppm

ND = None Detected

Mr. Robert Moody  
Raymark Industries  
75 East Main Street  
Stratford, CT 06497

ELI Project No.: 91-650-10

Collected By: ELI/J.M.

Date Received: 8-26-91

Sample Location: Raymark Industries  
75 East Main Street  
Stratford, CT 06497

Completion Date: 8-27-91

-----  
REPORT CERTIFIED BY:

Elaine A. Carino (J.M.)  
ELAINE A. CARINO  
LABORATORY DIRECTOR

ENVIRONMENTAL LABORATORIES, INC.



RESULTS OF ANALYSIS:

SAMPLE DESCRIPTION:

TRANSFORMER OIL  
Building No.19  
West Side Trans

- Laboratory  
Bldg. Transf. loc.  
in alleyway -

ELI SAMPLE NUMBER:

91-2667

Transformer removed  
by Richard

PARAMETER

UNITS

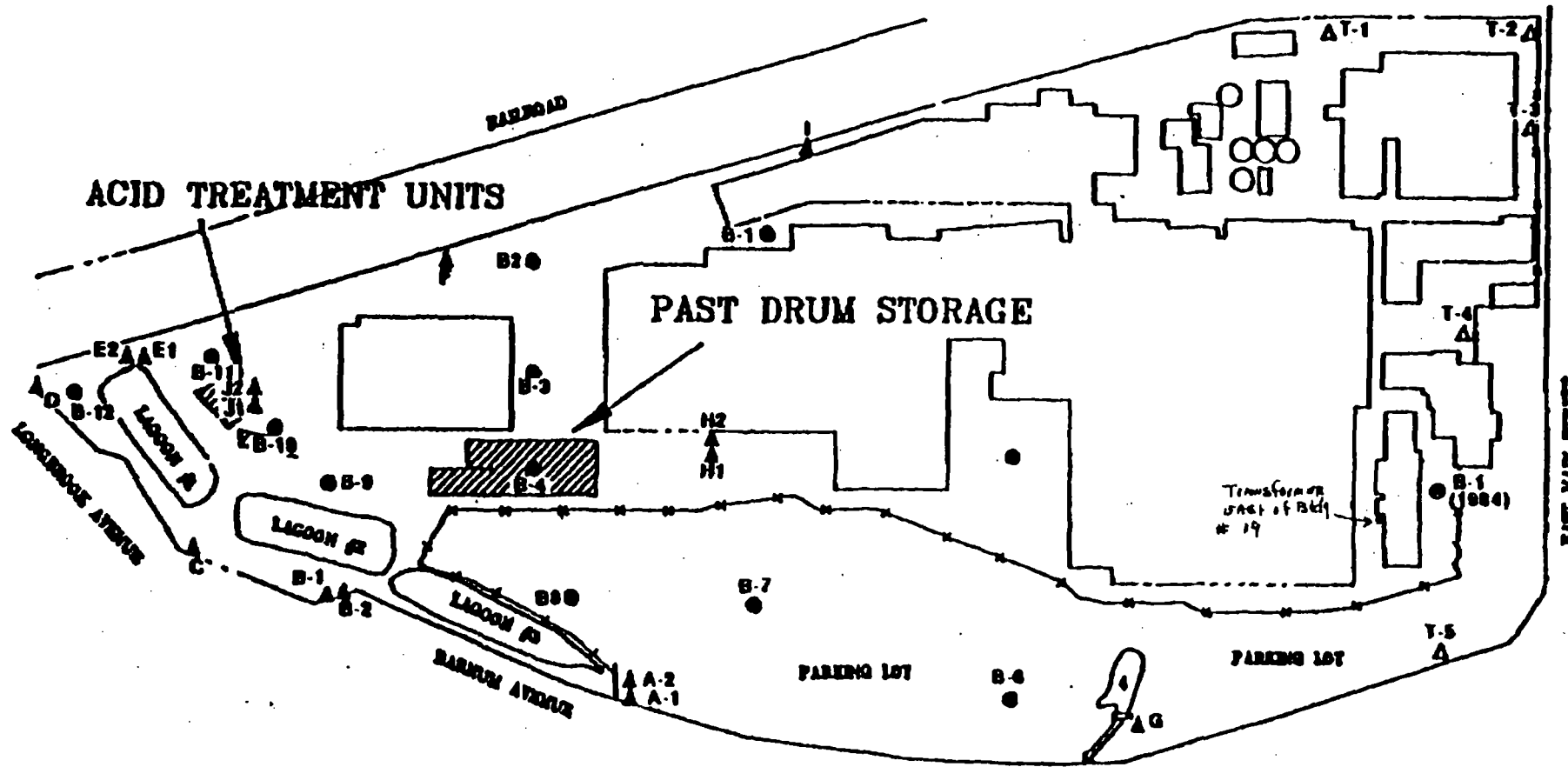
PCB's

12.8

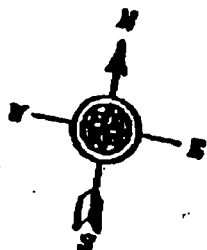
ppm

*Jim took*

ND = None Detected



DRAFT



**LEGEND**


- ▲ - MONITOR WELL LOCATIONS
- - TEST BORING LOCATIONS

0 300  
SCALE IN FEET

SOURCE: 1988 DRAFT PRELIMINARY ASSESSMENT REPORT

**RAYMARK INDUSTRIES, INC.  
STRATFORD, CONNECTICUT**

**SOIL BORING AND MONITOR WELL LOCATION MAP**

DATE	REVISED	PREPARED BY:
2/11/90	Transformer west of Bldg #19 added	 <p>LEGGETT, BRASS, HEARS &amp; GRAHAM, INC. Professional Ground Water Consultants 77 Danbury Road Wilton, CT 06097 203 747 1807</p>
		DATE 7/31/90 FIG. 1



ENVIRONMENTAL LABORATORIES, INC.  
analytical testing and environmental sciences

142 Temple Street New Haven CT 06510 (203) 789-1280

Laboratory  
The Code Industries Building  
26-28 Beaver Street  
Providence, RI 02903  
(603) 735-1800  
FAX (603) 735-6555

Conn. Cert. #PM-045

RECEIVED  
AUG 28 1991  
RAYMARK INDUSTRIES, INC.

Mr. Robert Moody  
RAYMARK INDUSTRIES, INC.  
75 East Main Street  
Stratford, CT 06497

ELI PROJECT NUMBER: 91-650-00

COLLECTED BY: ELI/JM

DATE RECEIVED: 7/17/91

SAMPLE LOCATION: STRATFORD, CT

COMPLETION DATE: 7/25/91

REPORT CERTIFIED BY:

Elaine A. Carino (T.M.)  
ELAINE A. CARINO  
LABORATORY DIRECTOR

5066D/16

RESULTS OF ANALYSIS:

SAMPLE DESCRIPTION:	LAGOON 4 EAST SIDE COMPOSITE SEDIMENT	BUILDING NO. 19 WEST SIDE TRANSFORMER PAD COMPOSITE SOIL	
ELI SAMPLE NUMBER:	91-2136	91-2137	
PARAMETER			UNITS
PCB's "	15.4	18.3	ppm

ND = None Detected

Comments: " Based on net weight.

5066D/17



# ENVIRONMENTAL LABORATORIES, INC.

analytical testing and environmental sciences

142 Temple Street New Haven CT 06510 (203) 789-1200

## CHAIN-OF-CUSTODY RECORD

Laboratory  
The Cook Industrial Building  
26-28 Beaver Street  
Ansonia, CT 06401  
(203) 735-1808  
FAX (203) 735-6590

Conn. Cert. #PH-0450

PROJECT NAME:

Raymark

PROJECT LOCATION:

~~Redacted~~ Stratford, CT

PROJECT NUMBER:

91-650-10

SOURCE CODES:

W = Well

O = Outfall

WO = Waste Oil

RO = Run Off

B = Bottom Sediment

SW = Sewage

SG = Sludge

S = Soil

R = River/Stream

LF = Landfill

WW = Wastewater

L = Lake/Ocean

X = Other; Specify

ITEM NO.	SAMPLE I.D.	SOURCE CODE	CONTAINER				ANALYSIS REQUIRED	COMMENTS	TRANSFER NUMBER AND CHECK		
			NO.	TYPE	SIZE	PRESERVE			1	2	3
1	Lagoon 4 East Side Comp.	S	1	G	500 ml		Method 8080 (P.C.B.'s only)		✓		
2	Transfer Pond Comp. No.	↓	↓	↓	↓		" "		✓		

Container Codes: P = Plastic E = EPA VIAL G = Glass A = Amber Glass B = Bacteria Bottle  
Preservative Codes: I = Iced F = Filtered N = Nitric Acid (HNO<sub>3</sub>) H = Hydrochloric Acid (HCl) S = Sodium Hydroxide (NaOH) T = Sodium Thiosulfate P = Sulfuric Acid  
O = Other; Specify

SAMPLER'S SIGNATURE	AFFILIATION	DATE	TIME	TRNSF NO.	ITEM NO.	TRANSFERS RELINQUISHED BY	ACCEPTED BY	DATE	TIME
Juan R. Melendez	ELI	7/17/91	1:20 AM	1	1-2	Juan R. Melendez	Chas. C. Casin	7/17/91	12:30
ADDITIONAL COMMENTS:				2					
				3					



ENVIRONMENTAL LABORATORIES, INC.  
analytical testing and environmental sciences

142 Temple Street New Haven CT 06510 (203) 789-1260

Laboratory  
The Cook Industrial Building  
26-28 Beaver Street  
Ansonia, CT 06401  
(203) 735-1808  
FAX (203) 735-6560

Cert. Cert. #PA-2450

Mr. Robert Moody  
RAYMARK INDUSTRIES, INC.  
75 East Main Street  
Stratford, CT 06497

ELI PROJECT NUMBER: 91-650-00

COLLECTED BY: ELI/JM

DATE RECEIVED: 7/17/91

SAMPLE LOCATION: STRATFORD, CT

COMPLETION DATE: 7/25/91

---

REPORT CERTIFIED BY:

Elaine A. Carino (J.M.)  
ELAINE A. CARINO  
LABORATORY DIRECTOR

5066D/16

RESULTS OF ANALYSIS:

SAMPLE DESCRIPTION:	LAGOON 4 EAST SIDE COMPOSITE SEDIMENT	BUILDING NO. 19 WEST SIDE TRANSFORMER PAD COMPOSITE SOIL
ELI SAMPLE NUMBER:	91-2136	91-2137

PARAMETER			UNITS
PCB's	15.4	18.3	ppm

RCCP : 50 Limit  
EPA : 10 Limit

ND = None Detected

Comments: " Based on net weight.

5066D/17

December 11, 1992

Mr. Robert Moody  
Raymark Industries, Inc.  
75 East Main Street  
Stratford, CT 06497

RE: Raymark  
ELI #91-545-10

Dear Mr. Moody:

The following is a brief summary of the activities completed at your facility on October 15, 1992.

### INTRODUCTION

In accordance with your request, ELI's field geologist collected three (3) soil samples on October 15, 1992. These samples were collected from the reported transformer location (see Location Map) at the northern part of the property. The purpose of this sampling program exercise was to identify whether the soil stains present contained PCB's.

### SAMPLE PROCEDURE

1. To ensure that a representative soil samples was collected, ELI composited equal amounts of the stained soil from each location (see Figure 1).
2. Samples were composited at a depth of 0'-6", utilizing stainless steel spatulas.
3. Each sample was placed in a 950 cc amber jar with a teflon lid and placed in a cooler with ice pack to maintain sample integrity in accordance with the RCRA 3013 sampling protocol.
4. A chain-of-custody was filled out at each sampling location by the field geologist prior to being immediately transported to ELI's laboratory for analysis.





5. ELI utilized a cleaned stainless steel spatula at each sampling location. Upon completion of the sampling program the stainless steel spatulas were decontaminated with alconox and DI water.
6. In accordance with the RCRA 3013 Health and Safety Plan, ELI collected these samples in a modified D level of protection (personal protection clothing, no respirator).

### SAMPLE LOCATION

As depicted in the sketch map (Figure 2), each approximate sample point is identified and corresponds to the analytical results presented in this report.

### ANALYTICAL RESULTS

As noted in Table #1, all three (3) soil samples collected and submitted for analysis indicate the presence of PCB's at concentrations which exceed the State Action Level of 1 ppb (0.001 ppm).

TABLE #1				
	Stained Soil #1	Stained Soil #2	Stained Soil #3	State Action Level
PCB's	1.52 ppm	65.4 ppm	9,420 ppm	0.001 ppm

### CONCLUSIONS

The results of the analytical analysis indicates that Aroclor 1268 was detected at concentrations of 1.52 ppm, 65.4 ppm and 9,420 ppm. These concentrations exceed the Connecticut threshold for PCB to remain in place. The federal level is 50 ppm. Connecticut may allow a waiver up to \_\_\_\_\_ ppm prior to remedial actions.

It should be noted that EPA Region I has also tested this stained area and found similar results.

Very truly yours,

David T. Williams  
Geologist

Richard J. Desrosiers  
Project Manager/Senior Hydrogeologist

DTW/RJD/gdb  
021RL/2



ENVIRONMENTAL LABORATORIES, INC.  
analytical testing and environmental sciences

142 Temple Street New Haven CT 06510 (203) 789-1260

EPA #CT-025  
Conn. Cert. #PH-0450

**NAME/ADDRESS:** Mr. Robert F. Moody  
Raymark Industries, Inc.  
75 East Main Street  
Stratford, CT 06497

**ELI PROJECT NUMBER:** 91-545-10

**ELI SAMPLE NUMBER(S):** 93-0626 - 93-0631

**COLLECTED BY:** ELI/KB

**DATE COLLECTED:** 2/11/93 **DATE RECEIVED:** 2/11/93

**COMPLETION DATE:** 3/2/93

**REPORT CERTIFIED BY:**

ELAINE A. CARINO  
LABORATORY DIRECTOR

Raymark

**RESULTS OF ANALYSIS:****METHOD 808****ORGANOCHLORINE PESTICIDES & PCBs****EXTRACTION DATE: 2/17/93****BY: BS****ANALYSIS DATE: 2/18/93 ANALYST: MW****& 3/2/93****DETECTION  
LIMIT  
( $\mu\text{g}/\text{m}^2$ )****CONCENTRATION  
( $\mu\text{g}/\text{m}^2$ )**

<b>SAMPLE IDENTIFIER:</b>	<b>TP 376</b>	<b>TP 377-1</b>	<b>TP 379-1</b>	<b>TP 379-2</b>	
<b>ELI SAMPLE NUMBER:</b>	<b>93-0626</b>	<b>93-0627</b>	<b>93-0628</b>	<b>93-0629</b>	
PCB-1016	ND	ND	ND	ND	0.50
PCB-1221	ND	ND	ND	ND	10.
PCB-1232	ND	ND	ND	ND	1.0
PCB-1242	ND	ND	ND	ND	0.50
PCB-1248	ND	ND	ND	ND	0.50
PCB-1254	0.79	ND	1.43	0.97	0.50
PCB-1260	ND	ND	ND	ND	0.50
PCB-1268	1.34	ND	0.70	ND	0.25

ND = None Detected

BDL = Below Detection Limit

**RESULTS OF ANALYSIS:**  
**METHOD 8080**  
**ORGANOCHLORINE PESTICIDES & PCBs**

**EXTRACTION DATE: 2/17/93    BY: BS**  
**ANALYSIS DATE: 2/18/93 ANALYST: MW**  
**& 3/2/93**

SAMPLE IDENTIFIER: ELI SAMPLE NUMBER:	CONCENTRATION ( $\mu\text{g}/\text{m}^2$ )		DETECTION LIMIT ( $\mu\text{g}/\text{m}^2$ )
	TP 380 93-0630	FIELD BLANK 93-0631	
PCB-1016	ND	ND	0.50
PCB-1221	ND	ND	10.
PCB-1232	ND	ND	1.0
PCB-1242	ND	ND	0.50
PCB-1248	ND	ND	0.50
PCB-1254	ND	ND	0.50
PCB-1260	ND	ND	0.50
PCB-1268	ND	ND	0.25

ND = None Detected  
BDL = Below Detection Limit

# Chain of Custody

Project Name: Project Location: Project Number:							ANALYSIS REQUIRED															LAB ID				
STA. NO.	DATE	TIME	C O M P	G R A B	SAMPLE IDENTIFICATION NUMBER	# OF BOTTLES	BOTTLE TYPE	BOTTLE SIZE	SOURCE	PRESERVE																LAB ID
1	376	2/11	1059		✓	TP-376	1	g/mL	250	X	He	1														93-0626
2	377	2/11	1000		✓	TP-377-1	1	g/mL	250	X	He	1														93-0627
3	379	2/11	0947		✓	TP-379-1	1	g/mL	250	X	He	1														93-0628
4	379	2/11	1027		✓	TP-379-2	1	g/mL	250	X	He	1														93-0629
5	380	2/11	0911		✓	TP-380	1	g/mL	250	X	He	1														93-0630
6		2/11	1131		✓	Field Blank	1	g/mL	250		He	1														93-0631

SOURCE CODES: W = WELL O = OUTFALL T = TANK SO = SLUDGE S = SOIL  
L = LAGOON TR = TREATED X = OTHER \* - DEFINE

PRESERVATIVE CODES: N = NITRIC ACID (HNO<sub>3</sub>) S = SODIUM HYDROXIDE (NaOH)  
H = HYDROCHLORIC ACID (HCl) I = ICED F = FILTERED T = SODIUM THIOSULFATE  
P = SULFURIC ACID (H<sub>2</sub>SO<sub>4</sub>) He = Hexane

BOTTLE CODES: <sup>g-class</sup> glass  
E = AMBER EPA VIAL  
A = AMBER GLASS 1 LITRE

SAMPLER'S SIGNATURE PRINT:	AFFILIATION	DATE	TIME	TRNSF NO.	ITEM NO.	TRANSFERS RELINQUISHED BY	ACCEPTED BY	DATE	TIME
KARON BALLARD		2/11/93		1	1-6	Karon Ballard	Denise La Rosa	2/11/93	12 PM
SIGN:				2					
Karon Ballard				3					

REMARKS:

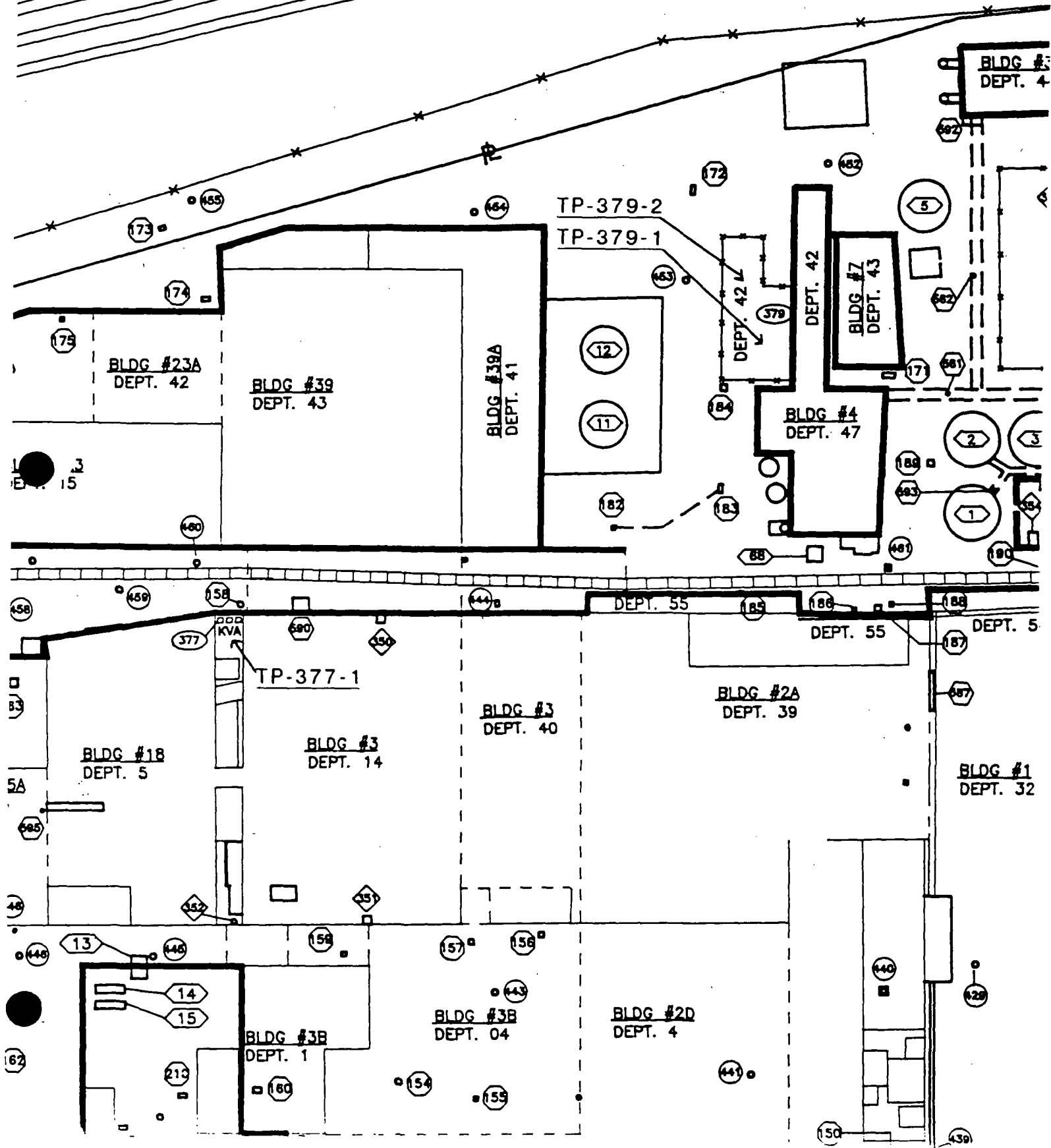
**analytical testing and environmental sciences**

**Conn. Cert. #PH-0450 EPA #CT-0**

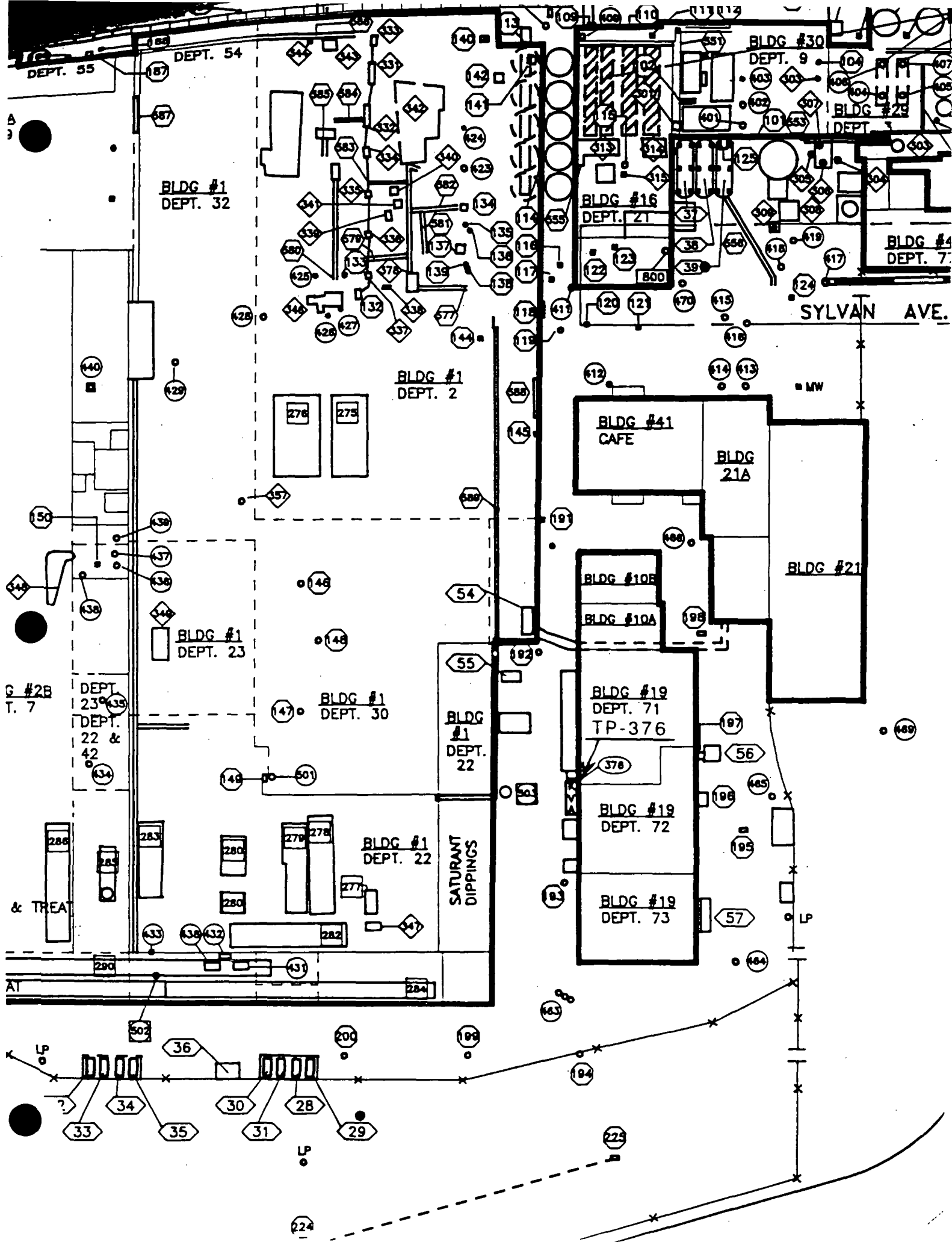
## Chain of Custody

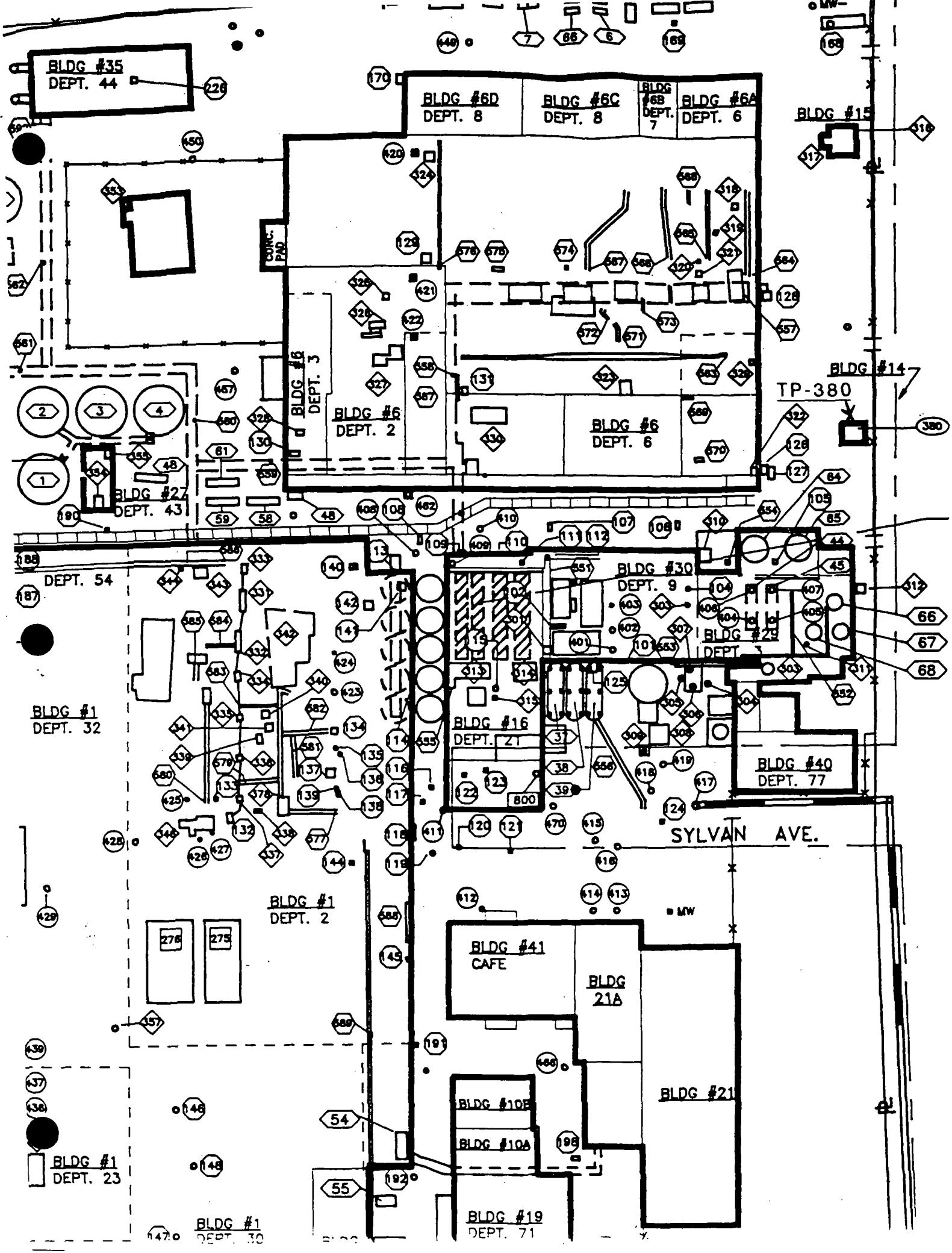
[illegible]

# METRO NORTH RAILROAD









BLDG #35  
DEPT. 44

BLDG #6D  
DEPT. 8

BLDG #6C  
DEPT. 8

BLDG #6B  
DEPT. 7

BLDG #6A  
DEPT. 6

BLDG #15

BLDG #6  
DEPT. 3

BLDG #6  
DEPT. 2

BLDG #6  
DEPT. 6

BLDG #14  
TP-380

BLDG #27  
DEPT. 43

DEPT. 54

BLDG #1  
DEPT. 32

BLDG #30  
DEPT. 9

BLDG #29  
DEPT. 5

BLDG #16  
DEPT. 21

BLDG #40  
DEPT. 77

BLDG #1  
DEPT. 2

BLDG #1  
DEPT. 23

BLDG #1  
DEPT. 30

BLDG #41  
CAFE

BLDG 21A

BLDG #10B

BLDG #10A

BLDG #19  
DEPT. 71

BLDG #21

SYLVAN AVE.

MW

DEVELOPMENT OF A GROUNDWATER  
MONITORING NETWORK

FOR

RAYBESTOS-MANHATTAN, INC.  
(INDUSTRIAL DIVISION)  
STRATFORD, CONNECTICUT

Thomas S. Czop  
Thomas S. Czop  
Field Engineer

Keith E. Warner  
Keith E. Warner, P.E.  
Project Manager

MARCH 1982

WORK ORDER NO. 01-6067

PREPARED BY

YORK WASTEWATER CONSULTANTS, INC.  
ONE RESEARCH DRIVE  
STAMFORD, CONNECTICUT

**RECEIVED**

APR 2 1982

HAZARDOUS MATERIALS  
MANAGEMENT UNIT

## 2.0 DEVELOPMENT OF THE GROUNDWATER MONITORING NETWORK

A groundwater monitoring network was developed to comply with RCRA requirements regarding the treatment of hazardous wastes. Observation wells were installed around the sedimentation ponds located in the Southwest corner of the Raybestos property. Permanent groundwater monitoring wells were subsequently installed upgradient and downgradient of the ponds.

### 2.1 Observation Well Installation

Since little was known about the four existing observation wells which had been previously installed (depth of screens, materials of construction, and depth to refusal), it was decided jointly by Raybestos and YWC to develop a monitoring network independently of any previous work that may have been done.

It was decided to place observation wells around all of the ponds so that any gradient in the groundwater elevation in the East-West direction could be measured by determining the relative heights of each well and the corresponding groundwater level.

Groundwater gradients in the North-South direction would be difficult to determine using piezometric methods because of the location of the ponds at the edge of the property. Observation wells could only be located right at the edge of the ponds on the South side. The North side of the ponds was not a good area for well installation because of heavy truck traffic, existing buildings, and the use of the area for storage of various materials. A "state-of-the-art" groundwater flow measurement meter was utilized to determine the groundwater flow at the North and South boundaries of the ponds. *det*

Nine new observation wells were installed around the ponds as listed in Table 1 and as shown in Figure 2.

Observation wells were bored with a hollow stem auger, and 30 ft. of Schedule 40, 2" diameter pipe was installed including 5 ft. of screened PVC at the end of the pipe. Approximately 25 ft. of pipe was installed at Well #10 because refusal was reached at that point. Steel pipes with caps were installed to encase the PVC pipe, protect it, and prevent foreign objects from getting into the well.

## 2.2 Groundwater Flow Direction Measurement

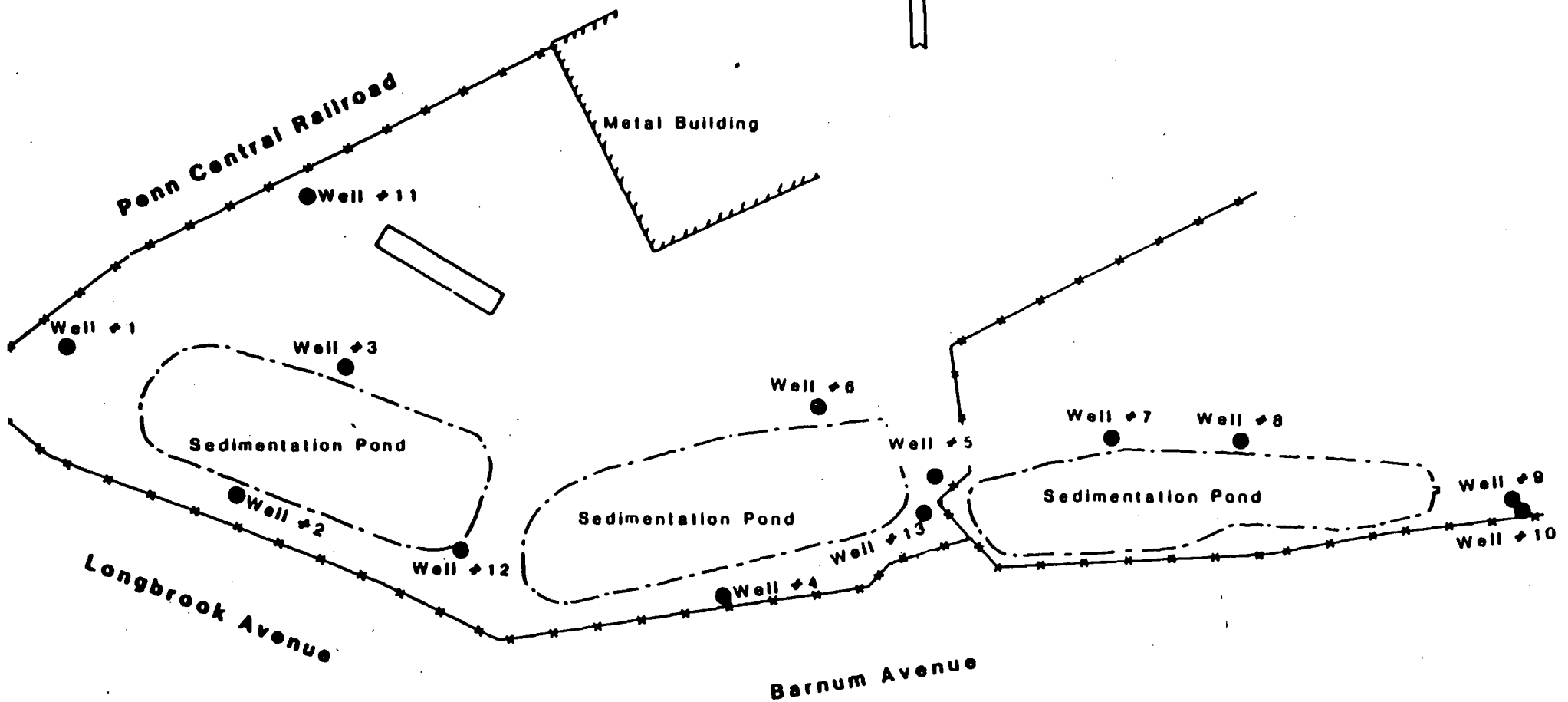
In addition to measurement of the groundwater gradient by piezometric means, the flow direction was measured with a K-V Model 30 groundwater flow meter. The device is especially suitable for areas where it is difficult to install observation wells long distances away from each other to measure the gradient.

The meter consists of a plastic cylindrical instrument that will fit in a 2" pipe. The instrument has eleven small metal probes on the bottom with one in the center and the other ten forming a circle. A nylon mesh "sock" or bag filled with gravel is fitted over the metallic probes. The central metallic probe generates heat for 27 seconds, and the other ten metallic probes (thermistors) measure small changes in temperature and indicate its magnitude with five vector components (pairs of the thermistors indicated magnitude). The five vectors are added to form a vector which shows the direction of the groundwater flow through the screen in the observation well.

**TABLE 1**  
**OBSERVATION WELL LOCATION AND ELEVATION**

<u>Well No.</u>	<u>Existing Or New</u>	<u>Location</u>	<u>Elevation Of Well Cap (Feet)</u>
1	New	West End	19.39
2	New	South End	18.99
3	New	North End	18.69
4	New	South End	16.11
5	Existing	North End	15.09
6	New	North End	16.57
7	New	North End	16.56
8	New	North End	15.82
9	Existing	East End	11.40
10	New	East End	12.02
11	Existing	North End	16.84
12	Existing	South End	17.97
13	New	South End	15.01

Figure 2  
Location of Observation  
Wells # 1-13



### 2.3 Results of Flow Investigations

The groundwater elevations measured in the new observation wells are presented in Table 2. Elevations were measured on November 10, 11, and 16. The wells are listed by number and are listed from West to East locations from the top to bottom of the table. There is clearly a downward gradient from West to East.

From Well #1 to Well #10, the elevation of the groundwater drops approximately 4 ft. (3.8 ft. on November 11, 4.3 ft. on November 16) over a distance of 660 ft. A definite gradient in the North-South direction could not be established from the groundwater elevation data. The North-South wells were only 90' apart and sometimes were very close to the ponds, which could have influenced groundwater elevations.

The groundwater elevation in Well #2 was always higher than #3, but the wells were really too close to each other, and to the pond to conclude that there is some flow in the Northerly direction. Wells #4 and #6 had groundwater elevations that were very close to each other, indicating that there is little flow in the North-South direction. It is noted that the water elevation in the second pond was low because it was not in use. The water level was probably about the same as the groundwater level and would have minimal influence, if any, on the elevations in wells #4 and #6.

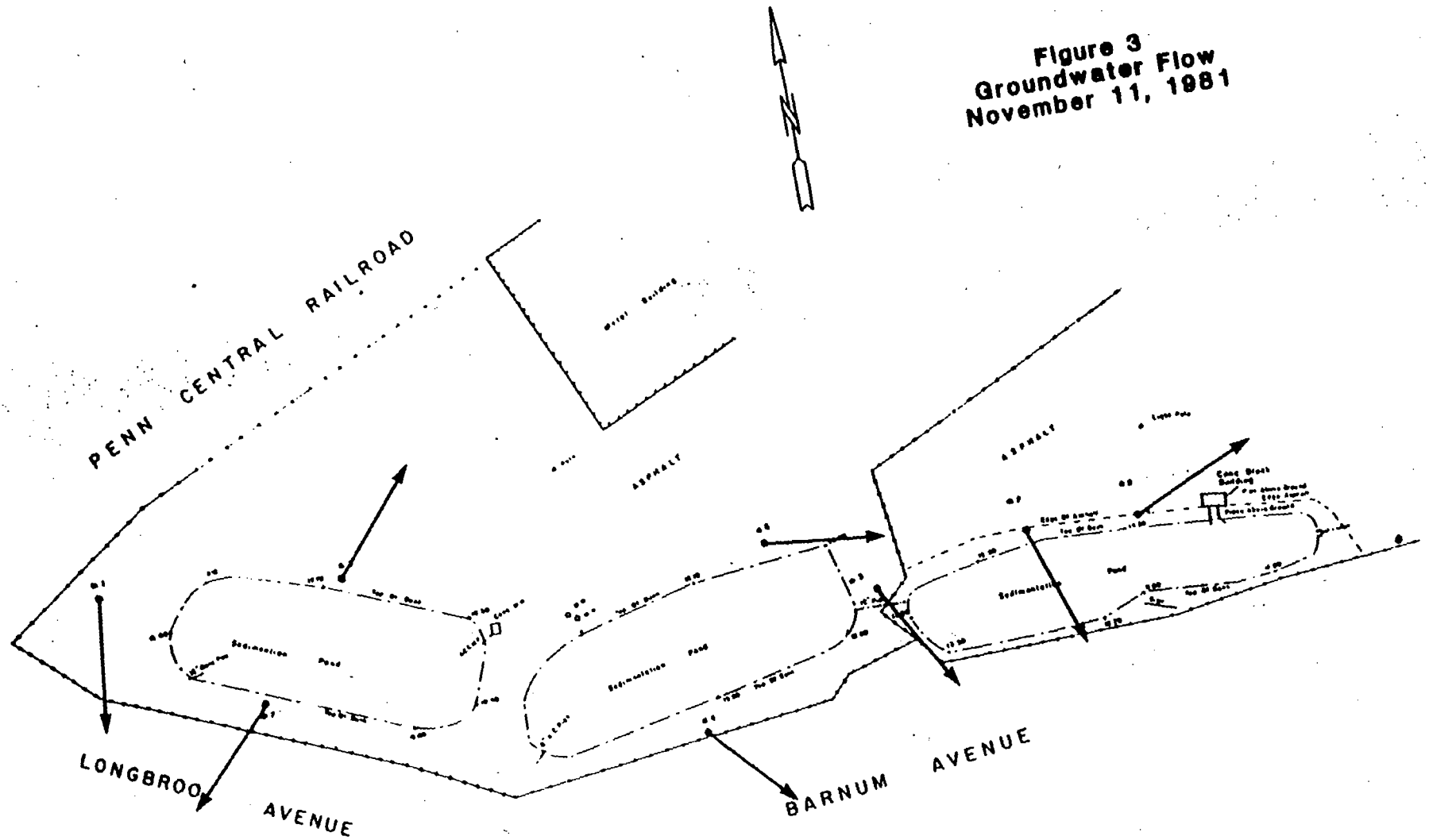
The K-V groundwater meter was used to determine flow direction independently of the elevation measurements. The meter was used on November 10, 11, and 16. Measurement on November 10th was a trial run to see what problems might be encountered on-site. The direction of groundwater flow at each well is shown in Figures 3 and 4 for



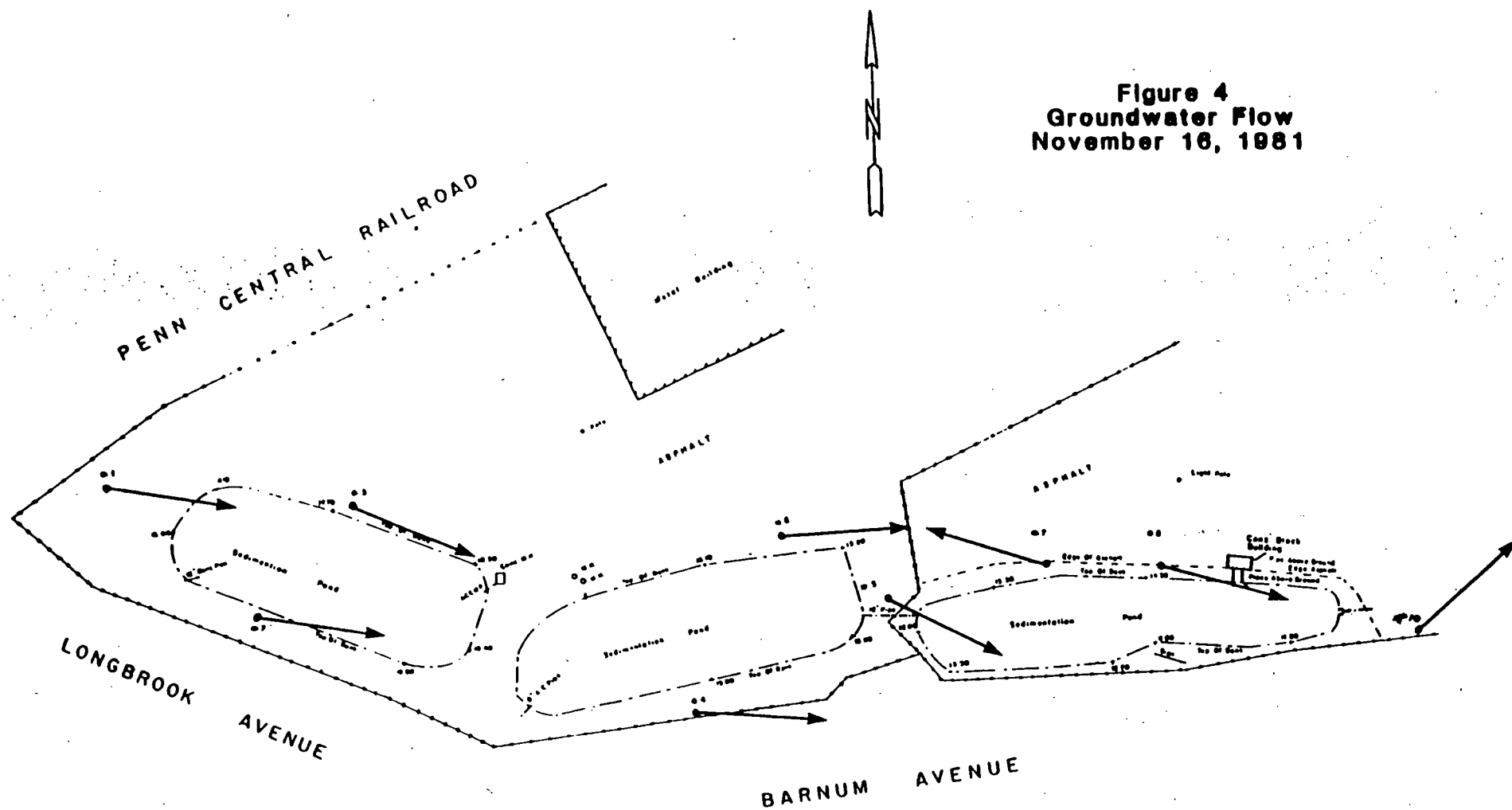
TABLE 2  
RAYBESTOS GROUNDWATER ELEVATIONS

<u>Well Number</u>	<u>Date</u>		
	<u>11/10 (Feet)</u>	<u>11/11 (Feet)</u>	<u>11/16 (Feet)</u>
1	7.51	7.56	7.31
2	7.82	7.66	7.57
3	6.94	7.52	6.94
4	5.82	6.07	6.19
13		5.51	5.26
6		6.12	5.82
7		5.23	5.31
8		4.99	3.99
10		3.77	3.02

Figure 3  
Groundwater Flow  
November 11, 1981



**Figure 4**  
**Groundwater Flow**  
**November 16, 1981**



November 11th and November 16th. Measurements on November 11th resulted in a somewhat scattered array of flow directions for the wells. The groundwater was generally flowing in a Southeast direction on November 11th. There was much less scatter on November 16th, and the groundwater flow was in an Easterly direction.

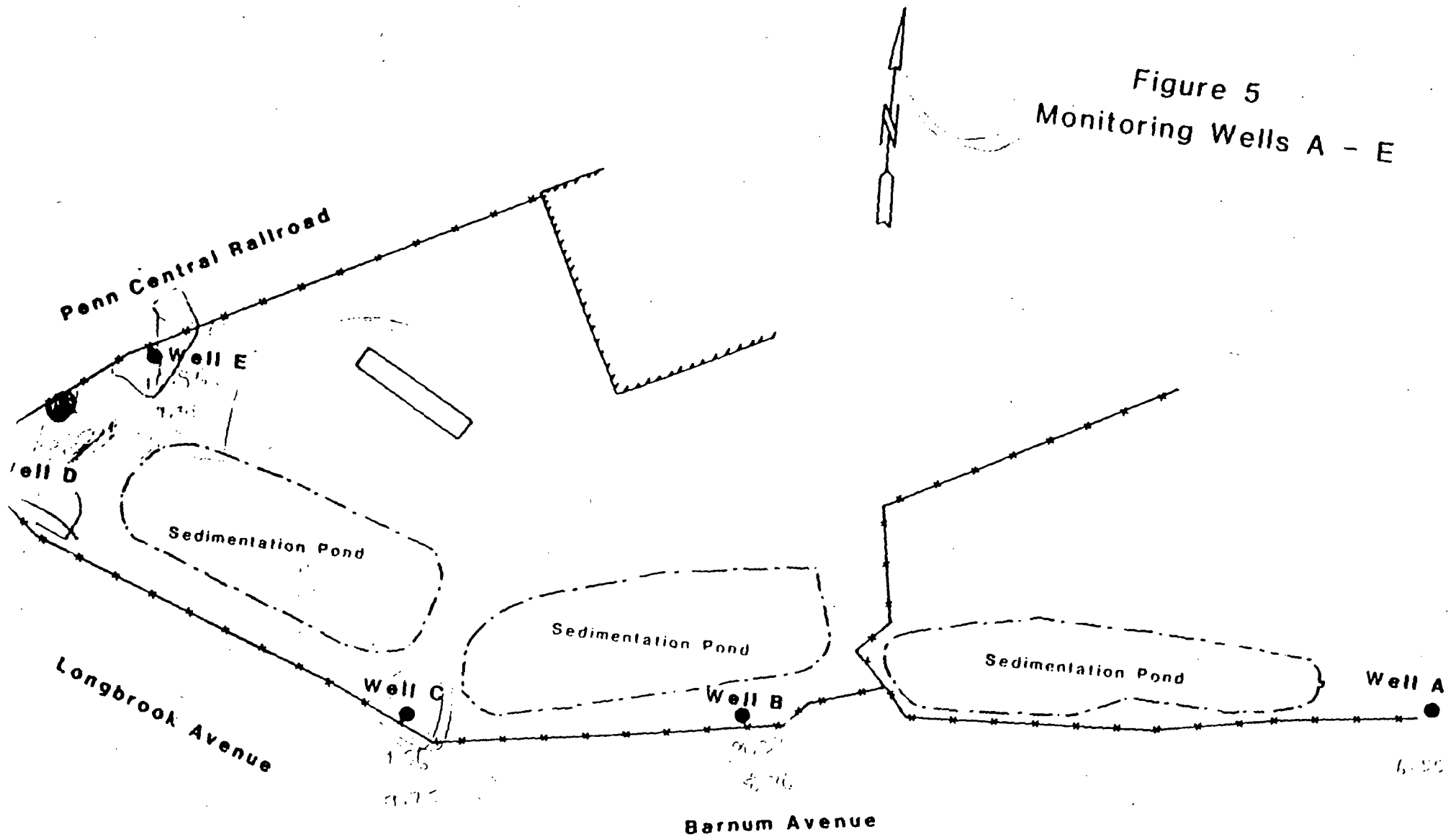
It may be concluded that the groundwater in the vicinity of the ponds at the Raybestos facility is flowing in an Easterly direction with the possibility of some flow in the Southeast direction. This is logical because an East to Southeast groundwater flow direction would go directly toward the Housatonic River.

#### 2.4 Placement Of Groundwater Monitoring Wells

Since the groundwater flows in an East to Southeast direction at the Raybestos facility, it was determined that upgradient and downgradient wells should be placed to intercept groundwater flowing toward the lagoons and away from the lagoons in any direction from East to Southeast. Figure 5 shows the location of the monitoring wells which were installed on February 22 and 23, 1982. Five monitoring wells were installed (2 upgradient and 3 downgradient) and they are labeled as letters A through E on Figure 5.

Well A has been installed to intercept any groundwater flow that could be flowing in an Easterly direction from sedimentation pond Nos. 1, 2, and 3. Monitoring Well B, which is located Southeast of Sedimentation Pond No. 2, has been installed to intercept groundwater flow moving in a Southeast direction from Sedimentation Pond No. 2. Observation Well C has been installed downgradient of Sedimentation Pond No. 1 and is placed to intercept any groundwater flow in a Southeasterly direction from that

Figure 5  
Monitoring Wells A - E



pond. Monitoring Well D is an upgradient well and has been installed to intercept any flow going in an Easterly direction from offsite toward the lagoons. Monitoring Well E is also an upgradient well which has been installed to intercept any flow entering the site in the South-easterly direction toward the sedimentation ponds. The physical description of the groundwater monitoring wells and their installation is described in the next section.

## 2.5 Physical Description Of Groundwater Monitoring Wells

Five monitoring wells were installed on February 22 and 23, 1982 in accordance with the Connecticut Department of Environmental Protection monitoring well specifications. Pertinent physical features of the installed monitoring wells are:

- well casings are 2" diameter schedule 80 PVC pipe;
- screens are PVC in 10' lengths;
- couplings are of threaded PVC;
- protective casings are made of steel, and are 3" in diameter and 5' long;
- steel casings have a non-lockable cap since all wells are within a security area of the plant;
- the annular area around the screens was backfilled with sand;
- the area around the well casing was backfilled with bentonite pellets;

- a concrete collar was installed around the steel protective casing;
- monitoring well holes were bored with hollow stem augers and PVC piping screen was installed with the auger still in the hole.

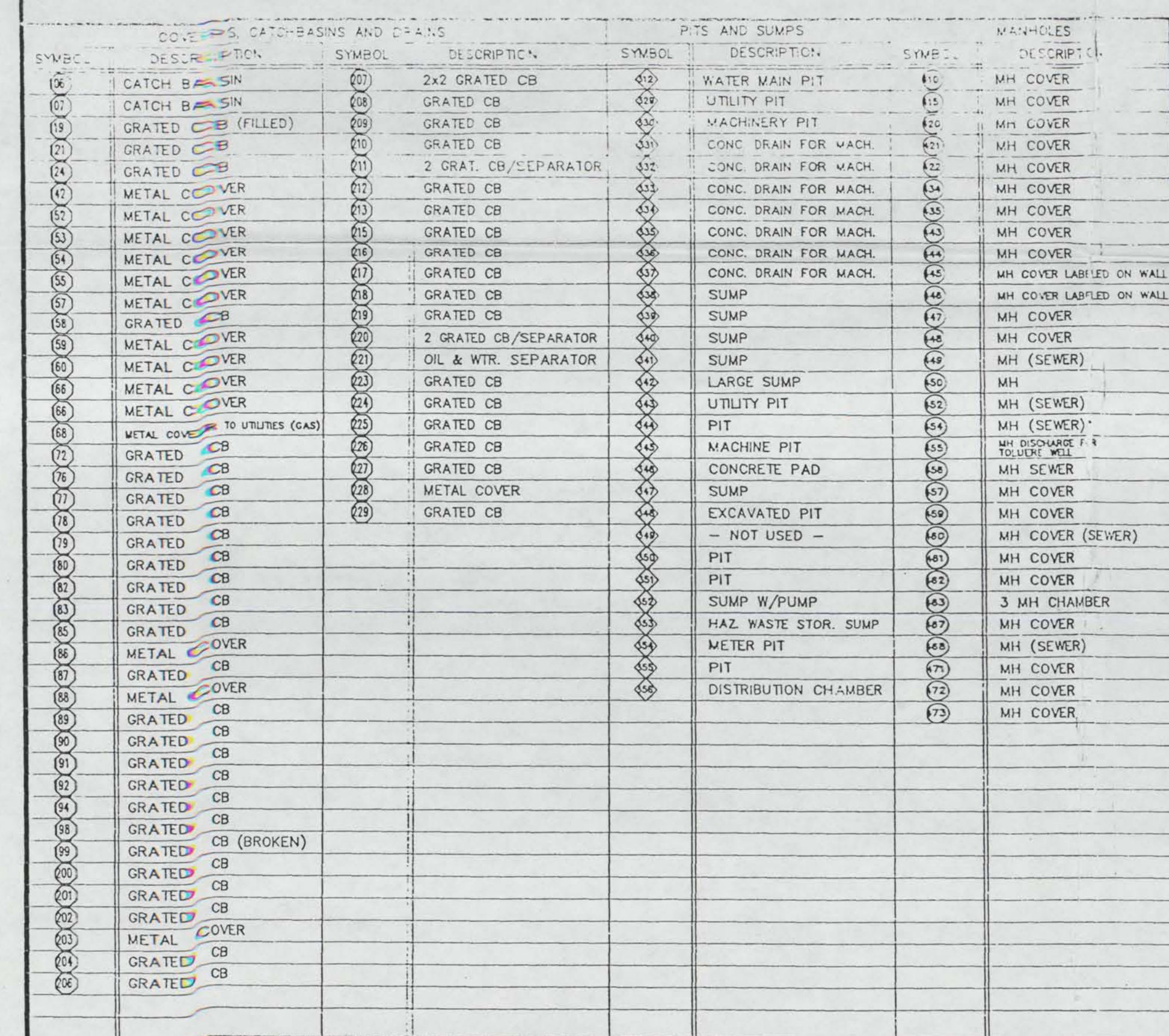
The lengths of PVC screen and pipe for each well are listed in Table 3. Photographs of pertinent aspects of the monitoring well installation are contained in Appendix A of this report.

TABLE 3  
LENGTHS OF PVC SCREEN AND PIPE

<u>Monitoring Well</u>	<u>Length Of PVC Screen</u>	<u><math>\Sigma \nabla</math></u>	<u>Top of Pipe</u>	<u>Length Of PVC Pipe</u>
A	10'	3.4'	12'	10'
B	10'	6'	16'	15'
C	10'	7.5'	18'	13.5'
D	10'	7'	19.4'	15'
E	10'	7'	19.4'	10'



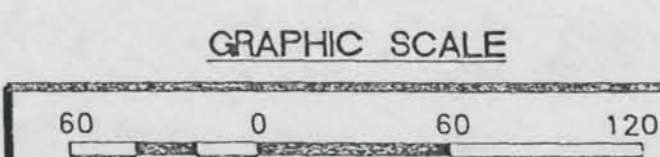
PRE-DIVERSION	
STATION 3 METER 4	
SUBCATCHMENT AREA <u>3</u>	
RUNOFF Q = <u>1.98</u> CFS = <u>88.9</u> GPM	
ANALYTE DETECTED	PRE-DIVERSION ppb
INORGANICS	
COPPER	142
LEAD	56.2
ZINC	245
SEMI VOLATILE	
2-NITROPHENOL	10 U
VOLATILE	
CHLOROETHANE	10 U
1,1-DICHLOROETHANE	42
1,1,1-TRICHLOROETHANE	200
TOLUENE	5 U
CHLOROBENZENE	10 U
XYLENE	5 U



POST-DIVERSION	
STATION 5	METER 5
SUBCATCHMENT AREA	5.6 + (1,2,3,4)
RUNOFF Q= 6.0 CFS	= 2,692 GPM
ANALYTE DETECTED	POST DIVERSION ppb
INORGANICS	
COPPER	50 U
LEAD	60.6 U
ZINC	82 U
SEMI VOLATILE	
2-NITROPHENOL	80
VOLATILE	
CHLOROETHANE	11
1,1-DICHLOROETHANE	13
1,1,1-TRICHLOROETHANE	51
TOLUENE	5 U
CHLOROBENZENE	10 U
XYLENE	5 U

**PIPING LEGEND**

ST-5	METER AND SAMPLING LOCATIONS
M-5	(SEE CHARTS FOR DATA)
----	HYDRAULICALLY CONNECTED, BUT ASSUMED PATHWAY (BLOCKAGE)
-----	HYDRAULICALLY CONNECTED
---ST---	STORM PIPING
---SAN---	SANITARY PIPING
--->---	DIRECTION OF FLOW
---X---	PIPE PLUGGED



# DRAINAGE SYSTEM AND SAMPLING PLAN

## FACILITY SITE PLAN

### STRATFORD, CT.

### RAYMARK INDUSTRIES INC.





**ENVIRONMENTAL LABORATORIES, INC.**

142 TEMPLE STREET  
NEW HAVEN, CT 06510  
(203) 789-1260  
FAX (203) 789-8261

April 25, 1994

Mr. Michael Hill  
U.S. Environmental Protection Agency  
Region I  
90 Canal Street, 3<sup>rd</sup> Floor  
Boston, Massachusetts 02203

RE: Hydrologic Runoff Analysis  
Pre- and Post-Stormwater Diversion  
ELI #93-563-15

Dear Mr. Hill:

Environmental Laboratories, Inc. (ELI) is submitting this hydrologic analysis of the drainage network at the Raymark facility. This analysis was initiated as part of the CERCLA Administrative Order Docket No. I-92-1072 and modified to accommodate a surface water diversion bypass around Lagoon 4 effectively isolating Lagoon 4. This bypass modification has resulted in additional sampling and analytical analyses testing of the surface water discharge.

The results indicate that the contaminate load discharge from the site is a result of the pipe network versus a specific source or spill. The original CERCLA order required the analyses of a SWMM model to evaluate potential contaminant loading to Ferry Creek from Raymark's point discharge to the municipal storm sewer under various storm events. Since the SWMM model is based upon contaminant loads for specific sources and concentration (load) within a subcatchment area, ELI requests a modification to the Order to remove the SWMM model from scope. Instead, ELI will provide a detailed analysis of the pipe network as it relates to contaminant load. This modification is based upon the analytical data collected in conjunction with the pre- and post-stormwater bypass around Lagoon 4.



Mr. Michael Hill  
U.S. Environmental Protection Agency  
Region I

- 2 -

April 25, 1994  
ELI #93-563-15

ELI is providing the following hydrologic analysis to support this position. The hydrologic analysis included:

- Drainage subcatchment areas based upon smoke testing performed at the facility.
- Subcatchment drainage area flow metering.
- Surface water sample collection under pre-diversion of surface water around Lagoon 4.
- Surface water sample collection under a post-diversion re-routed drainage network around Lagoon 4.
- Analytical analyses and this summary report.

This proposed modification would require Raymark to clean selective pipes to eliminate the contaminate load into the municipal storm sewer. To verify that the pipe cleaning has been effective, additional surface water samples will be collected for a specific target analyte list based upon the initial sampling.

We request that this plan be approved so that we can begin the pipe cleaning operation to mitigate the potential off-site migration of contaminated stormwater. If you have any questions, please do not hesitate to contact us.

Very truly yours,



Richard J. Desrosiers  
Project Manager

RJD/jd  
059RL

- c: Mr. Robert F. Moody - Raymark Industries, Inc.  
Mr. Mark Zimmerman, Esq. - Updike, Kelly and Spellacy, P.C.  
Mr. Robert G. Wasp, P.E. - Environmental Laboratories, Inc.



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**SURFACE WATER ANALYSIS**

**PURPOSE:**

The "hydrologic analysis and assessment of off-site contamination" study as outlined within the CERCLA Administrative Order, Docket No. I-92-1072, Attachment VIII required an analysis of the drainage pattern to characterize both the quantity and quality of the drainage discharges into and out of lagoon 4 and at any other identified off-site discharge locations. The scope of work outlined included:

- I. Rainfall-runoff analysis of the facility's drainage area using the Stormwater Management Model (SWMM) or equivalent;
- II. Determination of flows contributed by each subdrainage area(s) from the SWMM results;
- III. Flow and water quality measurements for chosen rainfall events at selected sampling locations to determine the sources of heaviest pollutant loads; and
- IV. Preparation of a report describing all of the results obtained from the SWMM and monitoring of the system.
- V. In addition, mitigating measures should be presented to eliminate the contaminated discharge.

This analysis will focus on the work conducted in conjunction with the completion of this phase of the "Order."

**WORK COMPLETED:**

In accordance with the "Order," Raymark has completed the following:

- I. A detailed drainage basin analysis to subdivide the site into distinct drainage subcatchment areas based upon: 1) visual drainage flow patterns, 2) site topography; and 3) results of smoke tests.
- II. A comprehensive smoke test was completed to determine the drainage pipe network at the facility. This included an evaluation of all pipes; catchbasins and manholes to determine stormwater drainage flow patterns. In addition, a smoke test was completed to evaluate all discharge points from the site and the discharge point from the site to Ferry Creek.

- III. Once the drainage patterns were determined and subcatchment areas defined, flow meters were established at the outfall of each subcatchment area. These meters were installed to monitor runoff in conjunction with sample collection.
- IV. Collection of surface water runoff samples at each of the metering station. These samples were collected and analyzed for full Appendix IX chemical constituents including dioxin/furans and asbestos.
- V. To eliminate the direct discharge from Lagoon 4 to the municipal storm sewer beneath Barnum Avenue Cut-Off, Raymark voluntarily constructed a stormwater diversion by-pass around Lagoon 4. This diversion connected the three points of discharge into Lagoon 4 to a new drainage by-pass pipe network avoiding direct stormwater discharge to the lagoon prior to its discharge off-site.
- VI. Once the diversion by-pass of Lagoon 4 was completed, an additional surface runoff sample was collected from the point where the stormwater discharges from the site. This sample was analyzed for the full Appendix IX constituents plus dioxin/furans and asbestos.
- VII. Analytical analyses of the samples and data entry of the samples into a database for tabular retrieval. A site plan as prepared showing sampling stations and contaminant loads. In addition, a flow analysis was completed using TR-55.
- VIII. This summary of the findings and presentation of mitigating measures to reduce the contaminant loads within the subcatchment areas.

#### SUBCATCHMENT AREAS

The subcatchment areas are presented on drawing RAY201. The site has been divided into nine subcatchment areas, based upon surface drainage patterns and flow through the storm drainage system. The subcatchment areas were defined as follows:

Smoke Test: was performed on the drainage pipe network. Smoke was forced into a manhole or catchbasin with a blower. The smoke exited catchbasins, grates, manholes, roof leaders and metal grates. These pathways were identified within each pipe network. If a blockage occurred, it was noted and smoke was injected at the net opening down from the blockage in an effort to delineate the network. This method tested all drainage pipes based upon the data provided within the RCRA Section 3013 Work Plan. All drainage pipes were located and the estimated flow direction has been identified on the attached plan (Drawing RAY201).

To facilitate the identification of the manholes, catchbasins and other metal grates, Raymark has uniquely labeled all covers, catchbasins, drains, etc. on-site for future reference. These features were surveyed and placed on the drawing to assist in the location of the drainage system.

A smoke test was completed to identify all discharge points to Lagoon 4 and from Lagoon 4 to Ferry Creek. This test confirmed that three pipe networks discharge to Lagoon 4. No other discharge locations from the site were identified. The smoke test from Lagoon 4 to Ferry Creek was incomplete due to a blockage within the municipal storm sewer just east of the Ware Chemical facility prohibited a complete pathway. The study did conclude that east of the blockage, smoke discharged at the outlet structure to Lagoon 4, and west of the blockage, smoke discharged the box culvert beneath I-95 which Ferry Creek flows. Thus, even though the blockage prevented smoke from discharging directly from Lagoon 4 to Ferry Creek, it is likely that surface water from the facility discharges to Ferry Creek. This blockage will prevent stormwater from flowing freely either from the Raymark facility or other upgradient discharge points within the municipal storm sewer.

Subcatchment Area 1 - Consists of 12.06 acres and drains the northerly property boundary from the railroad tracks and its drainage swale. The northerly portion drains overland to a central manhole (No. 461) located southeast of the "Boiler Room" then flows via pipe beneath Building 1 to an oil/water separator (No. 221) along with roof leaders which discharged to Lagoon 4. Currently, this drainage area was redirected around Lagoon 4 and the former discharge pipe has been sealed.

Subcatchment Area 2 - Consists of 2.75 acres and drains Building 2D Department 4, Building 3B Department 4, Building 3B Department 1; the westerly portion of the cure and treat along with the central courtyard and former acid neutralating platform. Surface runoff within the courtyard is directed east via catchbasins beneath Building 3B Departments 1 and 4 via drainage pipes. The roof leaders are connected to these drainage pipes and flows south to oil/water separator 220. Prior to the diversion water flowed south and discharged to Lagoon 4. Currently, the water has been diverted to Manhole 471 to the east and is diverted around Lagoon 4.

Subcatchment Area 3 - Consists of 6.34 acres and includes the central paved parking lot area. Prior to the closure of Settling Basins 1, 2 and 3, surface water from these basin flowed into this subcatchment area. Currently, overland flow from the pavement and Subcatchment Area 4 contributes to the discharge. Overland flow within this basin is collected at Catchbasins 229 and 219, and flow to a newly installed catchbasin in conjunction with the diversion by-pass. Formerly, the overland flow from Subcatchbasin 3 and 4 discharged directly to Lagoon 4.

Subcatchment Area 4 - Consists of 4.57 acres and drains the pavement areas south of the loading docks (Buildings 38 and 32A). In addition, roof leaders of Buildings 38, 32, 32A, 28, 22, 5A, 18, 44 and the southwesterly portion of Building 3 Department 14 are included in this drainage area. Only Building 44 and the passage way between 44 and 3B contain drainage pipes for roof leaders. Stormwater from the pavement and buildings is directed to oil/water separator 211 prior to flowing into Subcatchment Area 3.

Subcatchment Area 5 - Consists of 5.6 acres and includes the paved parking lot in the southeasterly portion of the facility, the runoff from East Main Street and the paved area around the cafeteria (Building 41), the three story brick buildings (Buildings 21 and 21A) and Buildings 10A, 10B and 19. All roof leaders are connected into a pipe network with catchbasins for overland flow which flows south from Building 19 to the fence line then west to the oil/water separator No. 221. The paved parking area runoff is collected in catchbasins and flows westerly to the diversion by-pass. Formerly, this subcatchment area drained directly to Lagoon 4.

Subcatchment Area 6 - Consists of 4.08 acres and includes Building 6, easterly portion of Building 1, Buildings 16, 30 and 40 along with the pavement between these building and along the northwesterly corner of Building 6. This subcatchment area has only a small runoff area. The smoke test did not define an outlet. It appears that the pipe network west of catchbasin 462 collapsed.

Subcatchment Area 7 - Consists of 1.57 acres and includes a portion of Building 43 and its associated pavement to the south. This drainage area is confined because the discharge pipe from Catchbasin 221 (assumed to flow previously to Settling Basin 2) has collapsed or is filled with sediment. This pipe has been permanently plugged. Thus, Subcatchment 7 may contribute to Subcatchments 3, 4 and 8.

Subcatchment Area 8 - Consists of 4.5 acres and includes Settling Basin 1, 2 and 3. This subcatchment area is isolated and surface runoff flows to the center of the basin and infiltrates.

Subcatchment Area 9 - Consists of 2.8 acres and includes the land north of Building 43. Surface runoff and roof leaders flows overland to the north. This subcatchment area does not contribute to the site.

### THE DRAINAGE PIPE NETWORK

Consists predominantly of three main drainage areas which contributed stormwater to Lagoon 4 prior to the diversion by-pass. These three drainage areas consist of:

Drainage Network 1 - Consists of 1,800± L.F. of drainage pipe divided into four subsections.

- Subsection 1A consists of the drainage pipes beneath Building 44 and collects roof leader runoff which drains to oil/water separator 211
- Subsection 1B consists of a drainage system which collects surface pavement runoff along the southerly portion of Buildings 38, 32A and 44 which drains to the oil/water separator number 211
- Subsection 1C consists of a drainage pipe which connects the oil/water separator 211 with Subsection 4 at Catchbasin 219

- Subsection 1D collects the surface runoff from the central parking lot area and flows along the southerly property boundary to the diversion by-pass. Previously, this drainage network discharged out on 24 inch RCP at the southwesterly portion of Lagoon 4. This drainage network drains Subcatchment Areas 3 and 4 comprising twenty-six percent (26%) of the site drainage area.

Drainage Network 2 - Consists of the smallest drainage network comprised of 850± L.F. of drainage pipe. This network predominantly drains the roof leaders from Buildings 3B and 3C along with a small pavement area south of Building 3C and the paved courtyard which includes three catchbasins (covered with pavement by the EPA). This network drains only Subcatchment Area 2 comprising seven percent (7%) of the site drainage area.

Drainage Network 3 - Consists of 2,100± L.F. drainage pipe divided into three subsections.

- Subsection 3A drains all the pavement area along the northerly property boundaries of Subcatchment Area 1. This includes the area adjacent to the "waste pile" and the pavement near solvent recovery, the boiler room and fuel oil tanks. Subsection 3A drains to Manhole 461. Subsection 3B initiates at Manhole 461 which receives Subsection 3A and redirects stormwater drainage south beneath Building 1 and then enters oil/water separator 221 northeast of Lagoon 4.
- Subsection 3C is the small reach between oil/water separator 211 which connects to the diversion by-pass at Manhole 471. Prior to the by-pass, Drainage Network 3 discharges to Lagoon 4. This drainage network comprises twenty-nine percent (29%) of the facility.

The remaining thirty-eight percent (38%) of the drainage area is divided into isolated drainage areas. These areas include eleven percent (11%) of the site which drains to the settling basins; thirteen percent (13%) which includes Subcatchment 5 with 1,100 L.F. of piping which previously was overland flow until the diversion connected clogged drainage pipes to the by-pass network; fourteen percent (14%) of the site drainage has no outlet and flows overland into other subcatchment areas.

## METERING

To assess the runoff from the site, five flow metering stations were established at points which would contribute to off-site flow. The meters were located at:

Meter 1 - Located at the oil/water separator No. 211 south of Building 44 and measured below from the upper reach of Drainage Network 1, Subcatchment Area 4.

Meter 2 - Located at the oil/water separator northwest of Lagoon 4 and measured flow from Drainage Network 2, Subcatchment Area 2.



**Meter 3** - Located at the oil/water separator northeast of lagoon and measured flow from Drainage Network 3, Subcatchment Area 1.

**Meter 4** - Located at the RCP in the southwesterly portion of Lagoon 4 and measured the flow from the pavement runoff contribution to Drainage Network 3 in Subcatchment Area 3.

**Meter 5** - Located at the outfall headwall and measured the total flow off-site. This meter station measures the surface runoff contribution to Lagoon 4 as an additive effect from Meters 1 through 4 from Subcatchment Area 5.

The metering instruments were installed on July 13, 1993 and were recorded continuously until August 11, 1993. During this time, two storm events occurred. A July 27, 1993 storm recorded a 0.55 inch storm event resulted in a 1.76 CFS peak flow rate from the site. On July 29, 1993 a 0.50 inch storm event recorded a 2.73 CFS peak flow rate at the outlet discharge structure (Meter 5).

The objective of the flow meters was to evaluate the rate of flow within each drainage basin and to establish a control to model the runoff/contaminant loads. Since the criteria established to collect a stormwater sample was a 0.5 inch storm event this objective was met on these dates and the instruments were removed. In addition to the flow meters, a contaminated rain gauge was used to determine the size of the storm event.

The flow metering indicated the following results during the sampling period:

Station Location	Metered Flow (CFS)	
	July 27, 1993	July 29, 1994
No. 1	1.11	0.99
No. 2	0.67	0.68
No. 3	1.78	2.44
No. 4	1.47	1.98
No. 5	1.76	2.74

**SAMPLING (Pre- and Post-Diversion By-Pass)**

The sampling program consisted of collecting runoff samples from the metered locations for full Appendix IX constituents plus dioxin/furans and asbestos. The samples were collected using a clean polyethylene dipper or grab sampler from the structures. The pre-diversion runoff sampling program consisted of five samples as proposed within the April 16, 1993 Work Plan.

The post-diversion runoff sampling program collected one sample at the point where surfaces discharged from the site. The sampling collection points are defined as follows:

Sample Station 1 - Was located at the northeasterly oil/water separator No. 221 (Meter Location 3). This station was only sampled under pre-diversion conditions. The sample was collected from the southerly catchbasin. The water level within the pipe was  $\frac{3}{4}$  full. The sample was collected directly below the surface at the outlet pipe.

Sample Station 2 - Was located at the northeasterly oil/water separator No. 220 (Meter Location 2). This station was only sampled under pre-diversion conditions. The sample was collected from the southerly catchbasin. Water was flowing freely through the pipe and the sample was collected directly below the surface at the out pipe.

Sample Station 3 - Was located at the twenty-four inch RCP discharge pipe located along the southwesterly bank of Lagoon 4 (Meter Location 4). This sample was collected only under the pre-diversion conditions. The lagoon water surface was observed two inches below the RCP. The sample was collected from the outlet structure as it flowed into the lagoon. A low flow was reported during sampling.

Sample Station 4 - Was located south of Building 44 at oil/water separator No. 211 (Meter Location 1). This sample was collected under the pre-diversion conditions. The water within the separator flowed freely. The sample was collected from the southerly catchbasin at the outlet.

Sample Station 5 - Was located at the discharge point from the site. This station was sampled under both pre- and post-diversion during storm events. The sample collected under the pre-diversion sampling, the sample was collected at the head wall within the drainage swale. The sample collected under the post-diversion construction the sample was collected from the junction box directly below the surface at the outlet structure.

The samples were collected during a storm event on:

pre-diversion construction	July 27, 1993	0.55 inch storm event
post-diversion construction	October 27, 1993	0.16 inch storm event

Upon completion of the stormwater sampling program the samples were placed on ice in accordance with EPA protocol. All samples were field preserved at the time of sample collection. All samples were shipped with chain-of-custody's and custody seals overnight to the appropriate laboratories to be analyzed under the Quality Assurance Project Plan (QAPP) for the Raymark facility.

## ANALYTICAL DATA

The results of the analytical data has gone through an extensive QA/QC and data validation evaluation. The results have been tabulated and are presented in Appendices A and B. The results indicate that the discharge from the Raymark facility meets drinking water MDL's for the chemical analytes analyzed. Only 1,1,1-Trichloroethane was reported above the MDL at Sampling Station 4 within the boundaries of the facility. Table I presents the chemical constituents detected within all sampling locations above the detection limit of the analytical instrumentation.

**TABLE I**

Analyte Detected	Station No. 1	Station No. 2	Station No. 3	Station No. 4	Station No. 5-Pre	Station No. 5-Post	MCL
Copper	462	336	142	160	194	50U	1,000*
Lead	111J	86J	56.2J	93.2J	81.6J	60.6U	20 <sup>1</sup>
Zinc	458	332	246	272	286	82U	5,000*
2-Nitrophenol	10U	10U	10U	10U	10U	806	—
Chloroethane	10U	4J	10U	14U	6U	11	—
1,1-Dichloroethane	3J	6	42	80	24	5U	—
1,1,1-Trichloroethane	14	19	200	460	93	51U	2,000
Toluene	9	5U	5U	7U	9	5U	2,000
Xylene (Total)	20	5U	5U	12U	2U	5U	440

Note 1) U - analyte not detected J - estimated value

2) all results presented in parts per billion (ppb)

\* secondary MCL

<sup>1</sup> CT drinking water standard

Bold indicates parameter exceeds MCL's

These results indicate that the data for 1,1,1-Trichloroethane exceeded the USEPA MCL standards at Station No. 4 however, the discharge from the site was below the MCL's. Lead was flagged as estimated values which exceed the Connecticut drinking water MCL. All other parameters analyzed were below MCL's.

The data indicates that the upper reach of pipe Network 1 contains volatile organic organics, specifically 1,1,1-Trichloroethane (TCA). This 950± linear feet of pipe contributes the volatile contaminate load to the discharge point. It appears that the 410 ppb concentration of TCA is diluted within Subcatchment Area 3 which reported a 200 ppb value of TCA at the twenty-four inch RCP discharge pipe. Thus, no additional source of TCA is apparent on-site.

The estimated lead levels reported exceed the Connecticut MCL's. This contaminate load is consistent across the site and can be contributed to the typical Raymark waste on-site. The sample collected during the post-diversion was collected for both dissolved and total lead. These results indicated a total lead concentration of 60.6 ppb and a dissolved concentration of 13.6 ppb. Thus, the lead concentration discharging off-site can be contributed to particulates in the surface water runoff. Thus, the lead load leaving the site is related to sediments and not dissolved lead.

### **MITIGATING MEASURES**

The results of this hydrologic analysis has indicated that the surface water discharge from the Raymark facility meets EPA and CT MCL's for drinking water except lead as total lead. Within the facility property boundaries, TCA exceeded MCL's at one location - Sampling Station No. 4. The levels of TCA were diluted through the on-site drainage network prior to the stormwater discharging the site. To ensure that this portion of the on-site drainage system does not impact off-site water quality in the future, Raymark proposes the following mitigating measures:

- I. Drainage Network 1, Subsection 1A (385 L.F.) and 1B (550 L.F.) will be cleaned to remove any residual contamination which may be present. This 935 L.F. of pipe will be jetted with clean water. The oil/water separator No. 211 will be temporarily blocked during this operation. A vacuum truck will be used to extract the jetted fluids at the oil/water separator No. 211. These fluids will be recycled during the jetting process. A final rinse will be completed once the sediments have been removed.
- II. The four oil/water separators will be cleaned where appropriate. These separators will act as sediment collecting sumps trapping particulates prior to discharge.

These mitigation measures will reduce contaminant and sediment loads from the Raymark facility. To ensure that the levels of contamination have been reduced, Raymark will collect surface water samples at the following locations after a 0.5 inch rain event.

Station 1 - Oil/water separator 211. This station will evaluate the contaminant load after the cleaning operation. These results will be compared to the July 27, 1993 data.

Station 3 - The newly installed catchbasin west of Lagoon 4. This reach was previously tested under pre-diversion and will evaluate the dilution effects of Subcatchment Area 3 on the drainage from the upper reach of Drainage Network 1.

Station 5 - Is the newly constructed junction box where all stormwater flows prior to leaving the site. The sampling point will be the outlet pipe.

The three samples collected will be analyzed for the target analytes detected above action levels. Specifically, Raymark will run:

- VOC's - SW846 Method 8240
- Metals - SW846 Method 6010

These three samples will fully evaluate the effects of the mitigative measures proposed. The results will be tabulate.

A report will be issued regarding the mitigative measures completed, sampling and analysis of results. This report will include a revised drawing and tables.

RJD/jd  
036RR

HATHAWAY STRICKLAND  
DOCKET NO 1-92-1072  
ASBESTOS ANALYSIS  
CERCLA STORMWATER (MF/L)

Sample ID	STATION#1	STATION#2	STATION#3	STATION#4	STATION#5	STATION#6	STATION 0-1	
Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Post-Diversion	
Units	MF/L	MF/L	MF/L	MF/L	MF/L	MF/L		
Date Sampled	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	10/27/93	
Remarks								
Laboratory	EE&G	EE&G	EE&G	EE&G	EE&G	EE&G	EE&G	
Asbestos High	31.67	22.2	152.92	167.97	106.55	141.83	190.36	
Asbestos Low	13.84	7.6	78.98	23.63	44.79	79.16	60.02	

J-Value is an estimated quantity.  
U-Analyte was not detected. Value is the sample detection limit.  
UJ-Analyte was not detected. Detection limit is estimated.  
R-Datum was rejected.

RAYMAR INDUSTRIES INC  
DOCKET NO I-92-1072  
DIOXIN/FURAN ANALYSIS  
CERCLA STORMWATER (NG/L)

Sample ID	STATION#1	STATION#2	STATION#3	STATION#4	STATION#5			
Diversal	Pre-Diversal	Pre-Diversal	Pre-Diversal	Pre-Diversal	Pre-Diversal			
Units	NG/L	NG/L	NG/L	NG/L	NG/L			
Date Sampled	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93			
Date Analyzed	8/2/93	8/3/93	8/3/93	8/11/93	8/3/93			
Remarks								
Laboratory	TRIANGLE LABS	TRIANGLE LABS	TRIANGLE LABS	TRIANGLE LABS	TRIANGLE LABS			
1,2,3,4,6,7,8-HpCDD	0.094 UJ	0.042 J	0.11 UJ	0.031 U	0.046 U			
1,2,3,4,6,7,8-HpCDF	0.063 UJ	0.0078 U	0.064 UJ	0.017 U	0.027 U			
1,2,3,4,7,8,9-HpCDF	0.067 UJ	0.0098 U	0.081 UJ	0.021 U	0.034 U			
1,2,3,4,7,8-HxCDD	0.064 UJ	0.01 U	0.076 UJ	0.021 U	0.031 U			
1,2,3,4,7,8-HxCDF	0.037 UJ	0.0055 U	0.045 UJ	0.012 U	0.019 U			
1,2,3,6,7,8-HxCDD	0.066 UJ	0.011 U	0.08 UJ	0.022 U	0.033 U			
1,2,3,6,7,8-HxCDF	0.035 UJ	0.0051 U	0.042 UJ	0.011 U	0.018 U			
1,2,3,7,8,9-HxCDD	0.06 UJ	0.0096 U	0.072 UJ	0.02 U	0.03 U			
1,2,3,7,8,9-HxCDF	0.044 UJ	0.0084 U	0.052 UJ	0.014 U	0.022 U			
1,2,3,7,8-PeCDD	0.087 UJ	0.015 U	0.082 UJ	0.032 U	0.027 U			
1,2,3,7,8-PeCDF	0.034 UJ	0.0073 U	0.042 UJ	0.017 U	0.013 U			
2,3,4,6,7,8-HxCDF	0.036 UJ	0.0053 U	0.044 UJ	0.011 U	0.018 U			
2,3,4,7,8-PeCDF	0.034 UJ	0.0074 U	0.042 UJ	0.017 U	0.014 U			
2,3,7,8-TCDD	0.033 UJ	0.0075 U	0.04 UJ	0.016 U	0.013 U			
2,3,7,8-TCDF	0.021 UJ	0.0046 U	0.026 UJ	0.011 U	0.0084 U			
OCDD	0.15 UJ	0.52 J	0.75 J	0.027 U	0.118 J			
OCDF	0.12 UJ	0.01 U	0.13 UJ	0.022 U	0.038 U			
TOTAL HpCDD	0.094 UJ	0.042 J	0.11 UJ	0.031 U	0.046 U			
TOTAL HpCDF	0.067 UJ	0.0181 J	0.081 UJ	0.021 U	0.034 U			
TOTAL HxCDD	0.066 UJ	0.011 U	0.08 UJ	0.022 U	0.033 U			
TOTAL HxCDF	0.044 UJ	0.0064 U	0.052 UJ	0.014 U	0.022 U			
TOTAL PeCDD	0.087 UJ	0.015 U	0.082 UJ	0.032 U	0.027 U			
TOTAL PeCDF	0.034 UJ	0.0074 U	0.042 UJ	0.017 U	0.014 U			
TOTAL TCDD	0.033 UJ	0.0075 U	0.04 UJ	0.016 U	0.013 U			
TOTAL TCDF	0.021 UJ	0.0046 U	0.026 UJ	0.011 U	0.0084 U			

J-Value is an estimated quantity.  
U-Analyte was not detected. Value is the sample detection limit.  
UJ-Analyte was not detected. Detection limit is estimated.  
R-Datum was rejected.

RAYMAR INDUSTRIES INC  
DOCKET NO I-82-1072  
CHLORINATED HERBICIDE ANALYSIS  
CERCLA STORMWATER (UG/L)

Sample ID	STATION#1	STATION#2	STATION#3	STATION#4	STATION#5	STATION#6	SW-DISCHARGE	SW-DISCHARGER
Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Post-Diversion	Post-Diversion
Units	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L
Date Sampled	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	10/27/93	10/27/93
Date Analyzed	8/23/93	8/23/93	8/23/93	8/23/93	8/23/93	8/23/93	11/18/93	12/13/93
Remarks						Dup of STATION#5		Reanalysis
Laboratory	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC
2,4-D	0.84 U	0.94 U	0.94 U	1.1 U	0.94 U	0.84 U	0.94 U	0.94 U
2,4,5-T	0.095 U	0.095 U	0.095 U	0.11 U	0.095 U	0.095 U	0.095 U	0.095 U
SILVEX	0.095 U	0.095 U	0.095 U	0.11 U	0.095 U	0.095 U	0.095 U	0.095 U

J-Value is an estimated quantity.  
U-Analyte was not detected. Value is the sample detection limit.  
UJ-Analyte was not detected. Detection limit is estimated.  
R-Datum was rejected.



RAYMA INDUSTRIES INC  
DOCKET NO 1-92-1072  
INORGANIC ANALYSIS  
CERCLA STORMWATER (UG/L)

Sample ID	STATION#1	STATION#2	STATION#3	STATION#4	STATION#5	STATION#6	STATION 0-1 (T)	STATION 0-1 (D)
Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Post-Diversion	Post-Diversion
Units	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L
Date Sampled	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	10/27/93	10/27/93
Remarks						Dup of STATION#5		
Laboratory	ELI	ELI	ELI	ELI	ELI	ELI	ELI	ELI
Calcium	NR	NR	NR	NR	NR	NR	NR	NR
Cyanide	NR	NR	NR	NR	NR	NR	NR	NR
Iron	NR	NR	NR	NR	NR	NR	NR	NR
Magnesium	NR	NR	NR	NR	NR	NR	NR	NR
Manganese	NR	NR	NR	NR	NR	NR	NR	NR
Potassium	NR	NR	NR	NR	NR	NR	NR	NR
Sodium	NR	NR	NR	NR	NR	NR	NR	NR
Aluminum	NR	NR	NR	NR	NR	NR	NR	NR
Antimony	5 UJ	5 UJ	5 UJ	5 UJ	5 UJ	5 UJ	5 U	50 U
Arsenic	40 UJ	40 UJ	40 UJ	40 UJ	40 UJ	40 UJ	40 U	20 U
Barium	200 U	200 U	200 U	200 U	200 U	200 U	200 U	138 U
Beryllium	10 U	10 U	10 U	10 U	10 U	10 U	10 U	5 U
Cadmium	10 U	10 U	10 U	10 U	10 U	10 U	10 U	5 U
Chromium	20 U	20 U	20 U	20 U	20 U	20 U	20 U	10 U
Cobalt	100 U	100 U	100 U	100 U	100 U	100 U	100 U	50 U
Copper	482	338	142	180	194	170	50 U	27 U
Lead	111 J	86 J	56.2 J	93.2 J	81.6 J	69.6 J	60.6 U	13.6 U
Mercury	0.5 UJ	0.5 UJ	0.5 UJ	0.5 UJ	0.5 UJ	0.5 UJ	0.5 U	0.5 U
Nickel	80 U	80 U	80 U	80 U	80 U	80 U	80 U	40 U
Selenium	10 UJ	10 UJ	10 UJ	10 UJ	10 UJ	10 UJ	10 U	5 U
Silver	R	R	R	R	R	R	20 U	10 U
Thallium	10 UJ	10 UJ	10 UJ	10 UJ	10 UJ	10 UJ	10 U	5 U
Tin	R	R	R	R	734 J	759 J	82.6 U	428 U
Vanadium	100 U	100 U	100 U	100 U	100 U	100 U	100 U	50 U
Zinc	458	332	248	272	288	272	82 U	78 U

J-Value is an estimated quantity.

U-Analyte was not detected. Value is the sample detection limit.

UJ-Analyte was not detected. Detection limit is estimated.

R-Datum was rejected.

NR-Not Reported

NATMAF INDUSTRIES INC  
DOCKET NO I-92-1072  
PCB ANALYSIS  
CERCLA STORMWATER (UG/L)

Sample ID	STATION#1	STATION#2	STATION#3	STATION#4	STATION#5	STATION#6	SW-DISCHARGE	
Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Post-Diversion	
Units	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	
Date Sampled	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	10/27/93	
Date Analyzed	8/14/93	8/14/93	8/14/93	8/14/93	8/14/93	8/14/93	12/9/93	
Remarks						Dup of STATION#5		
Laboratory	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	
Aroclor 1018	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	
Aroclor 1221	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	
Aroclor 1232	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	
Aroclor 1242	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	
Aroclor 1248	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	
Aroclor 1254	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	
Aroclor 1260	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	
Aroclor 1262	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	
Aroclor 1268	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	

J-Value is an estimated quantity.  
U-Analyte was not detected. Value is the sample detection limit.  
UJ-Analyte was not detected. Detection limit is estimated.  
R-Datum was rejected.

RAYMAR INDUSTRIES INC  
DOCKET NO I-92-1072  
ORGANOPHOSPHORUS PESTICIDE ANALYSIS  
CERCLA STORMWATER (UG/L)

1 of 1

Sample ID	STATION#1	STATION#2	STATION#3	STATION#4	STATION#5	STATION#6	SW-DISCHARGE	
Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Post-Diversion	
Units	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	
Date Sampled	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	10/27/93	
Date Analyzed	8/26/93	8/26/93	8/26/93	8/26/93	8/26/93	8/26/93	12/7/93	
Remarks						Dup of STATION#6		
Laboratory	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	
Dimethoate	1 U	1 U	1 U	1 U	1 U	1 U	1 U	
Disulfoton	1 U	1 U	1 U	1 U	1 U	1 U	1 U	
Famphur	1 U	1 U	1 U	1 U	1 U	1 U	1 U	
Methyl parathion	1 U	1 U	1 U	1 U	1 U	1 U	1 U	
Parathion	1 U	1 U	1 U	1 U	1 U	1 U	1 U	
Phorate	1 U	1 U	1 U	1 U	1 U	1 U	1 U	
Sulfotepp	1 U	1 U	1 U	1 U	1 U	1 U	1 U	
Thionazin	1 U	1 U	1 U	1 U	1 U	1 U	1 U	

J-Value is an estimated quantity.  
U-Analyte was not detected. Value is the sample detection limit.  
UJ-Analyte was not detected. Detection limit is estimated.  
R-Datum was rejected.

File: SDV505.XLS  
6/7/94 REV 1

RAYMAR INDUSTRIES INC  
DOCKET NO I-92-1072  
SEMIVOLATILE ORGANIC ANALYSIS  
CERCLA STORMWATER (UG/L)

1 of 4

Sample ID	STATION#1	STATION#2	STATION#3	STATION#4	STATION#5	STATION#6	SW-DISCHARGE	
Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Post-Diversion	
Units	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	
Date Sampled	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	10/27/93	
Date Analyzed	8/11/93	8/11/93	8/11/93	8/12/93	8/13/93	8/12/93	11/13/93	
Remarks						Dup of STATION#5		
Laboratory	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	
N-Nitrosodimethylamine	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Pyridine	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2-Picoline	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
N-Nitrosomethyl-ethylamine	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Methyl methansulfonate	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
N-Nitrosodiethylamine	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Ethyl methanesulfonate	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Pentachloroethane	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Aniline	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Phenol	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Bis(2-chloroethyl)ether	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2-Chlorophenol	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Benzyl alcohol	20 U	20 U	20 U	20 U	20 U	20 U	20 U	
o-Cresol	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
bis (2-chloro-1-methylethyl)ether	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Acetophenone	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
N-Nitrosopyrrolidine	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
N-Nitrosomorpholine	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
o-Toluidine	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
N-Nitroso-di-n-propylamine	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Hexachloroethane	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
m & p-Cresol	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Nitrobenzene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
N-Nitrosopiperidine	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Isophorone	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2-Nitrophenol	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2,4-Dimethylphenol	4 J	10 U	10 U	10 U	10 U	10 U	10 U	
Bis(2-chloroethoxy)methane	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
o,o,o,-Triethylphosphorothioate	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2,4-Dichlorophenol	10 U	10 U	10 U	10 U	10 U	10 U	10 U	

J-Value is an estimated quantity.

U-Analyte was not detected. Value is the sample detection limit.

UJ-Analyte was not detected. Detection limit is estimated.

R-Datum was rejected.

File: SDV508.XLS  
6/7/94 REV 1

KAYMAR INDUSTRIES INC  
DOCKET # 91-92-1072  
SEMIVOLATILE ORGANIC ANALYSIS  
CERCLA STORMWATER (UG/L)

2 of 4

Sample ID	STATION#1	STATION#2	STATION#3	STATION#4	STATION#5	STATION#6	SW-DISCHARGE	
Diversión	Pre-Diversión	Pre-Diversión	Pre-Diversión	Pre-Diversión	Pre-Diversión	Pre-Diversión	Post-Diversión	
Units	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	
Date Sampled	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	10/27/93	
Date Analyzed	8/11/93	8/11/93	8/11/93	8/12/93	8/13/93	8/12/93	11/13/93	
Remarks						Dup of STATION#5		
Laboratory	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	
a,a-Dimethylphenethylamine	20 U	20 U	20 U	20 U	20 U	20 U	20 U	
1,2,4-Trichlorobenzene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Naphthalene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
4-Chloroaniline	20 U	20 U	20 U	20 U	20 U	20 U	20 U	
2,6-Dichlorophenol	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Hexachloropropene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Hexachlorobutadiene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
p-Phenylenediamine	65 U	65 U	65 U	65 U	65 U	65 U	65 U	
N-Nitrosodi-n-butylamine	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
4-Chloro-3-methylphenol	20 U	20 U	20 U	20 U	20 U	20 U	20 U	
Isosafrole	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2-Methylnaphthalene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
1,2,4,5-Tetrachlorobenzene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Hexachlorocyclopentadiene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2,4,6-Trichlorophenol	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2,4,6-Trichlorophenol	50 U	50 U	50 U	50 U	50 U	50 U	50 U	
Safrole	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2-Chloronaphthalene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2-Nitroaniline	50 U	50 U	50 U	50 U	50 U	50 U	50 U	
1,4-Naphthoquinone	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Dimethylphthalate	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
m-Dinitrobenzene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Acenaphthylene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2,6-Dinitrotoluene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
3-Nitroaniline	50 U	50 U	50 U	50 U	50 U	50 U	50 U	

J-Value is an estimated quantity.

U-Analyte was not detected. Value is the sample detection limit.

UJ-Analyte was not detected. Detection limit is estimated.

R-Datum was rejected.

File: SDV806.XLS  
6/7/94 REV 1

RAYMA INDUSTRIES INC  
DOCKET NO I-92-1072  
SEMIVOLATILE ORGANIC ANALYSIS  
CERCLA STORMWATER (UG/L)

3 of 4

Sample ID	STATION#1	STATION#2	STATION#3	STATION#4	STATION#5	STATION#6	SW-DISCHARGE	
Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Post-Diversion	
Units	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	
Date Sampled	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	10/27/93	
Date Analyzed	8/11/93	8/11/93	8/11/93	8/12/93	8/13/93	8/12/93	11/13/93	
Remarks						Dup of STATION#5		
Laboratory	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	
Acenaphthene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2,4-Dinitrophenol	50 U	50 U	50 U	50 U	50 U	50 U	50 U	
4-Nitrophenol	50 U	50 U	50 U	50 U	50 U	50 U	50 U	
Dibenzofuran	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Pentachlorobenzene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2,4-Dinitrotoluene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
1-Naphthylamine	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2-Naphthylamine	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2,3,4,6-Tetrachlorophenol	20 U	20 U	20 U	20 U	20 U	20 U	20 U	
Diethylphthalate	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Fluorene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
4-Chlorophenyl-phenylether	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
5-Nitro-o-toluidine	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
4-Nitroaniline	50 U	50 U	50 U	50 U	50 U	50 U	50 U	
4,6-Dinitro-2-methylphenol	50 U	50 U	50 U	50 U	50 U	50 U	50 U	
Diphenylamine	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
sym-Trinitrobenzene	R	R	R	R	R	R	10 U	
4-Bromophenyl-phenylether	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Phenacetin	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Diallate	20 U	20 U	20 U	20 U	20 U	20 U	20 U	
Hexachlorobenzene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
4-Aminobiphenyl	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Pentachlorophenol	50 U	50 U	50 U	50 U	50 U	50 U	50 U	
Pronamide	20 U	20 U	20 U	20 U	20 U	20 U	20 U	
Pentachloronitrobenzene	20 U	20 U	20 U	20 U	20 U	20 U	20 U	
Phenanthrene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Anthracene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Carbazole	10 U	10 U	3 J	10 U	2 J	10 U	10 U	
Di-n-butylphthalate	10 U	10 U	10 U	10 U	10 U	10 U	2 J	
4-Nitroquinoline-1-oxide	R	R	R	R	R	R	10 U	

J-Value is an estimated quantity.

U-Analyte was not detected. Value is the sample detection limit.

UJ-Analyte was not detected. Detection limit is estimated.

R-Datum was rejected.

File: SDV506.XLS  
6/7/94 REV 1

HATMA INDUSTRIES INC  
DOCKET NO I-92-1072  
SEMIVOLATILE ORGANIC ANALYSIS  
CERCLA STORMWATER (UG/L)

- 4

Sample ID	STATION#1	STATION#2	STATION#3	STATION#4	STATION#5	STATION#6	SW-DISCHARGE	
Diverslon	Pre-Diverslon	Pre-Diverslon	Pre-Diverslon	Pre-Diverslon	Pre-Diverslon	Pre-Diverslon	Post-Diverslon	
Units	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	
Date Sampled	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	10/27/93	
Date Analyzed	8/11/93	8/11/93	8/11/93	8/12/93	8/13/93	8/12/93	11/13/93	
Remarks						Dup of STATION#5		
Laboratory	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	
Methapyrene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Isodrin	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Fluoranthene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Pyrene	10 U	10 U	10 U	10 U	2 J	2 J	10 U	
Aramite	20 U	20 U	20 U	20 U	20 U	20 U	20 U	
p-(Dimethylamino)azobenzene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Chlorobenzilate	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
3,3'-Dimethylbenzidine	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Kepone	20 U	20 U	20 U	20 U	20 U	20 U	20 U	
Butylbenzylphthalate	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
2-Acetylaminofluorene	20 U	20 U	20 U	20 U	20 U	20 U	20 U	
3,3'-Dichlorobenzidine	20 U	20 U	20 U	20 U	20 U	20 U	20 U	
Benzo(a)anthracene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Chrysene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Bis(2-ethylhexyl)phthalate	10 U	2 J	10 U	10 U	10 U	10 U	10 U	
DI-n-octylphthalate	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Benzo(b)fluoranthene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
7,12-Dimethylbenz(a)anthracene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Benzo(k)fluoranthene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Benzo(a)pyrene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
3-Methylcholanthrene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Indeno(1,2,3-cd)pyrene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Dibenzo(a,h)anthracene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	
Benzo(g,h,i)perylene	10 U	10 U	10 U	10 U	10 U	10 U	10 U	

J-Value is an estimated quantity.

U-Analyte was not detected. Value is the sample detection limit.

UJ-Analyte was not detected. Detection limit is estimated.

R-Datum was rejected.

File: SDV506.XLS  
8/7/94 REV 1

RAYMAN INDUSTRIES INC  
DOCKET NO I-92-1072  
VOLATILE ORGANIC ANALYSIS  
CERCLA STORMWATER (UG/L)

1 of 4

Sample ID	STATION#1	STATION#2	STATION#3	STATION#3DL	STATION#4	STATION#5	STATION#8	STATION#6MSD
Diverslon	Pre-Diverslon	Pre-Diverslon	Pre-Diverslon	Pre-Diverslon	Pre-Diverslon	Pre-Diverslon	Pre-Diverslon	Pre-Diverslon
Units	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L
Date Sampled	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93
Date Analyzed	8/9/93	8/9/93	8/9/93	8/10/93	8/10/93	8/10/93	8/10/93	8/10/93
Remarks				Diluted			Dup of STATION#5	Matrix Spike Dup of STATION#6MS
Laboratory	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC
Chloromethane	10 U	10 U	10 U	14 U	25 U	10 U	10 U	10 U
Bromomethane	10 U	10 U	10 U	14 U	25 U	10 U	10 U	10 U
Vinyl chloride	10 U	10 U	10 U	14 U	25 U	10 U	10 U	10 U
Chloroethane	10 U	4 J	10 U	14 U	25 U	6 J	6 J	4 J
Acetone	11 J	11 J	11 J	20	23 J	14 J	7 J	13
Carbon disulfide	5 U	5 U	5 U	7 U	12 U	5 U	5 U	5 U
1,1-Dichloroethane	5 U	5 U	5 U	7 U	12 U	5 U	5 U	45
1,1-Dichloroethane	3 J	6	42	37	80	24	24	23
1,2-Dichloroethane (total)	10 U	10 U	10 U	14 U	25 U	10 U	10 U	10 U
Chloroform	5 U	5 U	5 U	7 U	12 U	5 U	5 U	5 U
1,2-Dichloroethane	5 U	5 U	5 U	7 U	12 U	5 U	5 U	5 U
2-Butanone	10 U	10 U	10 U	14 U	25 U	10 U	10 U	10 U
1,1,1-Trichloroethane	14	19	200	200	460	93	88	90
Carbon tetrachloride	5 U	5 U	5 U	7 U	12 U	5 U	5 U	5 U
Bromodichloromethane	5 U	5 U	5 U	7 U	12 U	5 U	5 U	5 U
1,2-Dichloropropane	5 U	5 U	5 U	7 U	12 U	5 U	5 U	5 U
Trichloroethane	5 U	5 U	5 U	7 U	12 U	5 U	5 U	51
Dibromochloromethane	5 U	5 U	5 U	7 U	12 U	5 U	5 U	5 U
1,1,2-Trichloroethane	5 U	5 U	5 U	7 U	12 U	5 U	5 U	5 U
Benzene	5 U	5 U	5 U	7 U	12 U	5 U	5 U	50
4-Methyl-2-pentanone	10 U	10 U	10 U	14 U	25 U	10 U	10 U	10 U
2-Hexanone	10 U	10 U	10 U	14 U	25 U	10 U	10 U	10 U
Tetrachloroethane	5 U	5 U	5 U	7 U	12 U	5 U	5 U	5 U
Toluene	9	5 U	5 U	7 U	12 U	9	8	60
Ethylbenzene	2 J	5 U	5 U	7 U	12 U	5 U	5 U	5 U
Xylene (total)	20	5 U	5 U	7 U	12 U	2 J	5 U	5 U
1,2-Dichlorobenzene	5 U	5 U	5 U	7 U	12 U	5 U	5 U	5 U
1,3-Dichlorobenzene	5 U	5 U	5 U	7 U	12 U	5 U	5 U	5 U
1,4-Dichlorobenzene	5 U	5 U	5 U	7 U	12 U	5 U	5 U	5 U
Acetonitrile	40 U	40 U	40 U	57 U	100 U	40 U	40 U	40 U

J-Value is an estimated quantity.

U-Analyte was not detected. Value is the sample detection limit.

UJ-Analyte was not detected. Detection limit is estimated.

R-Datum was rejected.

File: SDV507.XLS  
8/7/94 REV 1



NATIONAL INDUSTRIES INC  
DOCKET NO 1-92-1072  
VOLATILE ORGANIC ANALYSIS  
CERCLA STORMWATER (UG/L)

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Sample ID	STATION#1	STATION#2	STATION#3	STATION#3DL	STATION#4	STATION#5	STATION#6	STATION#6MSD
Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion	Pre-Diversion
Units	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L
Date Sampled	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93
Date Analyzed	8/9/93	8/9/93	8/9/93	8/10/93	8/10/93	8/10/93	8/10/93	8/10/93
Remarks				Diluted			Dup of STATION#6	Matrix Spike Dup of STATION#6MS
Laboratory	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC
Propionitrile	20 U	20 U	20 U	29 U	50 U	20 U	20 U	20 U
Methyl Methacrylate	20 U	20 U	20 U	29 U	50 U	20 U	20 U	20 U
trans-1,4-Dichloro-2-butene	5 U	5 U	5 U	7 U	12 U	5 U	5 U	5 U
Dichlorodifluoromethane	10 UJ	10 UJ	10 UJ	14 U	25 UJ	10 UJ	10 UJ	10 U
Isobutyl Alcohol	100 U	100 U	100 U	140 U	250 U	100 U	100 U	100 U
1,4-Dioxane	R	R	R	29 U	R	R	R	20 U
trans-1,2-Dichloroethene	5 U	5 U	5 U	7 U	12 U	5 U	5 U	5 U
Methylene Chloride	10 U	10 U	10 U	14 U	25 U	10 U	10 U	10 U
Bromoform	10 U	10 U	10 U	14 U	25 U	10 U	10 U	10 U
Chlorobenzene	10 U	10 U	10 U	14 U	25 U	10 U	10 U	62
cis-1,2-Dichloroethene	10 U	10 U	10 U	14 U	25 U	10 U	10 U	10 U
1,1,2,2-Tetrachloroethane	10 U	10 U	10 U	14 U	25 U	10 U	10 U	10 U
cis-1,3-Dichloropropene	10 U	10 U	10 U	14 U	25 U	10 U	10 U	10 U
Chloroprene	20 U	20 U	20 U		50 U	20 U	20 U	

J-Value is an estimated quantity.  
U-Analyte was not detected. Value is the sample detection limit.  
UJ-Analyte was not detected. Detection limit is estimated.  
R-Datum was rejected.

File: SDV507.XLS  
8/7/94 REV 1

INDUSTRIAL DOCK NO 1-92-1072  
VOLATILE ORGANIC ANALYSIS  
CERCLA STORMWATER (UG/L)

Sample ID	STATION#6MS	TB	SW-DISCHARGE	TB			
Diversion	Pre-Diversion	Pre-Diversion	Post-Diversion	Post-Diversion			
Units	UG/L	UG/L	UG/L	UG/L			
Date Sampled	7/27/93	7/27/93	10/27/93	10/27/93			
Date Analyzed	8/10/93	8/10/93	11/7/93	11/7/93			
Remarks	Matrix Spike	Trip Blank		Trip Blank			
Laboratory	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC			
Chloromethane	10 U	10 U	10 U	10 U			
Bromomethane	10 U	10 U	10 U	10 U			
Vinyl chloride	10 U	10 U	10 U	10 U			
Chloroethane	5 J	10 U	11	10 U			
Acetone	12	10 U	10 U	7 U			
Carbon disulfide	5 U	5 U	5 U	5 U			
1,1-Dichloroethane	61	5 U	5 U	5 U			
1,1-Dichloroethane	25	5 U	13	6 U			
1,2-Dichloroethane (total)	10 U	10 U	10 U	10 U			
Chloroform	5 U	5 U	5 U	5 U			
1,2-Dichloroethane	6 U	5 U	5 U	5 U			
2-Butanone	10 U	10 U	10 U	10 U			
1,1,1-Trichloroethane	93	5 U	51	5 U			
Carbon tetrachloride	5 U	5 U	5 U	5 U			
Bromodichloromethane	6 U	5 U	5 U	5 U			
1,2-Dichloropropane	5 U	5 U	5 U	5 U			
Trichloroethane	53	5 U	5 U	5 U			
Dibromochloromethane	5 U	5 U	5 U	5 U			
1,1,2-Trichloroethane	5 U	5 U	5 U	5 U			
Benzene	51	5 U	5 U	5 U			
4-Methyl-2-pentanone	10 U	10 U	10 U	10 U			
2-Hexanone	10 U	10 U	10 U	10 U			
Tetrachloroethane	5 U	5 U	5 U	5 U			
Toluene	64	5 U	5 U	5 U			
Ethylbenzene	5 U	5 U	5 U	5 U			
Xylene (total)	5 U	5 U	5 U	5 U			
1,2-Dichlorobenzene	5 U	5 U	5 U	5 U			
1,3-Dichlorobenzene	5 U	5 U	5 U	5 U			
1,4-Dichlorobenzene	5 U	5 U	5 U	5 U			
Acetonitrile	40 U	40 U	40 U	40 U			

J-Value is an estimated quantity.

U-Analyte was not detected. Value is the sample detection limit.

UJ-Analyte was not detected. Detection limit is estimated.

R-Datum was rejected.

VOLATILE ORGANIC ANALYSIS  
CERCLA STORMWATER (UG/L)

Sample ID	STATION#6MS	TB	SW-DISCHARGE	TB			
Diverslon	Pre-Diverslon	Pre-Diverslon	Post-Diverslon	Post-Diverslon			
Units	UG/L	UG/L	UG/L	UG/L			
Date Sampled	7/27/93	7/27/93	10/27/93	10/27/93			
Date Analyzed	8/10/93	8/10/93	11/7/93	11/7/93			
Remarks	Matrix Spike	Trip Blank		Trip Blank			
Laboratory	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC			
Propionitrile	20 U	20 U	20 U	20 U			
Methyl Methacrylate	20 U	20 U	20 U	20 U			
trans-1,4-Dichloro-2-butene	5 U	5 U	5 U	5 U			
Dichlorodifluoromethane	10 U	10 UJ	10 U	10 U			
Isobutyl Alcohol	100 U	100 U	100 U	100 U			
1,4-Dioxane	20 U	R	20 U	20 U			
trans-1,2-Dichloroethene	5 U	5 U	5 U	5 U			
Methylene Chloride	10 U	10 U	10 U	10 U			
Bromoform	10 U	10 U	10 U	10 U			
Chlorobenzene	53	10 U	10 U	10 U			
cis-1,2-Dichloroethene	10 U	10 U	10 U	10 U			
1,1,2,2-Tetrachloroethane	10 U	10 U	10 U	10 U			
cis-1,3-Dichloropropene	10 U	10 U	10 U	10 U			
Chloroprene		20 U	20 U	20 U			

J-Value is an estimated quantity.  
U-Analyte was not detected. Value is the sample detection limit.  
UJ-Analyte was not detected. Detection limit is estimated.  
R-Datum was rejected.

RAYMAN INDUSTRIES INC  
DOCKET NO I-92-1072  
CYANIDE/SULFIDE ANALYSIS  
CERCLA STORMWATER (UG/L)

Sample ID	STATION#1	STATION#2	STATION#3	STATION#4	STATION#5	STATION#6		
Diverson	Pre-Diverson	Pre-Diverson	Pre-Diverson	Pre-Diverson	Pre-Diverson	Pre-Diverson		
Units	UG/L	UG/L	UG/L	UG/L	UG/L	UG/L		
Date Sampled	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93	7/27/93		
Remarks						Dup of STATION#5		
Laboratory	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC	AQUATEC INC		
Cyanide, Total	R	R	R	R	R	4.9 UJ		
Sulfide (MG/L)	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U		

J-Value is an estimated quantity.

U-Analyte was not detected. Value is the sample detection limit.

UJ-Analyte was not detected. Detection limit is estimated.

R-Datum was rejected.

File: SDV508.XLS  
6/7/94 REV 1

VOLUME CALCULATIONS FOR BASINS

SCALE: V: 1" = 60'

AREAS CALCULATED BY "POLYGON AREA"  
IN SITEPLANNER SITE 2a-26.0d1

AVERAGE FILL THICKNESS ESTIMATED  
FROM FILL ISOPACH MAP  
DATED NOV. 17, 1994

$A_{vf}$  = AVG FILL THICKNESS  
 $V_f$  = VOL. OF FILL

BASIN 1 AREA  
53,124 sq ft  
 $A_{vf_1} = 10.0$  ft  
 $V_{f_1} = 531,240$  cu ft  
= 19,676 cu yds.

BASIN 2  
AREA  
29,506 sq ft  
 $A_{vf_2} = 10.0$  ft  
 $V_{f_2} = 295,060$  cu ft  
= 10,928 cu yds

FENCE LINE

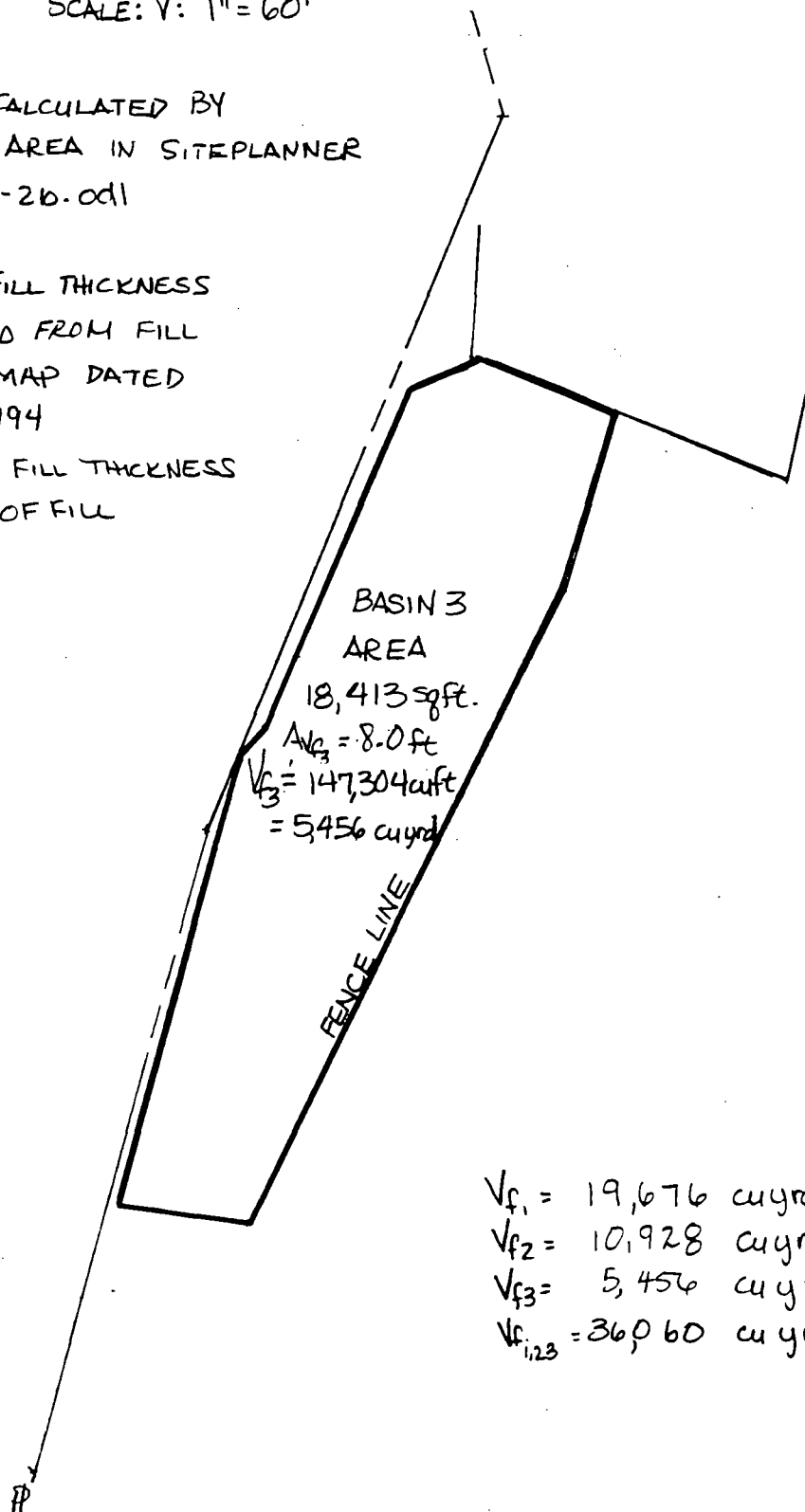
# VOLUME CALCULATIONS FOR BASINS

SCALE: V: 1" = 60'

AREAS CALCULATED BY  
"POLYGON AREA IN SITEPLANNER  
SITE 2A-26.0d1

AVERAGE FILL THICKNESS  
ESTIMATED FROM FILL  
ISOPACH MAP DATED  
Nov 17, 1994

$A_{V_f}$  = AVG FILL THICKNESS  
 $V_f$  = VOL. OF FILL



$V_{f_1} = 19,676$  cu yds  
 $V_{f_2} = 10,928$  cu yds  
 $V_{f_3} = 5,456$  cu yds  
 $V_{i,2,3} = 36,060$  cu yds

# VOLUME CALCULATIONS FOR BASINS

SCALE: V: 1" = 60'

AREAS CALCULATED BY "POLYGON AREA"  
IN SITEPLANNER SITE 29-2b.0d1

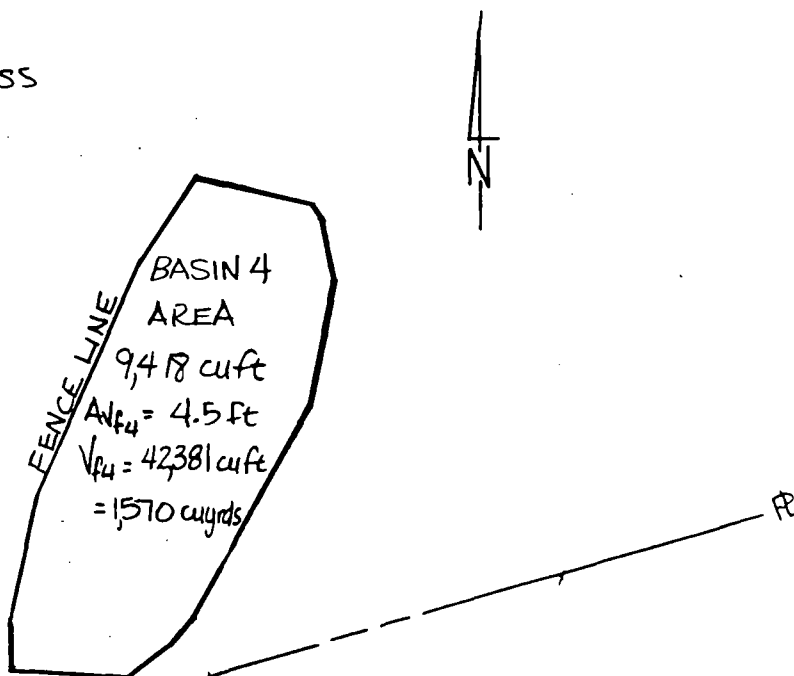
AVERAGE FILL THICKNESS ESTIMATED

FROM FILL ISOPACH MAP DATED

Nov. 17, 1994

$A_{vf}$  = AVG FILL THICKNESS

$V_f$  = VOL. OF FILL



$$V_{f4} = 1,570 \text{ cu yds}$$

$$V_{f,23} = 36,060 \text{ cu yds}$$

$$V_{f_{TOT}} = 37,630 \text{ cu yds}$$

## **Multivariate Statistical Analyses of Dioxin and Furan Levels in Fish, Sediment, and Soil Samples Collected Near Resource Recovery Facilities**

### **Final Report**

**By Karin M. Bauer**

**For Charles Fredette, Project Officer  
Connecticut Department of Environmental Protection  
Water Compliance Unit  
122 Washington Street  
Hartford, Connecticut 06106**

**Personal Services Agreement No. 91-1526  
MRI Project No. 9728-A  
Work Assignment No. 9**

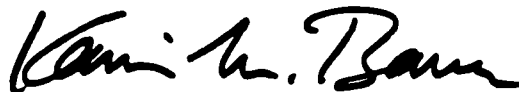
**December 9, 1992**



## PREFACE

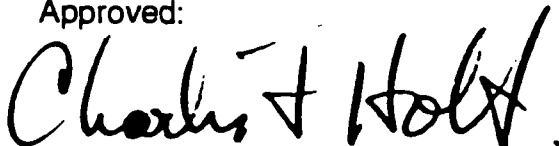
This report presents the results from a multivariate statistical analysis of polychlorinated dibenzo-*p*-dioxin (PCDD) and polychlorinated dibenzofuran (PCDF) data obtained from an environmental monitoring program. This program was designed to determine the impact of emissions from resource recovery facilities on the background levels of PCDDs and PCDFs in nearby areas. The work was carried out for the Connecticut Department of Environmental Protection under Personal Services Agreement No. 91-1526, MRI Project No. 9728-A, "Multivariate Statistical Analyses of Dioxin and Furan Levels in Fish, Sediment, and Soil Samples Collected Near Resource Recovery Facilities." The statistical analyses were performed by Ms. Karin M. Bauer, Principal Statistician, who was also the author of this report. Throughout the project, Dr. John S. Stanley, Head, Analytical Chemistry Section, provided guidance and consulting on chemical matters. Mr. Paul H. Cramer, Principal Chemist, provided additional assistance.

MIDWEST RESEARCH INSTITUTE



Karin M. Bauer  
Principal Statistician

Approved:



Charles F. Holt, Ph.D., Director  
Engineering and Environmental  
Technology Department

December 9, 1992

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## **SECTION 1**

### **INTRODUCTION**

This report presents the results and interpretation of a series of multi-variate statistical analyses performed on a data base containing levels of polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) in fish, sediment, and soil samples collected in Connecticut. Samples were collected in four cities over a 4-year period (1987 to 1990) during which a resource recovery facility went into operation. During the same time period, samples were also collected in a city where a facility had been in operation for several years. An additional four cities, each without a facility operating during the entire sampling period, were included in the study. The objective of this work was to determine whether incineration of municipal waste is affecting the levels of PCDDs and PCDFs found in the immediate environment of resource recovery facilities.

#### **1.1 PROJECT BACKGROUND**

The State of Connecticut's Department of Environmental Protection is responsible for monitoring the emissions of PCDDs and PCDFs from resource recovery facilities (RRFs). In order to assess the impact of the emissions of these analytes on the environment, the Water Compliance Unit in 1987 established an environmental monitoring program. Fish, sediment, and soil samples collected from 1987 through 1990 represent environmental levels during

both pre-operational and operational incineration activities from areas surrounding five RRFs and at four control sites. A total of 550 fish, 344 sediment, and 77 soil samples were analyzed for the 2,3,7,8-substituted isomers as well as for total tetra- through octachloro PCDDs and PCDFs.

Statistical analysis results based only on the 1987 and 1988 data were described in a preliminary draft report submitted to the Connecticut DEP on August 10, 1991. A presentation, based on that material, was given in poster format at the DIOXIN'91 international conference held in Research Triangle Park, North Carolina, in September 1991. A copy of the manuscript based on the 1987-88 data is attached as an appendix. Following the collection and analysis of the 1990 samples, the data bases were pooled and the complete data set was analyzed. The present report is a summary of the statistical analyses performed on the combined data set.

## **1.2 STUDY OBJECTIVES**

The primary objective of the Connecticut DEP environmental monitoring program is to assess whether incineration of municipal waste is affecting PCDD and PCDF levels. To that effect, analyte levels in samples collected before and after an RRF went into operation were compared using data from those four locations where such an installation took effect. In addition, results from samples collected near control sites (no RRF in operation) were compared to those from pre-operational and operational sites.

- Given the large amount of data and auxiliary information available from this program, a series of secondary study objectives were addressed as well. These consist of relating PCDD and PCDF levels in sediment and soil samples to both total organic carbon and grain size of the samples collected to detect a

potential effect of these physical properties on analyte levels. Similarly, the effect of lipid content of fish samples on PCDD and PCDF levels measured in these samples were examined to detect potential stratifications of fish species based on their PCDD and PCDF levels. Another set of analyses focussed on the interrelationship between individual isomers and/or homolog totals in the three matrices—fish, sediment, and soil—to uncover and compare potential analyte "fingerprints" in these matrices.

This report first presents a description of the chemical and physical sample properties data base (Section 2). Next, basic descriptive statistics for each measured variable are presented and discussed in Section 3. Section 4 is a synopsis of the two statistical procedures used for multivariate analysis. In Section 5, the results of each analysis are presented and discussed. The Appendix contains a reprint of the manuscript, based on a poster of this material presented at DIOXIN'91, and accepted for publication in *Chemosphere*.



## **SECTION 2**

### **DATA BASE DESCRIPTION**

Under a previous contract (Personal Services Agreement No. 89-1312) and the present contract (Personal Services Agreement No. 91-1526) with the Connecticut Department of Environmental Protection (DEP), Midwest Research Institute (MRI) analyzed fish, soil, and sediment samples collected over a 4-year period for 2,3,7,8-substituted chlorinated dioxins and furans. The results from these analyses were summarized in a series of data reports submitted to the Connecticut DEP between April 1989 and March 1992.

All data files generated under these contracts were made available on floppy diskettes for this work assignment. A single data file was then prepared for statistical analysis. Accuracy of the final data base was assessed by performing various frequency checks, random record comparisons with the individual reports, and other logical checks to determine consistency throughout the data base. The following subsections describe the complete data base.

#### **2.1 FIELD SAMPLE DESCRIPTORS**

The data base contained information on 971 field samples distributed as follows:

	1987	1988	1989 <sup>a</sup>	1990	Total
Fish	94	205	19	232	550
Sediment	78	107	0	159	344
<u>Soil</u>	<u>15</u>	<u>27</u>	<u>0</u>	<u>35</u>	<u>77</u>
Total	187	339	19	426	971

<sup>a</sup> Additional samples were collected in 1990 but the chemical analyses were done through another funding mechanism by an organization other than MRI.

For each sample, the DEP provided sampling information on the following parameters, each uniquely identifying the type and origin of the samples:

- Collection year (1987, 1988, 1989, or 1990);
- Number of years resource recovery facility has been in operation, if applicable;
- Sample type or matrix (fish, sediment, or soil);
- Town (total of nine) where the sample was collected;
- Water body (total of 21 ponds) where the fish or sediment sample was collected; and
- Fish species (total of 6 species).

The sampling characteristics for all field samples are presented in Tables 1, 2, and 3 for fish, sediment, and soil, respectively. Each table is organized by town, year of collection, water body (fish and sediment only), and fish species (fish only). The next to last column indicates the number of field samples within each category. Totals per town and year are shown in the last column. The third column in each table indicates the number of years the resource recovery facility was in operation at the time of sampling.

**Table 1. DISTRIBUTION OF 550 FISH SAMPLES**

Town	Collection year	Years of operation*	Water body	Fish species	No. of samples	Total
Bristol	1987	0	Grannis Pond	Brown Bullhead	7	37
	1987	0	Grannis Pond	White Sucker	9	
	1987	0	Grannis Pond	Yellow Perch	2	
	1987	0	Malones Pond	Brown Bullhead	9	
	1987	0	Malones Pond	Yellow Perch	10	
Bristol	1988	1	Grannis Pond	Brown Bullhead	14	56
	1988	1	Grannis Pond	White Sucker	8	
	1988	1	Grannis Pond	Yellow Perch	6	
	1988	1	Malones Pond	Brown Bullhead	13	
	1988	1	Malones Pond	Yellow Perch	15	
Bristol	1989	2	Grannis Pond	White Sucker	1	2
	1989	2	Malones Pond	Yellow Perch	1	
Bristol	1990	3	Grannis Pond	Brown Bullhead	9	48
	1990	3	Grannis Pond	White Sucker	10	
	1990	3	Grannis Pond	Yellow Perch	9	
	1990	3	Malones Pond	Brown Bullhead	10	
	1990	3	Malones Pond	Yellow Perch	10	
Hartford	1987	0	Keeney Cove	Carp	10	57
	1987	0	Keeney Cove	Channel Catfish	1	
	1987	0	Keeney Cove	White Catfish	6	
	1987	0	Keeney Cove	White Sucker	14	
	1987	0	Wethersfield Cove	Carp	2	
	1987	0	Wethersfield Cove	White Catfish	11	
	1987	0	Wethersfield Cove	White Sucker	13	
Hartford	1988	1	Keeney Cove	Carp	10	62
	1988	1	Keeney Cove	Channel Catfish	8	
	1988	1	Keeney Cove	White Sucker	10	
	1988	1	Wethersfield Cove	Carp	12	
	1988	1	Wethersfield Cove	Channel Catfish	7	
	1988	1	Wethersfield Cove	White Catfish	2	
	1988	1	Wethersfield Cove	White Sucker	13	
Hartford	1989	2	Keeney Cove	Carp	4	7
	1989	2	Keeney Cove	White Sucker	1	
	1989	2	Wethersfield Cove	Carp	1	
	1989	2	Wethersfield Cove	White Catfish	1	
Hartford	1990	3	Keeney Cove	Carp	11	45
	1990	3	Keeney Cove	Channel Catfish	2	
	1990	3	Keeney Cove	White Sucker	10	
	1990	3	Wethersfield Cove	Carp	10	
	1990	3	Wethersfield Cove	Channel Catfish	2	
	1990	3	Wethersfield Cove	White Sucker	10	

**Table 1 (continued)**

Town	Collection year	Years of operation <sup>a</sup>	Water body	Fish species	No. of samples	Total
Stafford (Control)	1989	0	Stafford Reservoir	Brown Bullhead	1	3
	1989	0	Stafford Reservoir	White Sucker	1	
	1989	0	Stafford Reservoir	Yellow Perch	1	
Stafford (Control)	1990	0	Stafford Reservoir	Brown Bullhead	10	27
	1990	0	Stafford Reservoir	White Sucker	7	
	1990	0	Stafford Reservoir	Yellow Perch	10	
Sterling	1990	0	Oneco Pond	White Sucker	10	41
	1990	0	Oneco Pond	Yellow Perch	11	
	1990	0	Sterling Pond	White Sucker	10	
	1990	0	Sterling Pond	Yellow Perch	10	
Union (Control)	1988	0	Bigelow Hollow	Brown Bullhead	7	21
	1988	0	Bigelow Hollow	White Sucker	4	
	1988	0	Bigelow Hollow	Yellow Perch	10	
Wallingford	1988	0	Community Lake	Brown Bullhead	1	35
	1988	0	Community Lake	Carp	8	
	1988	0	Community Lake	White Sucker	4	
	1988	0	Wharton Brook	Carp	12	
	1988	0	Wharton Brook	White Sucker	10	
Wallingford	1989	1	Community Lake	Carp	3	5
	1989	1	Community Lake	White Sucker	1	
	1989	1	Wharton Brook	White Sucker	1	
Wallingford	1990	2	Community Lake	Carp	13	43
	1990	2	Community Lake	White Sucker	10	
	1990	2	Wharton Brook	Carp	10	
	1990	2	Wharton Brook	White Sucker	10	
Willimantic <sup>b</sup>	1988	6	Willimantic Reservoir	Brown Bullhead	9	31
	1988	6	Willimantic Reservoir	White Sucker	12	
	1988	6	Willimantic Reservoir	Yellow Perch	10	
Willimantic <sup>b</sup>	1989	7	Willimantic Reservoir	Brown Bullhead	1	2
	1989	7	Willimantic Reservoir	White Sucker	1	
Willimantic <sup>b</sup>	1990	8	Willimantic Reservoir	Brown Bullhead	8	28
	1990	8	Willimantic Reservoir	White Sucker	10	
	1990	8	Willimantic Reservoir	Yellow Perch	10	

<sup>a</sup> Indicates number of years resource recovery facility has been in operation at time of sampling.

<sup>b</sup> Samples from this town have been excluded from selected statistical analyses (see Subsection 3.1.1, p. 14).

**Table 2. DISTRIBUTION OF 344 SEDIMENT SAMPLES**

Town	Collection year	Years of operation <sup>a</sup>	Water body	No. of samples	Total
Bridgeport <sup>b</sup>	1987	0	Black Rock Harbor	10	28
	1987	0	Bridgeport Harbor	6	
	1987	0	Brooklawn CC	2	
	1987	0	Mountain Grove Cemetery	4	
	1987	0	Rooster River	2	
	1987	0	Yellow Mill Channel	4	
Bridgeport	1988	1	Black Rock Harbor	7	19
	1988	1	Bridgeport Harbor	4	
	1988	1	Brooklawn CC	2	
	1988	1	Mountain Grove Cemetery	4	
	1988	1	Yellow Mill Channel	2	
Bridgeport	1990	3	Black Rock Harbor	5	19
	1990	3	Bridgeport Harbor	4	
	1990	3	Brooklawn CC	2	
	1990	3	Mountain Grove Cemetery	4	
	1990	3	Rooster River	2	
	1990	3	Yellow Mill Channel	2	
Bristol	1987	0	Grannis Pond	10	20
	1987	0	Malones Pond	10	
Bristol	1988	1	Grannis Pond	9	19
	1988	1	Malones Pond	10	
Bristol	1990	3	Grannis Pond	10	20
	1990	3	Malones Pond	10	
Hartford	1987	0	Hockanum Impoundment	10	30
	1987	0	Keeney Cove	10	
	1987	0	Wethersfield Cove	10	
Hartford	1988	1	Hockanum Impoundment	10	30
	1988	1	Keeney Cove	10	
	1988	1	Wethersfield Cove	10	
Hartford	1990	3	Hockanum Impoundment	10	30
	1990	3	Keeney Cove	10	
	1990	3	Wethersfield Cove	10	
Preston	1990	0	Fort Shantock	10	30
	1990	0	Norwich Reservoir	10	
	1990	0	Stoddard Hill	10	
Stafford (Control)	1990	0	Stafford Reservoir	10	10
Sterling	1990	0	Oneco Pond	10	20
	1990	0	Sterling Pond	10	
Union (Control)	1988	0	Bigelow Hollow	10	10
Wallingford	1988	0	Community Lake	10	20
	1988	0	Wharton Brook	10	
Wallingford	1990	2	Community Lake	10	20
	1990	2	Wharton Brook	10	
Willimantic <sup>c</sup>	1988	6	Willimantic Reservoir	9	9
Willimantic <sup>c</sup>	1990	8	Willimantic Reservoir	10	10

<sup>a</sup> Indicates number of years resource recovery facility has been in operation at time of sampling.

<sup>b</sup> No fish samples were collected in Bridgeport.

<sup>c</sup> Samples from this town have been excluded from selected statistical analyses (see Subsection 3.1.1, p. 14).

**Table 3. DISTRIBUTION OF 77 SOIL SAMPLES**

Town	Collection year	Years of operation <sup>a</sup>	No. of samples
Bridgeport	1987	0	7
Bridgeport	1988	1	7
Bridgeport	1990	3	7
Bristol	1987	0	4
Bristol	1988	1	4
Bristol	1990	3	4
Hartford	1987	0	4
Hartford	1988	1	4
Hartford	1990	3	4
Preston	1990	0	4
Stafford (Control)	1990	0	4
Sterling	1990	0	4
Union (Control)	1988	0	4
Wallingford	1988	0	4
Wallingford	1990	2	4
Willimantic <sup>b</sup>	1988	6	4
Willimantic <sup>b</sup>	1990	8	4

<sup>a</sup> Indicates number of years resource recovery facility has been in operation at time of sampling.

<sup>b</sup> Samples from this town have been excluded from selected statistical analyses (see Subsection 3.1.1, p. 14).

## 2.2 ANALYTICAL AND PHYSICAL MEASUREMENTS

Next, analytical results on all samples were provided. The complete list of homolog totals and isomers for which the samples were analyzed follows (the number preceding the analyte name is referred to as the analyte number in most figures and tables of this report):

1.	Total TCDF	14.	1,2,3,4,7,8-HxCDF
2.	Total TCDD	15.	1,2,3,6,7,8-HxCDF
3.	Total PeCDF	16.	2,3,4,6,7,8-HxCDF
4.	Total PeCDD	17.	1,2,3,7,8,9-HxCDF
5.	Total HxCDF	18.	1,2,3,4,7,8-HxCDD
6.	Total HxCDD	19.	1,2,3,6,7,8-HxCDD
7.	Total HpCDF	20.	1,2,3,7,8,9-HxCDD
8.	Total HpCDD	21.	1,2,3,4,6,7,8-HpCDF
9.	2,3,7,8-TCDF	22.	1,2,3,4,7,8,9-HpCDF
10.	2,3,7,8-TCDD	23.	1,2,3,4,6,7,8-HpCDD
11.	1,2,3,7,8-PeCDF	24.	1,2,3,4,6,7,8,9-OCDF
12.	2,3,4,7,8-PeCDF	25.	1,2,3,4,6,7,8,9-OCDD
13.	1,2,3,7,8-PeCDD		

For these 25 isomers and homolog totals, the following data were available:

- Data qualifier:
  - ND for not detected;
  - CDL for curve-based detection limit;
  - MPC for maximum possible concentration;
  - PQ for positive quantifiable;
  - GT for greater than;
- Limit of detection, in picograms per gram (pg/g) or equivalently in parts per trillion (ppt), if the data qualifier was either "ND" or "CDL"; and

- Concentration level (pg/g), if the data qualifier was "MPC", "PQ", or "GT". All concentrations in fish samples are reported on a whole weight basis. All soil and sediment results are reported on a dry weight basis.

In addition, information for the following physical sample properties was available:

- Percent lipid for all fish samples;
- Percent moisture for all soil and sediment samples;
- Total organic carbon (TOC) in mg/kg for all soil and sediment samples; and
- Grain size, expressed as percent sand (particle size from 0.062 to 2 mm), percent silt (particle size from 0.004 to 0.062 mm), and percent clay (particle size less than 0.004 mm), for all soil and sediment samples.

An average (weighted) particle size for all sediment and soil samples was derived from the percents of sand, silt, and clay, and the midpoints of the above particle size ranges for these three categories. The weighted average particle size was thus computed as follows:

$$\begin{aligned}\text{Particle size (mm)} &= \% \text{ sand} \times (2 + 0.0062)/2 + \% \text{ silt} (0.062 + 0.004)/2 + \% \text{ clay} \times (0.004 + 0.001)/2 \\ &= \% \text{ sand} \times 1.031 + \% \text{ silt} \times 0.033 + \% \text{ clay} \times 0.0025\end{aligned}$$

Thus, for large proportions of sand, the average particle size will be close to 1 mm, while for samples consisting primarily of silt and clay, the average particle size will be on the low end, between 0.003 and 0.033 mm. This calculated parameter was used in subsequent analyses whenever particle size was considered.



## **SECTION 3**

### **DESCRIPTIVE STATISTICS**

The analysis results presented and discussed in this report are based solely on the 971 field samples. Statistical analysis of the quality assurance sample results (e.g., method blanks, matrix spikes) is not included. A statistical analysis of the QA sample results could be undertaken in a subsequent task and incorporated with the results reported herein as a means of: (a) validating relationships, if they exist, among the samples and the chemical variables; and (b) analyzing the QA/QC results in a multivariate context.

The following subsections present basic descriptive statistics (that is univariate and bivariate) for the chemical data and the samples' physical properties—percent lipid for fish samples, and TOC and average particle size for soil and sediment samples.

#### **3.1 SIMPLE DESCRIPTIVE STATISTICS OF THE CHEMICAL DATA**

Throughout all analyses, half the limit of detection value was used for those samples with levels below detection limits (data qualifiers either "ND" or "CDL"). This approach was taken to conform with the approach chosen by the Connecticut DEP in their analysis of the data. When the analyte was detected, the reported concentration was used in the analyses.

### 3.1.1 Percent Nondetects in Each Matrix

As a first step, percentages of nondetects and detects (data qualifiers "PQ," "MPC," or "GT") were summarized for each matrix and each analyte separately. The results, both in frequency counts and in percentages, are shown in Tables 4, 5, and 6, for fish, sediment, and soil, respectively. The data were grouped depending on whether the resource recovery facility was operational at the time of sampling. The status of the facility is shown in the second column of Tables 4 through 6. All samples from Willimantic were excluded from the statistics in these three tables. Since the facility in Willimantic had been operating since 1982, the samples from this city could not be included in either the pre-operational or operational category. The analyte statistics are shown separately by status of the resource recovery facility. An upper limit of 25% for the total number of samples with levels below detection limit for a given analyte (i.e., at least 75% detects) was selected as the cutoff point for that analyte to be included in some of the subsequent multivariate analyses. These analytes are marked by an "\*\*\*" in Tables 4 to 6.

A total of 14 analytes (4 totals and 10 isomers) have levels below detection limits in more than 25% of the fish samples either before or after an RRF went into operation. Only two analytes—2,3,7,8-TCDD and 1,2,3,7,8,9-HxCDF—have levels below detection limit in more than 25% of the sediment or soil samples.

**Table 4. NUMBER AND PERCENT OF FISH SAMPLES WITH LEVELS  
BELOW OR ABOVE DETECTION LIMIT (LOD)**

Analyte	Status of resource recovery facility	Total No. <sup>a</sup> of samples	No. of samples		% of samples	
			Below LOD	Above LOD	Below LOD	Above LOD
Total TCDF	Pre-operational Operational	221 268	2 268	219 268	0.90 100	99 100
Total TCDD	Pre-operational Operational	217 265	10 5	207 260	4.6 1.9	95 98
Total PeCDF	Pre-operational Operational	219 268	7 268	212 268	3.2 100	97 100
Total PeCDD*	Pre-operational Operational	217 267	81 44	136 223	37 16	63 84
Total HxCDF*	Pre-operational Operational	220 268	72 34	148 234	33 13	67 87
Total HxCDD*	Pre-operational Operational	221 268	67 25	154 243	30 9.3	70 91
Total HpCDF*	Pre-operational Operational	220 268	117 42	103 226	53 16	47 84
Total HpCDD	Pre-operational Operational	221 268	11 268	210 268	5.0 100	95 100
2,3,7,8-TCDF	Pre-operational Operational	221 268	1 268	220 268	0.45 100	100 100
2,3,7,8-TCDD	Pre-operational Operational	217 265	12 3	205 262	5.5 1.1	94 99
1,2,3,7,8-PeCDF	Pre-operational Operational	219 268	38 18	181 250	17 6.7	83 93
2,3,4,7,8-PeCDF	Pre-operational Operational	219 268	34 1	185 267	16 0.37	84 100
1,2,3,7,8-PeCDD	Pre-operational Operational	217 267	23 10	194 257	11 3.7	89 96
1,2,3,4,7,8-HxCDF*	Pre-operational Operational	220 268	93 85	127 183	42 32	58 68
1,2,3,6,7,8-HxCDF*	Pre-operational Operational	220 268	124 96	96 172	56 36	44 64
2,3,4,6,7,8-HxCDF*	Pre-operational Operational	220 268	96 68	124 200	44 25	56 75
1,2,3,7,8,9-HxCDF*	Pre-operational Operational	220 268	185 254	35 14	84 95	16 5.2
1,2,3,4,7,8-HxCDD*	Pre-operational Operational	221 268	102 94	119 174	46 35	54 65
1,2,3,6,7,8-HxCDD*	Pre-operational Operational	221 268	70 30	151 238	32 11	68 89

Table 4 (continued)

Analyte	Status of resource recovery facility	Total No.* of samples	No. of samples		% of samples	
			Below LOD	Above LOD	Below LOD	Above LOD
1,2,3,7,8,9-HxCDD*	Pre-operational	221	111	110	50	50
	Operational	268	95	173	35	65
1,2,3,4,6,7,8-HpCDF*	Pre-operational	220	85	135	39	61
	Operational	268	41	227	15	85
1,2,3,4,7,8,9-HpCDF*	Pre-operational	220	183	37	83	17
	Operational	268	202	66	75	25
1,2,3,4,6,7,8-HpCDD	Pre-operational	221	3	218	1.4	99
	Operational	268		268		100
OCDF*	Pre-operational	220	129	91	59	41
	Operational	268	129	139	48	52
OCDD	Pre-operational	220	1	219	0.45	100
	Operational	268		268		100

\* Analytes with high occurrences of nondetects (over 25% during either pre-operational or operational status of the RRF).

\* Samples from Willimantic excluded.

**Table 5. NUMBER AND PERCENT OF SEDIMENT SAMPLES WITH LEVELS BELOW OR ABOVE DETECTION LIMIT (LOD)**

Analyte	Status of resource recovery facility	Total No.* of samples	No. of samples		% of samples	
			Below LOD	Above LOD	Below LOD	Above LOD
Total TCDF	Pre-operational	142	1	141	0.70	99
	Operational	155	3	152	1.9	98
Total TCDD	Pre-operational	162	12	150	7.4	93
	Operational	157	21	136	13	87
Total PeCDF	Pre-operational	163		163		100
	Operational	157	2	155	1.3	99
Total PeCDD	Pre-operational	162	4	158	2.5	98
	Operational	157	10	147	6.4	94
Total HxCDF	Pre-operational	166		166		100
	Operational	157	1	156	0.64	99
Total HxCDD	Pre-operational	166	1	165	0.60	99
	Operational	157	2	155	1.3	99
Total HpCDF	Pre-operational	166		166		100
	Operational	157	1	156	0.64	99
Total HpCDD	Pre-operational	166		166		100
	Operational	157		157		100
2,3,7,8-TCDF	Pre-operational	142	1	141	0.70	99
	Operational	155	5	150	3.2	97
2,3,7,8-TCDD*	Pre-operational	162	37	125	23	77
	Operational	157	49	108	31	69
1,2,3,7,8-PeCDF	Pre-operational	163	3	160	1.8	98
	Operational	157	13	144	8.3	92
2,3,4,7,8-PeCDF	Pre-operational	163	5	158	3.1	97
	Operational	157	10	147	6.4	94
1,2,3,7,8-PeCDD	Pre-operational	161	7	154	4.3	96
	Operational	157	33	124	21	79
1,2,3,4,7,8-HxCDF	Pre-operational	166	3	163	1.8	98
	Operational	157	10	147	6.4	94
1,2,3,6,7,8-HxCDF	Pre-operational	166	1	165	0.60	99
	Operational	157	14	143	8.9	91
2,3,4,6,7,8-HxCDF	Pre-operational	166	2	164	1.2	99
	Operational	157	18	139	11	89
1,2,3,7,8,9-HxCDF*	Pre-operational	166	82	84	49	51
	Operational	157	110	47	70	30
1,2,3,4,7,8-HxCDD	Pre-operational	166	17	149	10	90
	Operational	156	29	127	19	81
1,2,3,6,7,8-HxCDD	Pre-operational	166	3	163	1.8	98
	Operational	157	11	146	7.0	93

**Table 5 (continued)**

Analyte	Status of resource recovery facility	Total No.* of samples	No. of samples		% of samples	
			Below LOD	Above LOD	Below LOD	Above LOD
1,2,3,7,8,9-HxCDD	Pre-operational	166	2	164	1.2	99
	Operational	157	12	145	7.6	92
1,2,3,4,6,7,8-HpCDF	Pre-operational	166		166		100
	Operational	157		157		100
1,2,3,4,7,8,9-HpCDF	Pre-operational	166	8	158	4.8	95
	Operational	157	18	139	11	89
1,2,3,4,6,7,8-HpCDD	Pre-operational	166		166		100
	Operational	157		157		100
OCDF	Pre-operational	166		166		100
	Operational	155		155		100
OCDD	Pre-operational	166		166		100
	Operational	155		155		100

- \* Analytes with high occurrences of nondetects (over 25% during either pre-operational or operational status of the RRF).
- Samples from Willimantic excluded.

**Table 6. NUMBER AND PERCENT OF SOIL SAMPLES WITH LEVELS  
BELOW OR ABOVE DETECTION LIMIT (LOD)**

Analyte	Status of resource recovery facility	Total No.* of samples	No. of samples		% of samples	
			Below LOD	Above LOD	Below LOD	Above LOD
Total TCDF	Pre-operational	34	2	32	5.9	94
	Operational	34	1	33	2.9	97
Total TCDD	Pre-operational	34	6	28	18	82
	Operational	34	8	26	24	76
Total PeCDF	Pre-operational	34		34		100
	Operational	34		34		100
Total PeCDD	Pre-operational	34	2	32	5.9	94
	Operational	34	1	33	2.9	97
Total HxCDF	Pre-operational	35		35		100
	Operational	34		34		100
Total HxCDD	Pre-operational	35	1	34	2.9	97
	Operational	34		34		100
Total HpCDF	Pre-operational	35		35		100
	Operational	34		34		100
Total HpCDD	Pre-operational	35		35		100
	Operational	34		34		100
2,3,7,8-TCDF	Pre-operational	34	1	33	2.9	97
	Operational	34	1	33	2.9	97
2,3,7,8-TCDD*	Pre-operational	34	19	15	56	44
	Operational	34	15	19	44	56
1,2,3,7,8-PeCDF	Pre-operational	34		34		100
	Operational	34		34		100
2,3,4,7,8-PeCDF	Pre-operational	34	1	33	2.9	97
	Operational	34		34		100
1,2,3,7,8-PeCDD	Pre-operational	34		34		100
	Operational	34	2	32	5.9	94
1,2,3,4,7,8-HxCDF	Pre-operational	35		35		100
	Operational	34		34		100
1,2,3,6,7,8-HxCDF	Pre-operational	35		35		100
	Operational	34		34		100
2,3,4,6,7,8-HxCDF	Pre-operational	35		35		100
	Operational	34		34		100
1,2,3,7,8,9-HxCDF*	Pre-operational	35	17	18	49	51
	Operational	34	18	16	53	47
1,2,3,4,7,8-HxCDD	Pre-operational	35	4	31	11	89
	Operational	34	2	32	5.9	94
1,2,3,6,7,8-HxCDD	Pre-operational	35	1	34	2.9	97
	Operational	34		34		100

**Table 6 (continued)**

Analyte	Status of resource recovery facility	Total No.* of samples	No. of samples		% of samples	
			Below LOD	Above LOD	Below LOD	Above LOD
1,2,3,7,8,9-HxCDD	Pre-operational	35	1	34	2.9	97
	Operational	34		34		100
1,2,3,4,6,7,8-HpCDF	Pre-operational	35		35		100
	Operational	34		34		100
1,2,3,4,7,8,9-HpCDF	Pre-operational	35	2	33	5.7	94
	Operational	34		34		100
1,2,3,4,6,7,8-HpCDD	Pre-operational	35		35		100
	Operational	34		34		100
OCDF	Pre-operational	35		35		100
	Operational	34		34		100
OCDD	Pre-operational	35		35		100
	Operational	34		34		100

- \* Analytes with high occurrences of nondetects (over 25% during either pre-operational or operational status of the RRF).
- \* Samples from Willimantic excluded.



Figures 1. through 3 display the percent of samples with levels below detection limit for all analytes for fish, sediment, and soil samples, respectively. These figures clearly illustrate the trend from fish to sediment to soil samples: the number of analytes with levels below detection limit decreases considerably from fish to sediment to soil samples.

### **3.1.2 Simple Descriptive Statistics for PCDDs and PCDFs**

Basic descriptive statistics of the analytical results are summarized in Table 7. This table is arranged by analyte, matrix, and status of the RRF. For each combination of analyte, matrix, and status, the number of samples, mean, standard deviation, minimum, 5th and 25th percentiles, median or 50th percentile, 75th and 95th percentiles, maximum, and midrange have been computed. All statistics are in picograms per gram (pg/g). For levels below detection limits, half the detection limit was used for the computations. Again, the data from samples collected in Willimantic were excluded from these statistics for comparison of pre-operational and operational data.

It should be noted that the grouping of the samples into pre-operational and operational categories, as done in Tables 4 through 7, is somewhat misleading. In fact, of the nine cities considered in this study, only four (Bristol, Bridgeport, Hartford, and Wallingford) fit into pre-operational and operational status categories. As mentioned earlier, an RRF has been in operation in Willimantic since approximately 1982. The cities of Union and Stafford serve as controls, while Sterling was in pre-operational status when these data were corrected. Pre-operational and operational comparisons of PCDD and PCDF levels will be made using multivariate techniques on the Bristol, Bridgeport, Hartford, and Wallingford data only.

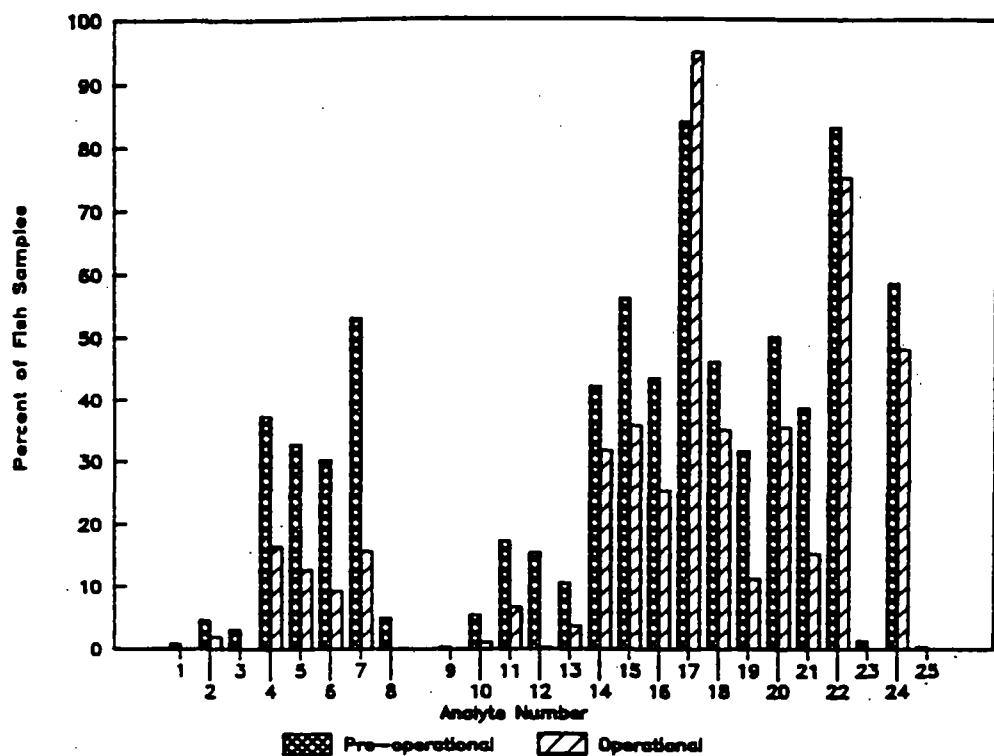


Figure 1. Histogram of percent nondetects by analyte in fish samples.

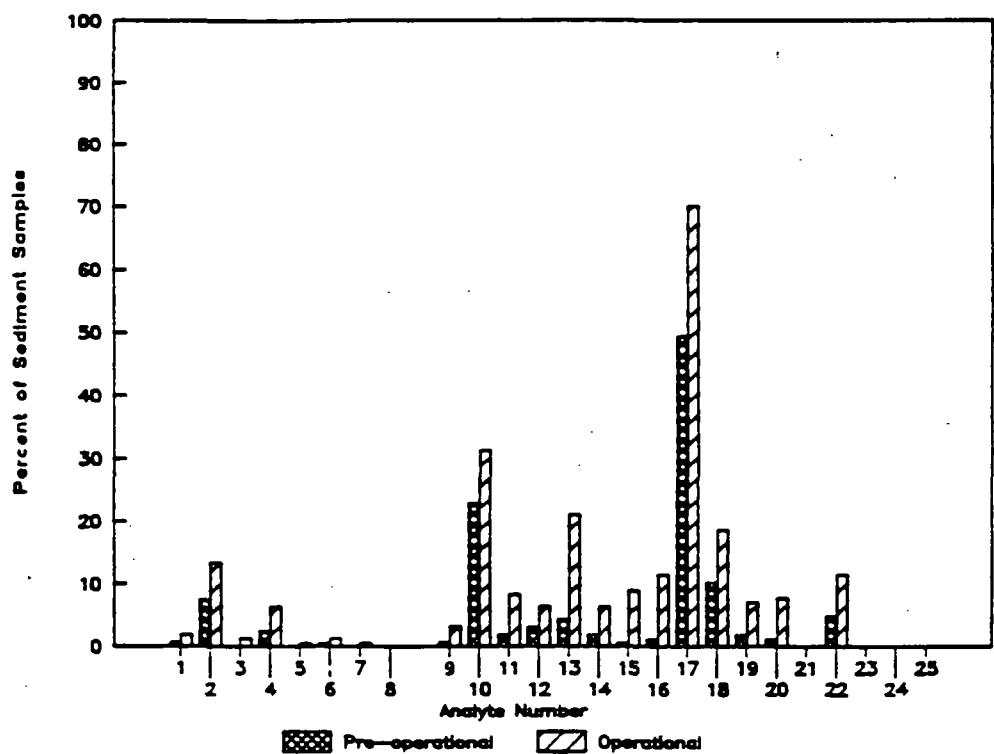


Figure 2. Histogram of percent nondetects by analyte in sediment samples.

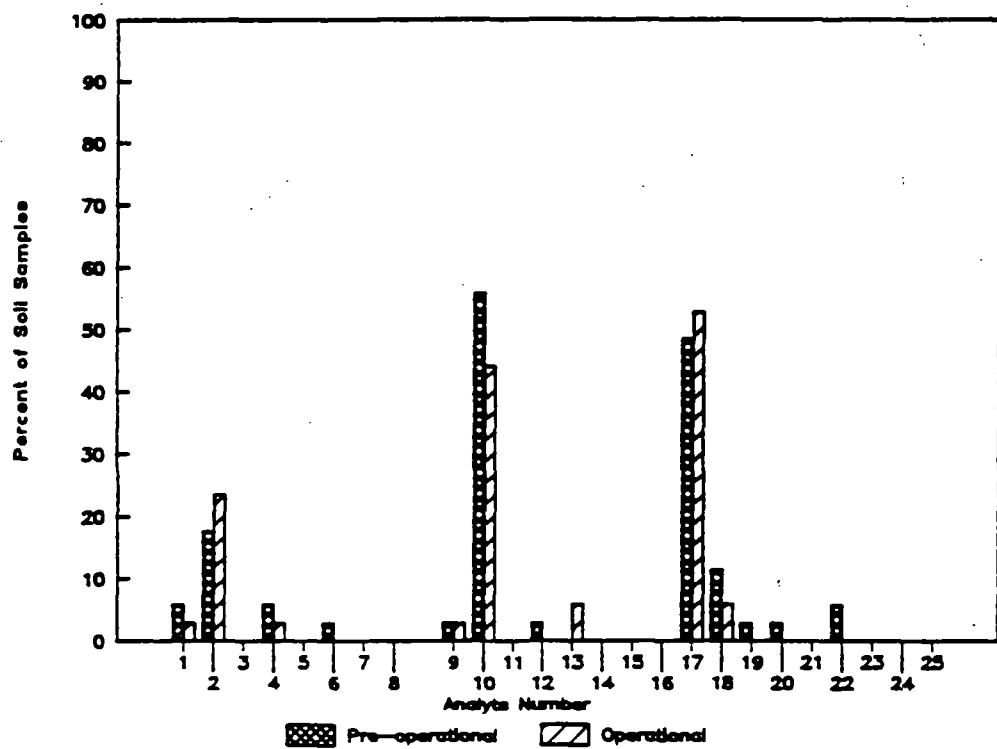


Figure 3. Histogram of percent nondetects by analyte in soil samples.

**Table 7. DESCRIPTIVE PCDD AND PCDF CONCENTRATION STATISTICS (pg/g)**

Analyte	Matrix	RRF Status	No. of samples	Mean	Standard deviation	Minimum	Percentiles					Maximum	Midrange <sup>a</sup>
							5th	25th	Median or 50th	75th	95th		
Total TCDF	Fish <sup>b</sup>	Pre-operational	221	4.66	5.50	0.06	0.36	1.05	2.71	6.50	15.09	45.70	5.46
		Operational	268	6.31	8.27	0.07	0.32	1.56	4.41	8.66	15.31	77.10	7.10
	Sediment <sup>c</sup>	Pre-operational	142	62.78	56.00	0.03	8.26	21.28	42.95	102.0	179.3	290.0	80.73
		Operational	155	77.45	99.82	0.04	1.95	14.20	56.80	107.0	238.0	929.0	92.80
	Soil <sup>d</sup>	Pre-operational	34	23.36	42.58	0.12	0.14	3.81	8.42	20.05	161.5	202.0	16.24
		Operational	34	50.37	69.15	0.08	0.64	7.50	17.75	62.15	225.5	272.0	54.65
Total TCDD	Fish	Pre-operational	217	1.20	1.58	0.02	0.08	0.28	0.56	1.36	5.29	8.37	1.08
		Operational	265	1.64	2.02	0.03	0.14	0.38	0.84	1.87	6.00	10.90	1.50
	Sediment	Pre-operational	162	9.34	9.52	0.07	0.25	3.06	5.38	3.60	29.20	48.70	10.54
		Operational	157	16.60	30.82	0.09	0.17	1.57	4.34	20.75	68.24	288.0	19.18
	Soil	Pre-operational	34	3.63	6.42	0.09	0.12	0.32	1.40	3.83	21.00	33.30	3.51
		Operational	34	8.66	13.20	0.06	0.12	0.30	2.91	11.10	47.60	49.70	10.80
Total PeCDF	Fish	Pre-operational	219	9.56	30.28	0.03	0.28	1.05	2.01	5.00	43.40	364.0	3.95
		Operational	268	12.37	38.38	0.15	0.48	1.53	3.24	7.62	60.97	444.0	6.30
	Sediment	Pre-operational	163	103.0	108.6	0.20	10.88	34.70	79.80	134.0	273.6	775.0	99.30
		Operational	157	124.6	177.6	0.07	2.34	24.00	71.90	153.5	509.9	1,330	129.5
	Soil	Pre-operational	34	42.97	72.61	0.59	2.23	7.72	13.25	37.32	264.3	343.0	29.61
		Operational	34	96.65	126.3	1.13	1.26	16.95	36.75	119.8	419.0	479.0	102.6
Total PeCDD	Fish	Pre-operational	217	0.54	1.20	0.01	0.02	0.04	0.10	0.35	3.23	9.08	0.31
		Operational	267	1.18	2.12	0.01	0.02	0.10	0.40	1.11	6.41	15.20	1.01
	Sediment	Pre-operational	162	28.85	42.13	0.03	1.11	6.79	14.95	33.45	113.4	327.0	26.66
		Operational	157	46.31	79.66	0.07	0.24	4.88	16.00	55.15	158.8	718.0	50.27
	Soil	Pre-operational	34	11.73	22.37	0.16	0.22	1.23	4.09	8.68	73.50	108.0	7.63
		Operational	34	19.82	27.91	0.07	0.35	4.14	8.23	25.27	108.3	121.0	21.13
Total HxCDF	Fish	Pre-operational	220	3.65	24.02	0.03	0.06	0.09	0.51	1.34	7.19	334.0	1.25
		Operational	268	4.44	11.00	0.03	0.07	0.26	1.31	2.95	26.27	120.0	2.69
	Sediment	Pre-operational	166	132.0	155.8	0.44	10.31	35.73	83.30	168.5	423.9	908.0	132.8
		Operational	157	152.0	198.8	0.67	2.94	26.90	73.40	230.0	499.6	1,610	203.1
	Soil	Pre-operational	35	40.99	71.77	0.72	2.34	6.78	12.60	45.50	252.2	373.0	38.72
		Operational	34	81.20	108.1	1.12	1.28	12.85	39.55	87.52	381.5	416.0	74.67

Analyte	Matrix	RRF Status	No. of samples	Mean	Standard deviation	Minimum	Percentiles					Maximum	Midrange*
							5th	25th	Median or 50th	75th	95th		
Total HxCDD	Fish	Pre-operational	221	2.28	5.71	0.03	0.08	0.09	0.53	1.75	11.69	47.90	1.66
		Operational	268	5.97	21.84	0.02	0.07	0.37	1.10	3.21	19.72	238.0	2.85
	Sediment	Pre-operational	166	130.2	176.3	0.09	9.78	27.55	63.65	155.5	564.8	950.0	128.0
		Operational	157	146.2	196.8	0.30	3.57	21.95	69.70	220.5	428.2	1,670	198.6
	Soil	Pre-operational	35	28.72	38.62	0.29	2.75	7.21	13.90	38.60	158.0	170.0	29.39
		Operational	34	48.19	58.70	1.98	2.00	12.45	25.15	60.07	215.8	233.0	47.62
Total HpCDF	Fish	Pre-operational	220	0.71	3.02	0.02	0.04	0.07	0.10	0.51	2.09	41.70	0.45
		Operational	268	1.02	1.39	0.02	0.05	0.17	0.49	1.13	3.97	8.81	0.95
	Sediment	Pre-operational	166	190.2	246.3	0.58	9.78	43.23	85.05	214.0	788.6	1,270	170.8
		Operational	157	233.8	309.4	0.61	6.44	29.75	97.40	370.5	743.2	1,990	340.8
	Soil	Pre-operational	35	33.21	44.84	1.68	2.08	7.15	13.60	43.70	182.4	188.0	38.55
		Operational	34	68.64	79.84	2.15	2.63	13.48	43.75	91.85	280.8	358.0	78.37
Total HpCDD	Fish	Pre-operational	221	5.86	28.18	0.03	0.14	0.43	1.07	2.50	18.50	381.0	2.07
		Operational	268	25.45	143.4	0.10	0.29	0.91	1.88	5.00	32.35	1,600	4.09
	Sediment	Pre-operational	166	647.0	883.2	1.77	29.88	126.8	268.0	712.5	2,750	5,820	585.8
		Operational	157	783.4	976.9	3.11	23.60	89.35	341.0	1,270	2,728	5,587	1,181
	Soil	Pre-operational	35	121.2	140.2	4.59	11.80	24.80	63.20	160.0	522.4	568.0	135.2
		Operational	34	222.0	255.9	8.04	10.11	58.25	117.5	289.5	921.8	960.0	233.3
2,3,7,8-TCDF	Fish	Pre-operational	221	2.29	2.34	0.08	0.25	0.58	1.38	3.71	6.98	15.10	3.18
		Operational	268	3.04	2.79	0.07	0.16	0.88	2.26	4.58	8.83	14.30	3.88
	Sediment	Pre-operational	142	13.58	13.60	0.03	1.08	4.63	9.39	19.72	38.40	101.0	15.09
		Operational	155	19.77	24.83	0.04	0.83	3.51	11.20	28.40	63.44	208.0	24.89
	Soil	Pre-operational	34	4.78	9.32	0.15	0.17	0.90	2.01	3.39	35.85	46.00	2.49
		Operational	34	9.77	15.16	0.18	0.18	1.57	3.09	11.35	58.45	61.60	9.78
2,3,7,8-TCDD	Fish	Pre-operational	217	0.93	1.30	0.03	0.07	0.15	0.34	1.11	3.99	8.37	0.97
		Operational	265	1.53	2.03	0.03	0.13	0.29	0.69	1.65	5.98	10.90	1.36
	Sediment	Pre-operational	162	2.30	3.75	0.04	0.15	0.34	1.13	2.51	9.78	23.10	2.18
		Operational	157	2.13	4.79	0.05	0.13	0.37	0.82	2.09	5.86	51.50	1.72
	Soil	Pre-operational	34	0.61	1.68	0.02	0.04	0.08	0.10	0.31	8.49	7.98	0.23
		Operational	34	0.64	1.21	0.01	0.05	0.15	0.25	0.57	4.91	5.78	0.42

Table 7 (continued)

Analyte	Matrix	RRF Status	No. of samples	Mean	Standard deviation	Minimum	Percentiles					Maximum	Midrange*
							5th	25th	Median or 50th	75th	95th		
1,2,3,7,8-PeCDF	Fish	Pre-operational	219	1.08	5.25	0.02	0.03	0.10	0.19	0.50	1.71	67.30	0.40
		Operational	268	0.52	0.62	0.02	0.03	0.14	0.28	0.61	1.88	3.89	0.48
	Sediment	Pre-operational	163	4.98	6.15	0.01	0.34	1.27	2.79	6.46	15.76	37.80	5.19
		Operational	157	5.68	8.58	0.04	0.13	0.94	3.22	6.97	17.21	82.90	6.03
	Soil	Pre-operational	34	1.74	3.08	0.04	0.12	0.49	0.70	1.29	12.00	15.00	0.80
		Operational	34	3.97	6.84	0.03	0.15	0.72	1.23	3.79	25.73	32.70	3.07
2,3,4,7,8-PeCDF	Fish	Pre-operational	219	1.08	2.17	0.02	0.03	0.18	0.37	0.82	5.02	15.70	0.64
		Operational	268	2.50	4.55	0.02	0.15	0.38	0.65	2.32	11.91	31.80	1.94
	Sediment	Pre-operational	163	5.36	6.80	0.01	0.23	1.30	3.11	7.88	15.00	49.50	6.58
		Operational	157	6.53	9.67	0.08	0.17	1.42	3.92	8.45	20.11	96.90	7.03
	Soil	Pre-operational	34	2.38	4.18	0.11	0.13	0.36	0.91	1.62	16.33	17.00	1.25
		Operational	34	5.20	7.58	0.20	0.20	1.10	2.08	5.99	27.90	34.20	4.89
1,2,3,7,8-PeCDD	Fish	Pre-operational	217	1.39	3.73	0.02	0.03	0.09	0.23	1.07	7.29	29.00	0.98
		Operational	267	1.08	1.91	0.02	0.05	0.18	0.40	0.97	5.30	15.20	0.79
	Sediment	Pre-operational	161	3.49	6.31	0.03	0.17	0.65	1.85	3.60	9.37	48.70	2.94
		Operational	157	4.08	8.05	0.04	0.13	0.52	1.73	5.53	11.10	87.80	5.01
	Soil	Pre-operational	34	0.87	1.28	0.03	0.03	0.20	0.39	0.92	5.18	5.23	0.72
		Operational	34	1.54	1.97	0.02	0.03	0.46	0.67	2.02	7.11	8.48	1.56
1,2,3,4,7,8-HxCDF	Fish	Pre-operational	220	7.15	28.44	0.03	0.06	0.09	0.13	0.42	40.83	218.0	0.34
		Operational	268	0.53	0.92	0.02	0.03	0.08	0.20	0.49	2.63	7.92	0.41
	Sediment	Pre-operational	166	16.12	25.53	0.03	1.46	3.85	8.93	18.95	50.37	193.0	15.10
		Operational	157	16.73	28.64	0.05	0.33	2.75	7.78	23.30	48.77	277.0	20.56
	Soil	Pre-operational	35	4.84	8.78	0.21	0.47	1.19	2.07	3.83	23.56	49.00	2.64
		Operational	34	11.00	21.91	0.27	0.33	1.99	3.63	10.18	75.23	117.0	8.20
1,2,3,6,7,8-HxCDF	Fish	Pre-operational	220	0.27	0.57	0.03	0.04	0.07	0.09	0.15	1.35	5.69	0.08
		Operational	268	0.49	0.89	0.01	0.03	0.08	0.16	0.42	2.73	7.01	0.34
	Sediment	Pre-operational	166	6.95	9.15	0.01	0.70	2.11	4.67	8.37	19.02	66.70	6.26
		Operational	157	7.92	10.83	0.07	0.32	1.30	4.00	11.75	22.84	94.40	10.45
	Soil	Pre-operational	35	2.32	3.77	0.20	0.21	0.52	0.76	2.05	14.76	17.80	1.53
		Operational	34	4.22	6.13	0.13	0.17	0.67	2.03	4.28	21.68	29.10	3.42

Table 7 (continued)

Analyte	Matrix	RRF Status	No. of samples	Mean	Standard deviation	Minimum	Percentiles					Maximum	Midrange*
							5th	25th	Median or 50th	75th	95th		
2,3,4,6,7,8-HxCDF	Fish	Pre-operational	220	0.57	1.62	0.03	0.04	0.08	0.10	0.28	2.09	14.60	0.20
		Operational	268	1.20	3.55	0.01	0.03	0.08	0.21	0.49	8.87	35.10	0.41
	Sediment	Pre-operational	166	8.41	10.50	0.11	0.69	2.21	5.33	10.90	30.09	71.10	8.70
		Operational	157	10.01	14.71	0.12	0.34	1.73	5.03	13.40	35.13	134.0	11.68
	Soil	Pre-operational	35	2.54	3.84	0.21	0.26	0.66	1.30	2.73	15.18	18.70	2.07
		Operational	34	6.53	10.10	0.25	0.27	1.26	2.96	6.54	34.63	48.20	5.28
1,2,3,7,8,9-HxCDF	Fish	Pre-operational	220	1.21	5.31	0.02	0.03	0.03	0.08	0.10	7.47	45.50	0.06
		Operational	268	0.07	0.09	0.02	0.02	0.03	0.07	0.08	0.15	1.15	0.05
	Sediment	Pre-operational	166	1.28	1.88	0.01	0.07	0.25	0.46	1.66	5.01	10.30	1.40
		Operational	157	0.81	1.03	0.01	0.07	0.25	0.49	0.88	3.35	6.40	0.64
	Soil	Pre-operational	35	0.58	0.95	0.04	0.05	0.08	0.17	0.58	3.98	4.02	0.50
		Operational	34	0.35	0.45	0.02	0.03	0.07	0.16	0.51	1.45	2.24	0.44
1,2,3,4,7,8-HxCDD	Fish	Pre-operational	221	0.30	0.44	0.02	0.03	0.07	0.13	0.28	1.22	2.46	0.21
		Operational	268	0.45	0.71	0.02	0.03	0.07	0.19	0.45	2.10	4.74	0.38
	Sediment	Pre-operational	166	4.24	5.38	0.05	0.27	0.95	2.60	4.94	15.24	31.60	3.99
		Operational	156	4.43	5.63	0.01	0.20	0.52	2.14	6.53	14.56	42.00	6.01
	Soil	Pre-operational	35	1.29	1.76	0.08	0.09	0.29	0.60	1.60	6.66	8.49	1.31
		Operational	34	1.59	2.13	0.03	0.03	0.36	0.92	1.97	8.06	10.20	1.61
1,2,3,6,7,8-HxCDD	Fish	Pre-operational	221	1.04	1.89	0.02	0.06	0.08	0.33	0.92	5.02	10.80	0.84
		Operational	268	2.06	4.01	0.02	0.05	0.20	0.62	2.09	9.92	36.30	1.89
	Sediment	Pre-operational	166	15.26	20.55	0.10	0.91	3.07	6.86	18.07	73.39	107.0	15.00
		Operational	157	17.50	21.38	0.18	0.39	2.37	8.42	27.70	58.43	150.0	25.33
	Soil	Pre-operational	35	2.66	3.66	0.25	0.29	0.57	1.13	3.62	14.42	17.30	3.05
		Operational	34	4.88	6.32	0.22	0.27	1.38	2.85	4.83	24.35	26.90	3.45
1,2,3,7,8,9-HxCDD	Fish	Pre-operational	221	0.24	0.45	0.02	0.05	0.07	0.09	0.19	1.08	5.10	0.12
		Operational	268	0.37	0.81	0.02	0.02	0.07	0.14	0.37	1.64	4.80	0.29
	Sediment	Pre-operational	166	11.24	15.09	0.09	0.95	2.59	6.07	12.07	35.40	98.30	9.49
		Operational	157	12.88	16.77	0.07	0.33	2.21	6.59	17.60	39.83	146.0	15.39
	Soil	Pre-operational	35	2.75	3.57	0.28	0.32	0.80	1.12	3.46	14.58	15.70	2.66
		Operational	34	4.60	5.54	0.09	0.17	1.31	2.38	5.49	20.37	20.60	4.18

Table 7 (continued)

Analyte	Matrix	RRF Status	No. of samples	Mean	Standard deviation	Minimum	Percentiles					Maximum	Midrange*
							5th	25th	Median or 50th	75th	95th		
1,2,3,4,6,7,8-HpCDF	Fish	Pre-operational	220	0.62	1.57	0.03	0.06	0.08	0.15	0.44	2.75	15.50	0.36
		Operational	268	0.57	0.78	0.02	0.06	0.12	0.26	0.60	2.36	4.20	0.47
	Sediment	Pre-operational	166	93.58	141.6	0.23	4.85	19.95	43.15	101.3	400.5	1,146	81.30
		Operational	157	98.63	150.3	0.51	2.74	16.25	41.10	137.5	286.8	1,100	121.3
	Soil	Pre-operational	35	17.00	22.98	1.01	1.13	4.14	8.76	20.90	89.16	105.0	18.76
		Operational	34	32.92	42.40	1.56	1.63	7.17	19.65	40.85	154.0	202.0	33.68
1,2,3,4,7,8,9-HpCDF	Fish	Pre-operational	220	0.11	0.24	0.02	0.02	0.04	0.07	0.08	0.36	2.80	0.04
		Operational	268	0.09	0.09	0.02	0.02	0.03	0.07	0.08	0.28	0.90	0.05
	Sediment	Pre-operational	166	6.52	17.21	0.07	0.36	1.19	3.13	6.03	16.70	205.0	4.84
		Operational	157	6.40	6.19	0.11	0.32	1.12	3.18	9.60	20.24	70.50	8.49
	Soil	Pre-operational	35	1.21	1.84	0.03	0.03	0.31	0.57	1.46	6.30	9.70	1.15
		Operational	34	2.68	4.19	0.07	0.12	0.54	1.19	2.94	15.87	20.60	2.40
1,2,3,4,6,7,8-HpCDD	Fish	Pre-operational	221	2.95	7.47	0.08	0.17	0.34	0.94	1.85	17.72	76.50	1.52
		Operational	268	9.16	38.26	0.13	0.23	0.71	1.40	3.70	26.03	419.0	2.99
	Sediment	Pre-operational	166	306.9	416.5	0.94	13.48	60.73	125.0	342.8	1,310	2,680	282.0
		Operational	157	371.7	465.8	2.02	11.77	44.25	170.0	564.5	1,407	2,330	520.3
	Soil	Pre-operational	35	55.26	64.61	2.34	5.47	12.50	28.50	76.50	239.6	270.0	64.00
		Operational	34	100.8	113.0	3.64	4.49	26.88	55.80	128.8	395.0	398.0	101.9
OCDF	Fish	Pre-operational	220	0.26	0.29	0.04	0.06	0.13	0.15	0.28	0.94	1.70	0.15
		Operational	268	0.37	0.60	0.04	0.06	0.13	0.17	0.36	1.25	5.00	0.23
	Sediment	Pre-operational	166	155.2	206.7	0.41	5.18	31.65	69.50	218.0	658.4	1,000	186.4
		Operational	155	184.6	235.7	1.13	3.74	22.80	70.50	300.0	643.4	1,480	277.2
	Soil	Pre-operational	35	25.73	31.53	1.15	1.77	5.23	11.50	41.50	116.0	116.0	36.27
		Operational	34	41.90	46.34	1.64	1.91	9.88	26.55	49.05	159.5	212.0	39.17
OCDD	Fish	Pre-operational	220	12.70	55.15	0.08	0.73	1.13	2.06	6.37	43.97	568.0	5.24
		Operational	268	74.38	416.6	0.59	0.88	1.84	4.23	10.97	49.03	4,450	9.14
	Sediment	Pre-operational	166	2,131	2,215	7.70	137.3	495.0	1,155	3,066	7,409	9,170	2,573
		Operational	155	2,758	2,974	16.70	115.2	632.0	1,680	4,010	9,340	16,700	3,378
	Soil	Pre-operational	35	814.3	1,128	20.40	33.84	112.0	369.0	1,120	3,970	4,970	1,008
		Operational	34	2,027	2,067	19.40	29.45	271.8	970.0	3,398	6,278	8,540	3,126

\* Midrange: Concentration range covering middle 50% of data (75th - 25th percentile).

\* Fish: All concentrations in pg/g wet weight.

\* Sediment: All concentrations in pg/g dry weight.

\* Soil: All concentrations in pg/g dry weight.



In each row of Table 7, the median is lower than the mean, indicating positive skewness of the distributions of the concentration levels (i.e., longer tails to the right). This can also be observed in the 25th and 75th percentiles in each case. These values are such that the middle 50% of the concentration levels range between these two numbers. The range between these two percentiles is called the midrange and is shown in the last column of Table 7.

For example, during pre-operational status, the middle 50% of 2,3,7,8-TCDD levels in fish range between 0.15 and 1.11 pg/g, covering a midrange of 0.97 pg/g. The median level, however, is 0.34 pg/g while the average level is 0.93 pg/g. The maximum level is 6.37 pg/g, clearly a large value when compared to the 95th percentile of 3.99 pg/g. A few large values influence the mean level but do not affect the median. The median is generally a better representation of the midpoint of a log normal distribution.

Concentration box plots for each analyte in each matrix were shown in the preliminary draft report (submitted to the Connecticut DEP in August 1991). These box plots (not shown in this report) provided a visual means of assessing the shape of the distribution of the analyte. The shapes were clearly skewed to the right, with most values on the low side, and a few large values. This is typical of environmental PCDD and PCDF levels and the shape of their distribution compares to that of the log-normal distribution. To take this shape into account when comparing the data, one therefore takes the natural logarithm of the concentration levels prior to statistical analyses. This approach has been taken throughout this report.

### 3.2 CORRELATIONS BETWEEN PAIRS OF ANALYTES

Correlation coefficients between each pair of analytes were computed, separately for fish (17 isomers only), sediment, and soil samples. All field samples, including those from Willimantic, were considered here since the correlation analysis respects the pairing of each two analytes from a given sample. The correlation coefficient is a measure of the mutual relationship between the levels of two analytes and can range between -1 and +1. A positive correlation indicates that the levels of two analytes increase or decrease simultaneously. A negative correlation indicates that as the level of one analyte increases, the level of the other decreases. Thus +1 and -1 indicate perfect correlation while a correlation close to zero denotes that no relationship exists between the levels of two analytes.

The correlation coefficients between analytes were calculated based on the log-transformed concentrations. The log-transformation was performed to improve on the skewness of the distributions of the analyte levels mentioned earlier. All correlation coefficients but one (-0.04) were positive. Tables 8, 9, and 10 list all correlation coefficients between pairs of analyte levels that are greater than or equal to 0.50, for fish, sediment, and soil samples, respectively.

The cutoff value of 0.50 for the correlation coefficients was selected for the following reason. The correlation coefficient,  $R$ , between two analyte levels,  $X$  and  $Y$ , is a measure of the linear dependency between  $X$  and  $Y$ . In the context of a linear regression of  $Y$  on  $X$ ,  $R^2$ , the square of the correlation coefficient, will be the proportion of total variation about the mean of  $Y$  explained by the regression of  $Y$  on  $X$ . Thus, setting the cutoff for  $R$  at 0.50 is equivalent to setting the cutoff for  $R^2$  at 25%. For all practical purposes, anything below that figure can be considered negligible in terms of linear association and predictive power.

**Table 8. CORRELATIONS<sup>a</sup> BETWEEN ALL PAIRS OF 17 ISOMERS BASED ON LOG CONCENTRATION LEVELS<sup>b</sup> IN FISH**

Analyte	Analyte No.	No. of samples	Analyte No.															
			9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
2,3,7,8-TCDF	9	550																
2,3,7,8-TCDD	10	543	0.51															
1,2,3,7,8-PeCDF	11	548	0.58	0.77														
2,3,4,7,8-PeCDF	12	548		0.78	0.71													
1,2,3,7,8-PeCDD	13	545		0.83	0.79	0.74												
1,2,3,4,7,8-HxCDF*	14	549		0.60	0.55		0.63											
1,2,3,6,7,8-HxCDF*	15	549		0.70	0.67	0.77	0.69	0.65										
2,3,4,6,7,8-HxCDF*	16	549		0.68	0.65	0.70	0.72	0.59	0.72									
1,2,3,7,8,9-HxCDF*	17	549																
1,2,3,4,7,8-HxCDD*	18	550		0.74	0.71	0.69	0.79	0.57	0.72	0.62								
1,2,3,6,7,8-HxCDD*	19	550		0.83	0.77	0.77	0.89	0.61	0.75	0.69		0.82						
1,2,3,7,8,9-HxCDD*	20	550		0.77	0.71	0.75	0.82	0.60	0.80	0.68		0.85	0.68					
1,2,3,4,6,7,8-HpCDF*	21	549		0.77	0.72	0.69	0.80	0.74	0.66	0.71		0.74	0.84	0.82				
1,2,3,4,7,8,9-HpCDF*	22	549							0.51	0.55	0.53			0.52	0.50			
1,2,3,4,6,7,8-HpCDD	23	550		0.75	0.70	0.71	0.77	0.54	0.70	0.59		0.79	0.87	0.88	0.83			
OCDF*	24	549		0.56	0.52		0.54		0.51			0.56	0.81	0.65	0.67	0.51	0.68	
OCDD*	25	549		0.53		0.52	0.53		0.51			0.56	0.61	0.66	0.84		0.85	0.66
Minimum correlation			0.04	0.23	0.17	0.02	0.26	0.38	0.01	0.39	-0.04	0.16	0.19	0.17	0.31	0.28	0.08	0.21
Maximum correlation			0.58	0.83	0.79	0.78	0.89	0.74	0.80	0.72	0.59	0.85	0.89	0.88	0.84	0.55	0.88	0.68

<sup>a</sup> Analytes with high occurrences of nondetects (over 25% during either pre-operational or operational status of the RRF).

<sup>b</sup> Only correlation coefficients above 0.50 are shown.

<sup>c</sup> All levels below LOD were replaced with 1/2 LOD.

<sup>d</sup> Correlation range for OCDD (Analyte No. 25) is -0.04 to 0.85.

**Table 9. CORRELATIONS<sup>a</sup> BETWEEN ALL PAIRS OF ANALYTES BASED ON LOG CONCENTRATION LEVELS<sup>b</sup> IN SEDIMENTS**

Analyte	Analyte No.	No. of samples	Analyte No.							
			1	2	3	4	5	6	7	8
Total TCDF	1	316								
Total TCDD	2	338	0.84							
Total PeCDF	3	339	0.88	0.73						
Total PeCDD	4	338	0.81	0.75	0.90					
Total HxCDF	5	342	0.80	0.67	0.90	0.91				
Total HxCDD	6	342	0.78	0.64	0.85	0.91	0.98			
Total HpCDF	7	342	0.70	0.60	0.78	0.85	0.93	0.95		
Total HpCDD	8	342	0.63	0.53	0.72	0.82	0.90	0.95	0.97	
2,3,7,8-TCDF	9	316	0.93	0.79	0.83	0.83	0.82	0.83	0.79	0.75
2,3,7,8-TCDD <sup>a</sup>	10	338	0.59	0.60	0.58	0.57	0.63	0.62	0.63	0.59
1,2,3,7,8-PeCDF	11	339	0.87	0.70	0.85	0.84	0.85	0.86	0.80	0.77
2,3,4,7,8-PeCDF	12	339	0.79	0.60	0.80	0.78	0.75	0.73	0.66	0.62
1,2,3,7,8-PeCDD	13	337	0.76	0.66	0.84	0.90	0.91	0.91	0.86	0.84
1,2,3,4,7,8-HxCDF	14	342	0.87	0.72	0.89	0.89	0.92	0.91	0.86	0.83
1,2,3,6,7,8-HxCDF	15	342	0.86	0.69	0.88	0.89	0.93	0.92	0.86	0.85
2,3,4,6,7,8-HxCDF	16	342	0.80	0.63	0.88	0.86	0.91	0.88	0.83	0.80
1,2,3,7,8,9-HxCDF <sup>a</sup>	17	342	0.56		0.53	0.52	0.56	0.55	0.55	0.55
1,2,3,4,7,8-HxCDD	18	341	0.70	0.57	0.71	0.78	0.82	0.83	0.82	0.83
1,2,3,6,7,8-HxCDD	19	342	0.68	0.59	0.78	0.85	0.93	0.95	0.95	0.95
1,2,3,7,8,9-HxCDD	20	342	0.78	0.64	0.83	0.90	0.94	0.97	0.95	0.94
1,2,3,4,6,7,8-HpCDF	21	342	0.77	0.65	0.81	0.86	0.94	0.95	0.98	0.95
1,2,3,4,7,8,9-HpCDF	22	342	0.77	0.83	0.81	0.85	0.93	0.93	0.93	0.91
1,2,3,4,6,7,8-HpCDD	23	342	0.82	0.54	0.71	0.81	0.90	0.94	0.97	1.00
OCDF	24	340	0.69	0.58	0.74	0.81	0.89	0.93	0.97	0.96
OCDD	25	340	0.53		0.65	0.75	0.84	0.86	0.86	0.94
Minimum correlation			0.53	0.45	0.53	0.52	0.58	0.55	0.55	0.53
Maximum correlation			0.93	0.79	0.90	0.91	0.96	0.97	0.98	1.00

Table 9 (continued)

Analyte	Analyte No.	No. of samples	Analyte No.															
			9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
2,3,7,8-TCDF	9	316																
2,3,7,8-TCDD <sup>a</sup>	10	338	0.62															
1,2,3,7,8-PeCDF	11	339	0.90	0.68														
2,3,4,7,8-PeCDF	12	339	0.77	0.53	0.85													
1,2,3,7,8-PeCDD	13	337	0.81	0.64	0.85	0.74												
1,2,3,4,7,8-HxCDF	14	342	0.89	0.66	0.91	0.79	0.90											
1,2,3,6,7,8-HxCDF	15	342	0.88	0.65	0.90	0.78	0.91	0.94										
2,3,4,6,7,8-HxCDF	16	342	0.83	0.66	0.87	0.76	0.90	0.91	0.92									
1,2,3,7,8,9-HxCDF <sup>a</sup>	17	342	0.57		0.53		0.54	0.60	0.59	0.51								
1,2,3,4,7,8-HxCDD	18	341	0.76	0.57	0.77	0.63	0.83	0.83	0.84	0.76	0.61							
1,2,3,6,7,8-HxCDD	19	342	0.75	0.63	0.79	0.66	0.87	0.86	0.88	0.84	0.54	0.80						
1,2,3,7,8,9-HxCDD	20	342	0.85	0.65	0.87	0.74	0.93	0.93	0.93	0.90	0.57	0.87	0.95					
1,2,3,4,6,7,8-HpCDF	21	342	0.85	0.67	0.85	0.70	0.88	0.90	0.91	0.86	0.59	0.84	0.94	0.96				
1,2,3,4,7,8,9-HpCDF	22	342	0.63	0.67	0.85	0.72	0.89	0.91	0.92	0.88	0.60	0.85	0.92	0.95	0.95			
1,2,3,4,6,7,8-HpCDD	23	342	0.73	0.60	0.75	0.60	0.83	0.82	0.84	0.78	0.56	0.82	0.95	0.94	0.95	0.91		
OCDF	24	340	0.78	0.61	0.80	0.65	0.83	0.84	0.85	0.80	0.55	0.82	0.91	0.93	0.97	0.91	0.96	
OCDD <sup>c</sup>	25	340	0.63	0.54	0.67	0.55	0.78	0.73	0.76	0.73		0.74	0.88	0.87	0.85	0.83	0.94	0.86
Minimum correlation			0.57	0.42	0.53	0.39	0.54	0.60	0.59	0.51	0.39	0.57	0.54	0.57	0.59	0.60	0.54	0.55
Maximum correlation			0.90	0.68	0.91	0.85	0.93	0.94	0.94	0.92	0.61	0.87	0.95	0.97	0.98	0.95	1.00	0.97

<sup>a</sup> Analytes with high occurrences of nondetects (over 25% during either pre-operational or operational status of the RRF).

<sup>b</sup> Only correlation coefficients above 0.50 are shown.

<sup>b</sup> All levels below LOD were replaced with 1/2 LOD.

<sup>c</sup> Correlation range for OCDD (Analyte No. 25) is 0.45 to 0.94.

**Table 10. CORRELATIONS<sup>a</sup> BETWEEN ALL PAIRS OF ANALYTES BASED ON LOG CONCENTRATION LEVELS<sup>b</sup> IN SOILS**

Analyte	Analyte No.	No. of samples	Analyte No.							
			1	2	3	4	5	6	7	8
Total TCDF	1	76								
Total TCDD	2	76	0.83							
Total PeCDF	3	76	0.92	0.76						
Total PeCDD	4	76	0.94	0.79	0.91					
Total HxCDF	5	77	0.88	0.70	0.98	0.90				
Total HxCDD	6	77	0.85	0.89	0.93	0.88	0.95			
Total HpCDF	7	77	0.82	0.64	0.89	0.86	0.95	0.93		
Total HpCDD	8	77	0.79	0.65	0.87	0.84	0.90	0.96	0.90	
2,3,7,8-TCDF	9	76	0.92	0.80	0.88	0.91	0.86	0.87	0.82	0.82
2,3,7,8-TCDD <sup>a</sup>	10	78								
1,2,3,7,8-PeCDF	11	76	0.91	0.75	0.93	0.93	0.91	0.92	0.85	0.86
2,3,4,7,8-PeCDF	12	76	0.87	0.79	0.90	0.88	0.88	0.85	0.81	0.81
1,2,3,7,8-PeCDD	13	76	0.92	0.74	0.92	0.95	0.94	0.94	0.92	0.90
1,2,3,4,7,8-HxCDF	14	77	0.90	0.78	0.93	0.92	0.93	0.93	0.89	0.87
1,2,3,6,7,8-HxCDF	15	77	0.90	0.73	0.96	0.92	0.97	0.94	0.92	0.88
2,3,4,6,7,8-HxCDF	16	77	0.84	0.67	0.94	0.88	0.95	0.93	0.90	0.88
1,2,3,7,8,9-HxCDF <sup>a</sup>	17	77	0.52		0.59	0.56	0.60	0.59	0.54	0.55
1,2,3,4,7,8-HxCDD	18	77	0.73	0.65	0.71	0.73	0.75	0.75	0.73	0.78
1,2,3,6,7,8-HxCDD	19	77	0.79	0.61	0.89	0.85	0.94	0.95	0.95	0.94
1,2,3,7,8,9-HxCDD	20	77	0.85	0.69	0.91	0.89	0.94	0.97	0.93	0.95
1,2,3,4,6,7,8-HpCDF	21	77	0.83	0.66	0.91	0.87	0.96	0.94	0.99	0.91
1,2,3,4,7,8,9-HpCDF	22	77	0.82	0.69	0.84	0.86	0.88	0.86	0.93	0.83
1,2,3,4,6,7,8-HpCDD	23	77	0.79	0.65	0.87	0.84	0.91	0.96	0.92	1.00
OCDF	24	77	0.76	0.59	0.83	0.79	0.89	0.88	0.94	0.88
OCDD	25	77	0.61	0.55	0.64	0.61	0.64	0.72	0.61	0.65
Minimum correlation			0.37	0.35	0.40	0.42	0.39	0.42	0.39	0.42
Maximum correlation			0.94	0.83	0.98	0.95	0.98	0.97	0.99	1.00

Table 10 (continued)

Analyte	Analyte No.	No. of samples	Analyte No.															
			9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
2,3,7,8-TCDF	9	76																
2,3,7,8-TCDD*	10	76																
1,2,3,7,8-PeCDF	11	76	0.95															
2,3,4,7,8-PeCDF	12	76	0.87		0.90													
1,2,3,7,8-PeCDD	13	76	0.91		0.93	0.85												
1,2,3,4,7,8-HxCDF	14	77	0.94		0.97	0.91	0.94											
1,2,3,6,7,8-HxCDF	15	77	0.92		0.95	0.89	0.96	0.97										
2,3,4,6,7,8-HxCDF	16	77	0.88		0.93	0.88	0.92	0.95	0.97									
1,2,3,7,8,9-HxCDF*	17	77	0.58		0.62	0.53	0.57	0.62	0.66	0.62								
1,2,3,4,7,8-HxCDD	18	77	0.78		0.74	0.68	0.78	0.78	0.77	0.89	0.55							
1,2,3,6,7,8-HxCDD	19	77	0.82		0.88	0.81	0.92	0.88	0.93	0.93	0.59	0.71						
1,2,3,7,8,9-HxCDD	20	77	0.86		0.91	0.85	0.95	0.93	0.95	0.94	0.61	0.79	0.96					
1,2,3,4,6,7,8-HpCDF	21	77	0.86		0.89	0.84	0.93	0.92	0.95	0.93	0.58	0.74	0.96	0.94				
1,2,3,4,7,8,9-HpCDF	22	77	0.85		0.85	0.82	0.91	0.90	0.90	0.88	0.53	0.73	0.89	0.90	0.93			
1,2,3,4,6,7,8-HpCDD	23	77	0.81		0.85	0.81	0.90	0.86	0.89	0.88	0.55	0.78	0.95	0.95	0.93	0.84		
OCDF	24	77	0.75		0.80	0.75	0.86	0.83	0.85	0.85	0.53	0.70	0.89	0.88	0.94	0.87	0.90	
OCDD*	25	77	0.62		0.65	0.64	0.66	0.84	0.61	0.62		0.63	0.67	0.71	0.63	0.57	0.83	0.64
Minimum correlation			0.42	0.10	0.45	0.43	0.44	0.44	0.42	0.46	0.10	0.22	0.42	0.48	0.40	0.43	0.41	0.35
Maximum correlation			0.95	0.47	0.97	0.91	0.96	0.97	0.97	0.97	0.66	0.79	0.96	0.97	0.99	0.93	1.00	0.94

- \* Analytes with high occurrences of nondetects (over 25% during either pre-operational or operational status of the RRF).
- Only correlation coefficients above 0.50 are shown.
- All levels below LOD were replaced with 1/2 LOD.
- Correlation range for OCDD (Analyte No. 25) is 0.31 to 0.85.

To read these tables, choose any two analytes (any row and any column); the header of each column corresponds to the analyte name and number shown in the first two columns; the number at the intersection of a row and column is the correlation between the log-concentration levels of these two analytes. For example in Table 8, the correlation between 2,3,7,8-TCDD (row, Analyte No. 10) and 2,3,7,8-TCDF (column, Analyte No. 9) is 0.51. Since a correlation matrix is symmetrical about its diagonal, we chose to only show the lower left half of the matrix.

The last two rows in each table list the minimum and maximum correlations of a given analyte (column) with all the remaining analytes. These two statistics encompass all correlations in each column, including those below 0.50 not shown in the tables. They provide a summary of overall correlations in the data sets. For example, the correlations between log-concentration levels of 2,3,7,8-TCDD (Analyte No. 10) with all other 16 isomers range between 0.23 and 0.83 in fish samples (Table 8). Note that the maximum and minimum correlations between OCDD and all other analytes are provided in a footnote to the tables due to table configuration.

As shown in Tables 8 to 10, the correlations between analyte levels (logs) are generally higher in soil and sediments samples than in fish samples. For example, the correlation coefficients between analytes in the soil samples are all very high (most greater than 0.80), indicating that a few analytes are sufficient to "fingerprint" soil samples with respect to the 25 analytes investigated. However, 2,3,7,8-TCDD in soil does not correlate well with any of the other analytes (correlation between 0.10 and 0.47). This fact is less pronounced in the sediment samples. As Table 8 shows, the correlation coefficients above 0.50 become less frequent for fish samples, with only a few coefficients in the high range. Thus to "fingerprint" fish samples with respect to these analytes, measurements on a large number of analytes will be necessary.



The correlation coefficients between pairs of analytes (log-transformed concentration level) form the basis for the cluster analysis of analyte levels performed in Section 5.

### **3.3 SIMPLE DESCRIPTIVE STATISTICS OF PHYSICAL SAMPLE PROPERTIES**

Correlations between analyte levels and other sample properties were computed to investigate possible relationships between concentration levels and percent lipid in fish, and total organic carbon and particle size in sediment and soil samples. All concentrations were log-transformed prior to these correlation analyses, and particle size is as defined previously (Subsection 2.2). The resulting correlation coefficients are shown in Table 11. The first five rows in this table list the number of samples for which data were available, the mean and standard deviation, as well as the smallest and largest of each measurement.

#### **3.3.1 Correlations Between Concentration Levels (Logs) and Lipid Content in Fish**

Correlations between analyte levels (logs) and lipid content (% lipid) are all positive but generally low, ranging from 0.16 to 0.64. In light of the earlier discussion, these correlations would correspond to percent variability explained by means of a regression line of between 3% and 41%. Their statistical significance is due mainly to the large sample size (550 samples).

**Table 11. CORRELATIONS BETWEEN LOG CONCENTRATION AND  
LIPID CONTENT (FISH), AND TOC AND  
PARTICLE SIZE (SEDIMENT AND SOIL)**

Analyte	Fish	Sediment		Soil	
	Lipid Content (%)	Total organic carbon (mg/kg)	Particle <sup>a</sup> size (mm)	Total organic carbon (mg/kg)	Particle size (mm)
No. of samples	550	343	335	77	68
Mean	5.57	98,236	0.482	47,414	0.737
Standard deviation	3.87	98,439	0.268	31,244	0.131
Minimum	0.00	400	0.055	7,200	0.469
Maximum	22.18	750,000	1.03	140,000	1.02
Total TCDF		0.43	-0.45	0.40	-0.46
Total TCDD		0.31	-0.42	0.42	-0.39
Total PeCDF		0.28	-0.45	0.38	-0.50
Total PeCDD		0.23	-0.46	0.48	-0.50
Total HxCDF		0.20	-0.54	0.39	-0.47
Total HxCDD		0.16	-0.55	0.44	-0.49
Total HpCDF		0.13	-0.61	0.41	-0.44
Total HpCDD		0.09 <sup>b</sup>	-0.61	0.40	-0.53
2,3,7,8-TCDF	0.60	0.40	-0.52	0.47	-0.44
2,3,7,8-TCDD	0.64	0.25	-0.43	0.17 <sup>b</sup>	-0.13 <sup>b</sup>
1,2,3,7,8-PeCDF	0.61	0.37	-0.47	0.48	-0.47
2,3,4,7,8-PeCDF	0.52	0.37	-0.36	0.34	-0.51
1,2,3,7,8-PeCDD	0.55	0.24	-0.47	0.48	-0.50
1,2,3,4,7,8-HxCDF	0.39	0.34	-0.53	0.49	-0.46
1,2,3,6,7,8-HxCDF	0.46	0.29	-0.53	0.44	-0.45
2,3,4,6,7,8-HxCDF	0.42	0.24	-0.43	0.41	-0.46
1,2,3,7,8,9-HxCDF	0.16	0.33	-0.40	0.29	-0.37
1,2,3,4,7,8-HxCDD	0.50	0.27	-0.54	0.37	-0.48
1,2,3,6,7,8-HxCDD	0.58	0.11	-0.56	0.41	-0.45
1,2,3,7,8,9-HxCDD	0.54	0.21	-0.56	0.45	-0.52
1,2,3,4,6,7,8-HpCDF	0.48	0.19	-0.60	0.43	-0.44
1,2,3,4,7,8,9-HpCDF	0.31	0.21	-0.57	0.47	-0.42
1,2,3,4,6,7,8-HpCDD	0.53	0.09 <sup>b</sup>	-0.61	0.40	-0.52
OCDF	0.34	0.12	-0.60	0.47	-0.47
OCDD	0.29	0.05 <sup>b</sup>	-0.56	0.32	-0.58

<sup>a</sup> Average weighted particle size based on percent sand, silt, and clay.

<sup>b</sup> Correlation not significant at the 5% level.

### **3.3.2 Correlations Between Concentration Levels (Logs) and TOC and Particle Size of Sediment and Soil**

Correlations between log concentration levels and TOC are all positive, though low. That is, the analyte levels increase with increasing TOC; however, the relationship between the two is weak, as shown by the low correlations for either sediment or soil, ranging from 0.05 to 0.43 in sediment and from 0.17 to 0.49 in soil.

The negative correlations between analyte levels (logs) and particle size underline the inverse relationship between concentration levels and particle size: the larger the particle, the higher the level. However, these correlations, although statistically significant, are relatively low, ranging from -0.61 to -0.36 in sediment and from -0.58 to -0.13 in soil.

### **3.3.3 Relationship between Fish Species and Lipid Content**

An analysis of variance was performed on the percent lipid content in fish samples to investigate the relationship between fish species and their lipid content. This analysis showed that percent lipid varies significantly between fish species. The six fish species could be clearly separated into three groups:

1. Brown bullhead (99 samples, average percent lipid of 2.05%);
2. Yellow perch (115 samples, average percent lipid of 5.05%) and white sucker (190 samples, average percent lipid of 5.58%) ; and

3. White catfish (20 samples, average percent lipid of 7.82%), channel catfish (20 samples, average percent lipid of 8.38%), and carp (106 samples, average percent lipid of 8.48%).

*This grouping will be used in subsequent multivariate analyses.*

## **SECTION 4**

### **MULTIVARIATE STATISTICAL METHODS**

This section presents a brief overview of the multivariate techniques used in Section 5. To this point, simple statistics such as means, medians, standard deviations, and frequencies of all the available variables have been discussed. These descriptive statistics provide an efficient summary of the data space, but only on an individual variable basis. To show relationships between any pair of variables, if they exist, sample correlation coefficients are needed. The limitations of these two-way relationships are immediately obvious, however. For example, if one were to look at all two-way relationships between the 17 individual isomers on the one hand and the physical properties, collection year, and sample location on the other, plotting and analyzing would require as many as 510 bivariate graphs. Nor would these relationships include the 136 two-way plots between pairs of isomers. Not only would it be a large analytical task, but most important, relationships between more than two variables would never be considered. Thus, the objective is to be able to consider sets of variables simultaneously. The problem then becomes one of dimensionality; namely, with more than three variables, we are practically unable to visualize any relationship in space.

Multivariate techniques, or pattern recognition techniques, have been developed and provide rules for combining variables in an optimal way (the criterion of optimality varies from one technique to another) so as to reduce the dimensionality of the set of variables with as little loss of information as possible.

In this context, two statistical techniques have been used to complement each other; cluster analysis and principal component analysis.

There are three major objectives in pattern recognition: data reduction, feature extraction, and classification. In cluster analysis, in which a priori knowledge of possible grouping of variables (chemical parameters and physical properties) is required, natural groupings and structure among variables are studied without prejudice. The same technique can be applied to the samples in the study. In principal component analysis, the goal is to define linear combinations of the original variables and thus to reduce the dimension of the data space. The Biomedical Programs (BMDP) and the Statistical Analysis System (SAS) software packages have been used exclusively in this study.

#### **4.1 CLUSTER ANALYSIS**

Given a large set of variables, multivariate relationships are sought instead of the simple bivariate relationships found with correlation analysis techniques. Cluster analysis is applied to identify similarities between analytes, not between samples. Analytes contributing nearly the same information to the data structure are clustered; one can then select one or more analytes from a cluster, discard others if desired, and proceed. Cluster analysis thus shows whether there are natural groups of analytes with similar behavior.

The first step in cluster analysis, then, is to define a measure of association for clustering variables. In this study, the correlation coefficient serves as this measure. The single linkage option in the BMDP P1M procedure was selected for the clustering. This linkage process continues until all variables have been included in a single large cluster. The clustering results are then

printed in a dendrogram or tree diagram. The determination of the final number of clusters is, however, left to the user and depends on a chosen minimum similarity value (the correlation coefficient in this case). If the value chosen is too high (i.e., close to 1), then many clusters will be formed, and most of them might be single-point clusters. Because outputs provided by the classical statistical software packages show results after complete clustering, a decision as to an optimum similarity value can be made a posteriori. This cluster analysis technique was applied to the 25 analytes. All concentration data have been log-transformed prior to cluster analysis to improve on the skewness of the distributions of analyte results.

## **4.2 PRINCIPAL COMPONENT ANALYSIS**

Principal component analysis (PCA) is an effective tool for data reduction; and, at the same time, it provides a means for data representation in two- or three-dimensional space. In PCA, the goal is to assess the structure of the variables within a given set of variables, independently of any relationship they may have to variables outside this set.

The first step in PCA is to choose the chemical variables and physical properties to be included in the set and a measure of association between them. In order to treat all variables equally, we have chosen the correlation matrix as the measure of association (i.e., the data were autoscaled). Again, all concentration data have been log-transformed for PCA to improve on the skewness of the distributions of analyte results.

The original variables are combined into linear combinations in a manner that accounts for the maximum amount of variance present in the data. If the starting set of variables contains  $m$  variables, then  $m$  principal components will

be generated. The first principal component chosen is that which represents the single best linear combination of variables. In other words, it accounts for the highest percentage of the total variance in the data. The second principal component is chosen to account for as much of the remainder of variance as possible. Hence, in PCA, the principal components are orthogonal to each other, which means they are uncorrelated. This process continues until  $m$  linear combinations are found.

In general, a few principal components are sufficient to explain a large amount of the variance in the data. The importance of a principal component is mostly judged by its associated eigenvalue in the correlation matrix. Eigenvalues decrease with decreasing importance of the principal components. Past experience indicates that principal components with eigenvalues below 1.0 explain little correlated behavior among the measured variables and that they often reflect random variance contributions due to measurement and sampling error. Therefore, principal components with eigenvalues below 1.0 are generally discarded. A second criteria in the selection of the final number of principal components is the final communality estimates associated with each variable. Final communality estimates are a measure of how well the variables are accounted for by the number of principal components selected. Thus, an additional component, although having a low eigenvalue, might be included in the final set.

The coefficients of the variables (or transformed variables) in the linear combinations are called loadings. They are a measure of the relative contribution of a variable within a principal component to the variance explained. In fact, the principal component loadings are the correlation coefficients between the original (or transformed) variables and the principal components, and thus provide the key information about the nature of the principal component. These loadings can be used to order the variables within a given principal component



and help affix a "label" to this principal component. This step in the PCA is the most difficult because it is largely a matter of judgement. Interpretation of the results begins at this point.

Once the principal components have been derived and a final selection of significant principal components made, principal component scores for each data point (sample) are calculated from the standardized data. This is done by using the linear combinations of variables with their loadings on each principal component. Thus, if three principal components are retained, three scores will be computed for each sample, and three two-dimensional plots of the scores can be generated: principal component 2 vs. principal component 1, principal component 3 vs. principal component 1, and principal component 3 vs. principal component 2. One three-dimensional plot can also be created. Because the principal components are linear combinations of the original (transformed) variables, a principal component plot should contain more information about the data structure than any simple two-variable plot. The second most difficult step is then to give practical meaning to any pattern seen on the principal component plots. The analyst should take care not to oversimplify the relationships between variables; however, it is equally important not to read too much into the patterns found.

When plotting the two-or three-dimensional principal component plots, the data points can be labeled using available codes. These codes could be those representing selected categorical variables, for example, fish species, sampling location (town or pond), number of years resource recovery facility has been in operation, matrix type, or combinations of them. An additional approach is to plot only those scores lying between -3 and +3, thus including all but a few large values. This achieves a higher resolution of the plots so that possible patterns in the figures can be made more visible.

## **SECTION 5**

### **MULTIVARIATE ANALYSIS RESULTS**

Following the initial univariate and bivariate analyses of the data (simple descriptive statistics and correlations), a series of multivariate procedures, as described in Section 4, were applied. The same techniques were used to analyze various subsets of the data, depending on the analysis objective under investigation. Cluster analysis results for the chemical information are provided first, followed by those from the principal component analyses.

#### **5.1 CLUSTER ANALYSIS OF ANALYTES**

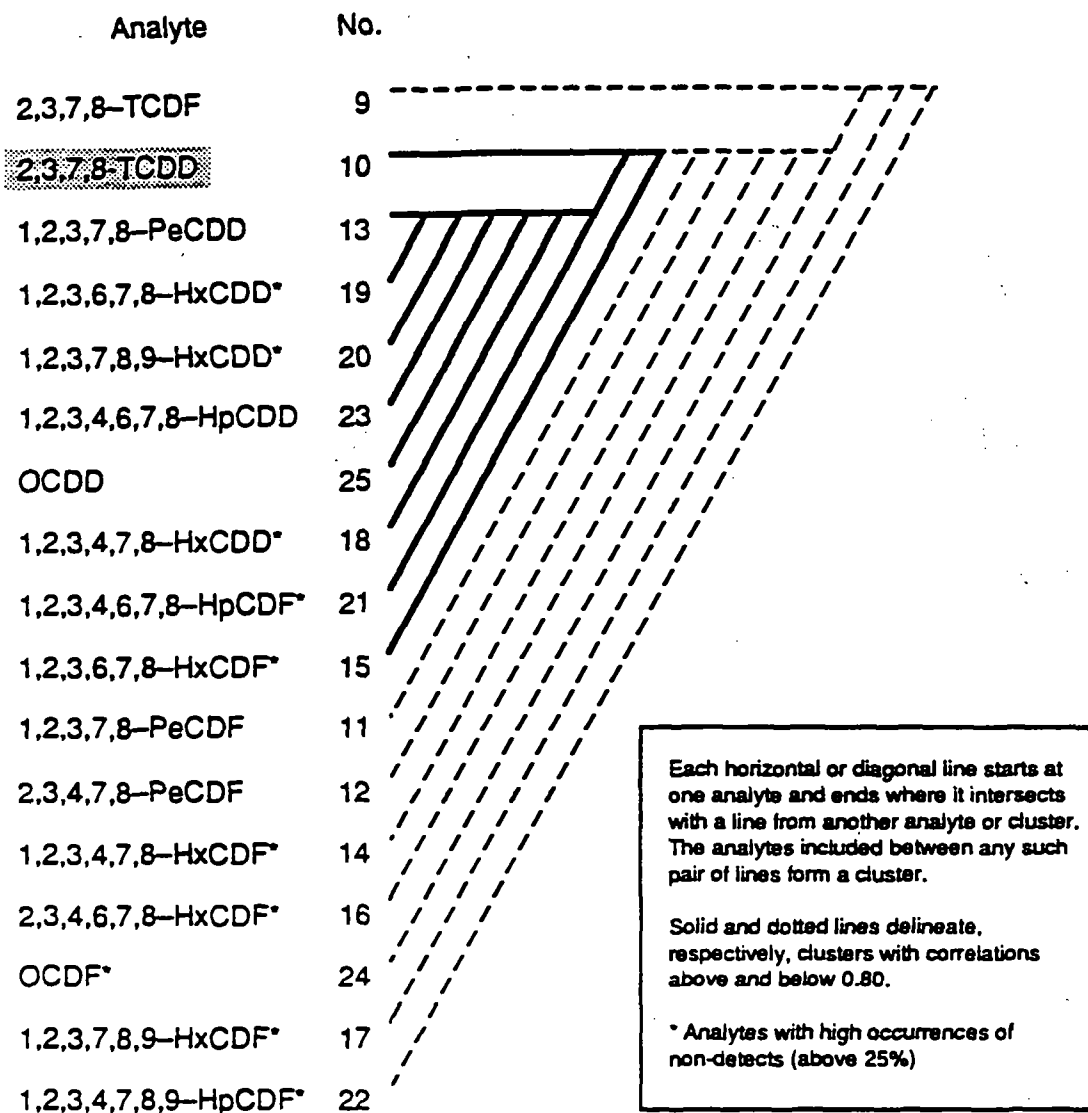
The objective of cluster analysis is to uncover possible significant interrelationships among analytes or groups of analytes. The input for the cluster analysis was the matrix of correlations between pairs of analyte levels (Tables 8, 9, and 10 for fish, sediment, and soil, respectively). Levels below detection limits were replaced by one-half the limit of detection. Since the detection limit for most analytes is well below typical values, the effect of this procedure for handling nondetects is minimal. All cluster analyses were performed on the log-transformed concentration levels to resolve two potential problems: (1) non-normality of the PCDD and PCDF concentration levels and (2) sensitivity of the cluster analysis to extreme concentration values.

Absolute correlations [in this case, simple correlations since all but one (value of -0.04) were positive] were used as the measure of similarity between any two analytes. When a cluster contained more than one analyte, the correlation between any two clusters was computed as the maximum correlation over all possible pairings of analytes between two clusters. At each step in the clustering process, the two clusters with the maximum correlation were combined. The process continued until all analytes were included into a single cluster (group). Cluster analysis was performed separately on log-concentration levels measured in fish, sediment, and soil samples. In all clustering analyses, fish concentrations are on a whole wet weight basis, while sediment and soil levels are on a dry weight basis.

#### **5.1.1 Isomer Patterns in Fish Samples**

The correlations between log-transformed levels of all pairs of the 17 isomers measured in fish were used in this analysis (see correlation matrix in Table 8). The clustering results are shown in Figure 4 in the form of a tree diagram. A companion table (Table 12) has been developed to show the correlation (degree of similarity) between two clusters at the time they are joined during the final clustering process. The isomer names (Column 1) and numbers (Column 2) are listed on the left side of the diagram in Figure 4. The tree diagram is printed over the correlation matrix and shows the clusters formed during the linkage steps. Solid lines (Table 12 and Figure 4) indicate boundaries of clusters such that the correlation between the last two clusters joined exceeds 0.80. Broken lines are used in all other cases.

The first and third columns in Table 12 list the two isomers (names and numbers) that form the cluster boundaries when a cluster is considered. The fourth column shows the number of isomers included in that cluster. The last column indicates the correlation at the time of clustering. For example, take



**Figure 4. Tree diagram showing clustering of all 17 isomers based on correlations of log-transformed concentration levels in fish.**

**Table 12. SUMMARY TABLE OF ISOMER CLUSTERS FORMED  
AND THEIR CORRELATIONS IN FISH SAMPLES**

Isomer	Isomer No.	Other boundary of cluster (Isomer No.)	No. of isomers in cluster	Correlation* when cluster formed
2,3,7,8-TCDF	9	22	17	0.54
2,3,7,8-TCDD	10	24	14	0.68
1,2,3,7,8-PeCDD	13	21	7	0.84
1,2,3,6,7,8-HxCDD*	19	13	2	0.89
1,2,3,7,8,9-HxCDD*	20	13	3	0.89
1,2,3,4,6,7,8-HpCDD	23	13	4	0.88
OCDD	25	13	5	0.86
1,2,3,4,7,8-HxCDD*	18	13	6	0.85
1,2,3,4,6,7,8-HpCDF*	21	10	8	0.84
1,2,3,6,7,8-HxCDF*	15	10	9	0.80
1,2,3,7,8-PeCDF	11	10	10	0.79
2,3,4,7,8-PeCDF	12	10	11	0.78
1,2,3,4,7,8-HxCDF*	14	10	12	0.76
2,3,4,6,7,8-HxCDF*	16	10	13	0.72
OCDF*	24	9	15	0.59
1,2,3,7,8,9-HxCDF*	17	9	16	0.59
1,2,3,4,7,8,9-HpCDF*	22	9	17	0.54

\* Isomers with high occurrences of nondetects (over 25% during either pre-operational or operational status of the RRF).

\* Clusters with correlations above 0.80 are between the two solid black lines. Results are based on log-transformed concentrations levels; nondetects are replaced by one-half the detection limit. (Results in Table 12 correspond to those shown in Figure 4).

1,2,3,6,7,8-HxCDD, isomer No. 19. A two-isomer cluster (the only one of size two) consisting of 1,2,3,6,7,8-HxCDD (No. 19) and 1,2,3,7,8-PeCDD (No. 13 in Column 3) is formed first in the clustering process with a correlation of 0.89 (last column).

A strongly correlated cluster of nine isomers is that bound by 1,2,3,6,7,8-HxCDF (isomer No. 15 in Column 2) and 2,3,7,8-TCDD (isomer No. 10 in Column 3), with an overall correlation of 0.80 (last column). This grouping is also reflected in Figure 4 by the solid lines connecting the isomers in this cluster. All seven dioxins and two furans are included in this group. However, five (marked by an \*\*\* in Table 12) of the nine isomers were not detected in a large proportion of fish samples (see Table 4).

No other subgroup exhibits an overall correlation greater than 0.80. The two penta furans (isomers Nos. 11 and 12) and two hexa furans (isomers Nos. 14 and 16) join the above mentioned cluster of nine isomers, one isomer at a time, yielding decreasing correlations from 0.79 to 0.72. Thus one could possibly consider one large cluster of 13 of the 17 isomers, with a final correlation of 0.72. The remaining four isomers—OCDF, 1,2,3,7,8,9-HxCDF, 1,2,3,4,7,8,9-HpCDF, and 2,3,7,8-TCDF (isomers Nos. 24, 17, 22, and 9, respectively, in Column 2 of Table 12)—behave each as single-isomer clusters with low correlations (0.59 to 0.54) with the above cluster of 13 isomers. It is worth mentioning that 2,3,7,8-TCDF is joined to the set during the very last clustering step, with a low correlation of 0.54 with the other 16 isomers.

### **5.1.2 Analyte Patterns in Sediment and Soil Samples**

A similar analysis was performed on the log-transformed concentration levels, separately in sediment and soil. In each case, all 25 analytes were

included in the analysis. The cluster analysis in sediment and soil operates on the correlation matrices shown in Tables 9 and 10 for sediment and soil, respectively.

The cluster results are presented in a similar fashion to those for fish. The clustering results for sediment are shown in Figure 5 (tree diagram) and in the companion table (Table 13) of correlations between clusters. The clustering results for soil are shown in Figure 6 (tree diagram) and in the matched similarity table (Table 14).

Both homolog totals and isomers were considered in the clustering process for soil and sediment. The tree diagrams in Figures 5 and 6 show clearly the high correlations between homolog totals and selected isomers (solid lines). These diagrams also show the similarities between the clustering of the analytes measured in both sediment and soil samples. Strongly correlated pairs of analytes (correlations above 0.97) in both sediment and soil samples are: Total HxCDD and 1,2,3,7,8,9-HxCDD (Nos. 6 and 20); Total HpCDF and 1,2,3,4,6,7,8-HpCDF (Nos. 7 and 21); and Total HpCDD and 1,2,3,4,6,7,8-HpCDD (Nos. 8 and 23).

For both sediment and soil samples, 21 of the 25 analytes can be clustered into one large group with an overall high correlation of 0.87 (sediment) and 0.85 (soil). The four analytes excluded from that group for each sample type are: Total TCDD, 2,3,7,8-TCDD, 1,2,3,7,8,9-HxCDF, and 1,2,3,4,7,8-HxCDD (Nos. 2, 10, 17, and 18, respectively). Of these four analytes, the levels of two were below detection limit in a large proportion of the samples: 2,3,7,8-TCDD (27% and 50% of the samples below LOD in sediment and soil, respectively) and 1,2,3,7,8,9-HxCDF (59% and 51% of the samples below LOD in sediment and soil, respectively). For both sediment and soil, 2,3,7,8-TCDD does not correlated

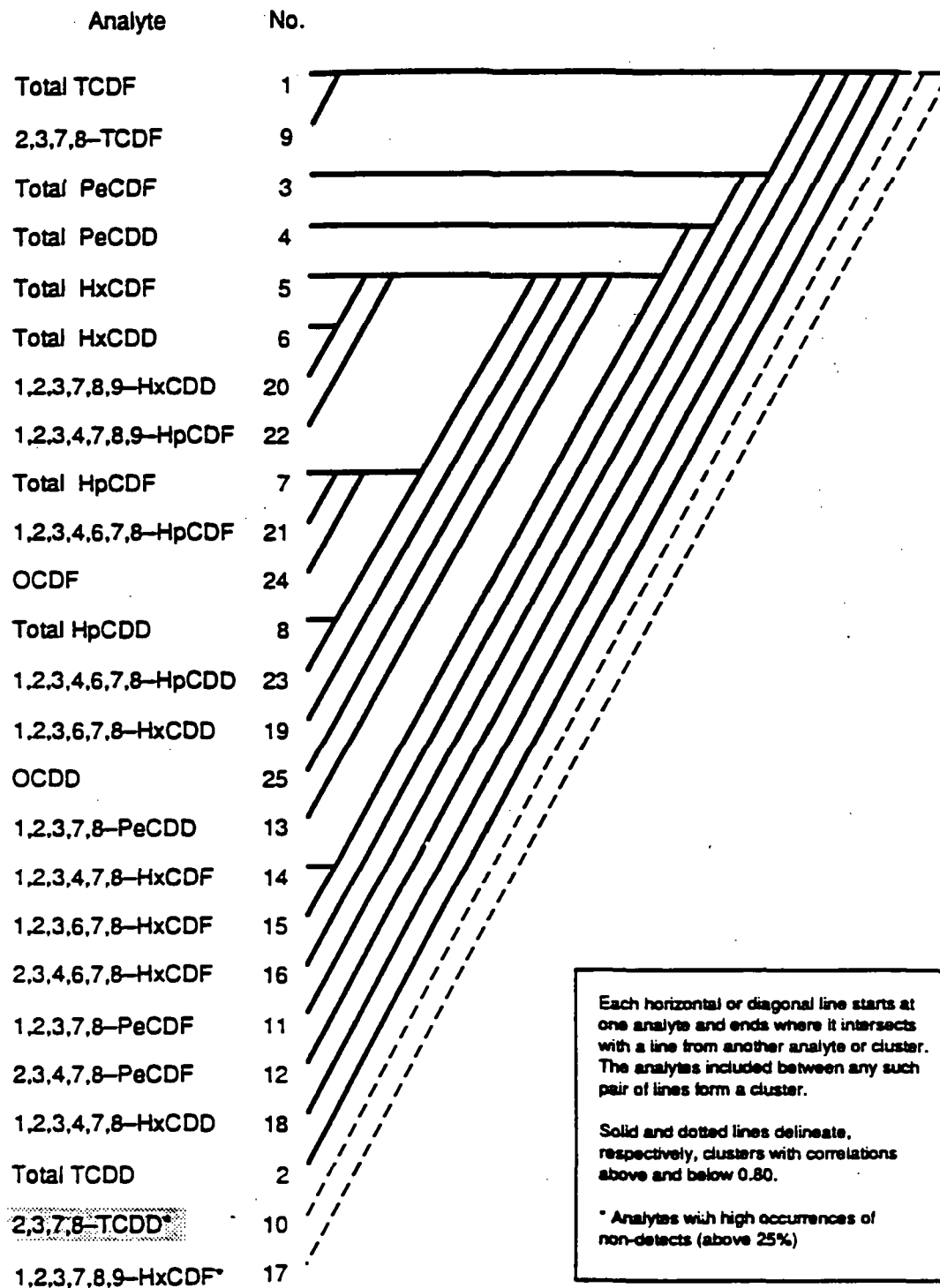


Figure 5. Tree diagram showing clustering of all 25 analytes based on correlations of log-transformed concentration levels in sediment.

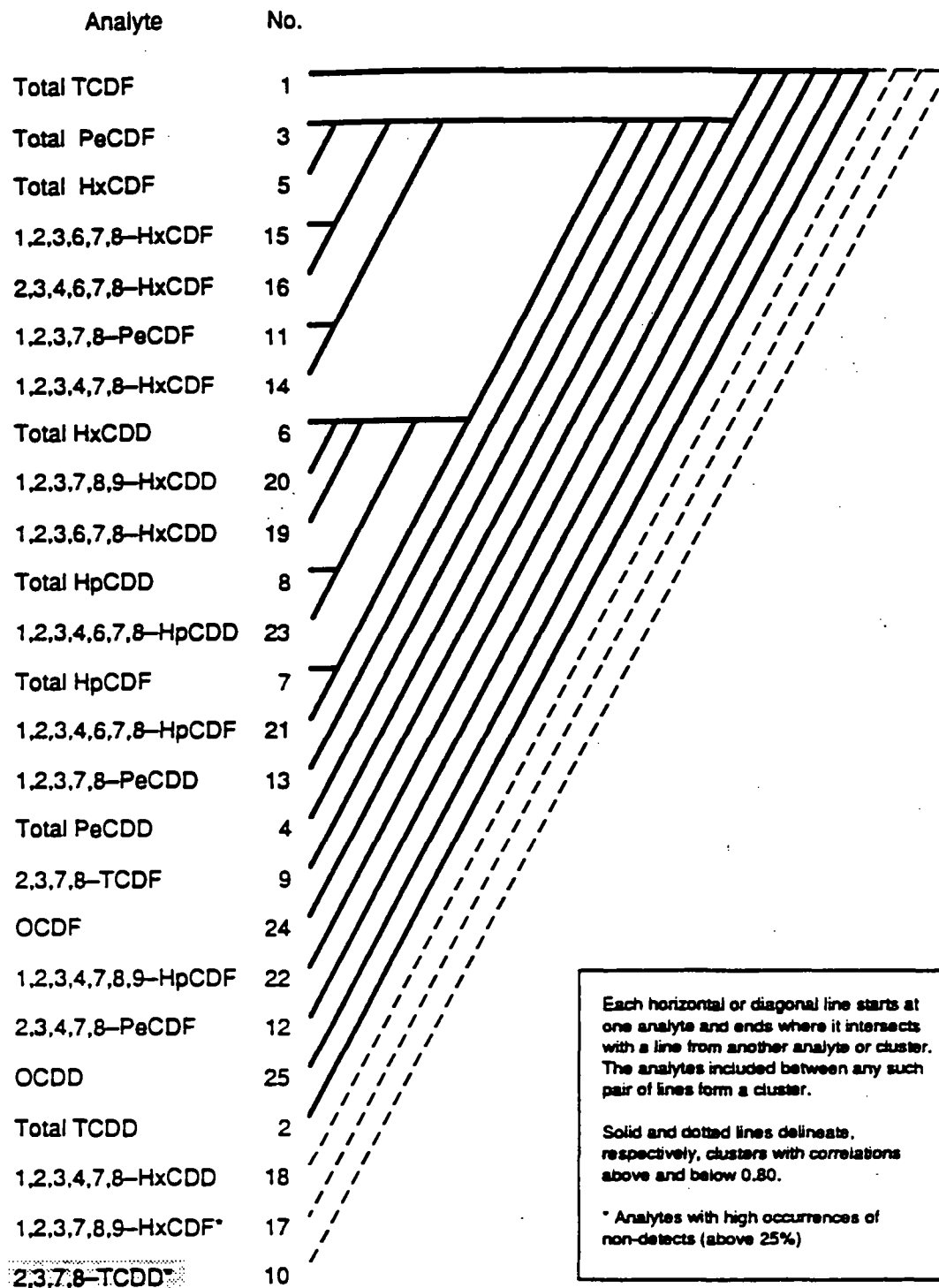


**Table 13. SUMMARY TABLE OF ANALYTE CLUSTERS FORMED  
AND THEIR CORRELATIONS IN SEDIMENT SAMPLES**

Analyte	Analyte No.	Other boundary of cluster (Analyte No.)	No. of analytes in cluster	Correlation* when cluster formed
Total TCDF	1	17	25	0.57
2,3,7,8-TCDF	9	1	2	0.93
Total PeCDF	3	11	18	0.90
Total PeCDD	4	16	16	0.91
Total HxCDF	5	15	14	0.92
Total HxCDD	6	20	2	0.97
1,2,3,7,8,9-HxCDD	20	5	3	0.96
1,2,3,4,7,8,9-HpCDF	22	5	4	0.96
Total HpCDF	7	23	5	0.97
1,2,3,4,6,7,8-HpCDF	21	7	2	0.98
OCDF	24	7	3	0.97
Total HpCDD	8	23	2	1.00
1,2,3,4,6,7,8-HpCDD	23	5	9	0.95
1,2,3,6,7,8-HxCDD	19	5	10	0.95
OCDD	25	5	11	0.94
1,2,3,7,8-PeCDD	13	5	12	0.92
1,2,3,4,7,8-HxCDF	14	15	2	0.94
1,2,3,6,7,8-HxCDF	15	4	15	0.91
2,3,4,6,7,8-HxCDF	16	3	17	0.90
1,2,3,7,8-PeCDF	11	1	20	0.90
2,3,4,7,8-PeCDF	12	1	21	0.87
1,2,3,4,7,8-HxCDD	18	1	22	0.85
Total TCDD	2	1	23	0.84
2,3,7,8-TCDD*	10	1	24	0.66
1,2,3,7,8,9-HxCDF*	17	1	25	0.57

\* Analytes with high occurrences of nondetects (over 25% during either pre-operational or operational status of the RRF.

\* Clusters with correlations above 0.80 are between the two solid black lines. Results are based on log-transformed concentrations levels; nondetects are replaced by one-half the detection limit. (Results in Table 13 correspond to those shown in Figure 5.)



**Figure 6. Tree diagram showing clustering of all 25 analytes based on correlations of log-transformed concentrations levels in soil.**

**Table 14. SUMMARY TABLE OF ANALYTE CLUSTERS FORMED  
AND THEIR CORRELATIONS IN SOIL SAMPLES**

Analyte	Analyte No.	Other boundary of cluster (Analyte No.)	No. of analytes in cluster	Correlation* when cluster formed
Total TCDF	1	10	25	0.47
Total PeCDF	3	24	17	0.94
Total HxCDF	5	3	2	0.98
1,2,3,6,7,8-HxCDF	15	16	2	0.98
2,3,4,6,7,8-HxCDF	16	3	4	0.97
1,2,3,7,8-PeCDF	11	14	2	0.97
1,2,3,4,7,8-HxCDF	14	3	6	0.97
Total HxCDD	6	21	7	0.96
1,2,3,7,8,9-HxCDD	20	6	2	0.97
1,2,3,6,7,8-HxCDD	19	6	3	0.96
Total HpCDD	8	23	2	1.00
1,2,3,4,6,7,8-HpCDD	23	6	5	0.96
Total HpCDF	7	21	2	0.99
1,2,3,4,6,7,8-HpCDF	21	3	13	0.96
1,2,3,7,8-PeCDD	13	3	14	0.96
Total PeCDD	4	3	15	0.95
2,3,7,8-TCDF	9	3	16	0.95
OCDF	24	1	18	0.94
1,2,3,4,7,8,9-HpCDF	22	1	19	0.93
2,3,4,7,8-PeCDF	12	1	20	0.91
OCDD	25	1	21	0.85
Total TCDD	2	1	22	0.83
1,2,3,4,7,8-HxCDD	18	1	23	0.79
1,2,3,7,8,9-HxCDF*	17	1	24	0.65
2,3,7,8-TCDD*	10	1	25	0.47

\* Analytes with high occurrences of nondetects (over 25% during either pre-operational or operational status of the RRF.

\* Clusters with correlations above 0.80 are between the two solid black lines. Results are based on log-transformed concentration levels; nondetects are replaced by one-half the detection limit. (Results in Table 14 correspond to those shown in Figure 6.)

well with any other analyte (this was clearly shown in the correlation tables presented earlier (Tables 9 and 10).

Although the clustering in sediment and soil samples differs slightly during the process as shown by different subgroupings (Figures 5 and 6), the levels of a large number of analytes are highly correlated. Thus, once a small number of analytes are quantified, most of the information about the distribution of analyte levels is known. This fact is not true for fish samples (Figure 4). Choosing a similar cut-off (i.e., correlation of 0.84), one can only cluster 8 out of 17 isomers into one group (cluster bound by 2,3,7,8-TCDD and 1,2,3,4,6,7,8-HpCDF in Figure 4). All other isomers would form single-isomer clusters. The "fingerprinting" is thus quite similar in sediment and soil samples, but very unlike that in fish samples.

## **5.2 PRINCIPAL COMPONENT ANALYSIS RESULTS**

Principal component analysis (PCA) was performed on various subsets of the data base to address some of the considerations mentioned in the introductory section of this report. The objective of PCA is to reduce the dimensionality of the original set of variables (e.g., number of analytes) by deriving a few new components as linear combinations of the original variables. This approach then provides a description of the structure of the data with as little loss of information as possible by means of principal component plots.

As in cluster analysis, all levels below detection limits were replaced with one half the detection limit, and all analyses were performed on the log-transformed concentration levels. As in all previous analyses, fish levels were on a wet, whole weight basis, while sediment and soil levels were on a dry weight basis. The PCA were performed on the correlation matrix of each set of

variables considered; that is, the concentration levels, after log-transformation, were autoscaled (standardized) so as to be treated with equal importance. Of the results from many PCAs performed, only a few relevant analyses will be discussed here.

### **5.2.1 Comparison of Pre-operational and Operational Isomer Data in Fish, Sediment, and Soil Samples**

The main purpose of the Connecticut DEP monitoring program is to establish whether the operation of an RRF has an impact on the levels of PCDDs and PCDFs in the environment. To that end, only samples collected around the four cities qualifying for a pre-operational and operational categorization were considered for analysis. The four cities and the number of years of operation of their RRF are as follows:

	Years of operation of RRF				Total No. of samples in PCA
	1987	1988	1989	1990	
Bristol	0	1	2	3	203
Bridgeport	0	1	2	3	70
Hartford	0	1	2	3	260
Wallingford	0	0	1	2	128

To investigate the effect of an RRF operation on PCDD and PCDF levels within each city, PCA was performed individually for each city. Accordingly, the results are shown individually for each city.

The concentration levels (logs) of all 17 isomers measured in fish, sediment, and soil, were included in the PCA. The profiles of the significant

principal components (eigenvalues above or close to 1) are shown in Tables 15 through 18 for Bristol, Bridgeport, Hartford, and Wallingford, respectively. These tables present the loading (correlation) of each analyte with the given principal component, and its individual contribution (percent variance) to the variance explained by the given principal component. Within each component, the analytes are listed in order of decreasing absolute loadings; a minus (plus) sign preceding a loading denotes a negative (positive) correlation of that analyte with the principal component. The last column lists cumulative percentage within each component. Only those analytes which contribute above average (i.e., over  $5.9\% = 1/17$ ) to the overall variability in the data set are shown in these tables.

Tables 15 through 18 provide the eigenvalue associated with a principal component and the percent of the total variance in each data set selected, explained by the principal component. In a few cases, a principal component with an eigenvalue below 1 was kept in the analysis for plotting purposes (two principal components are needed to plot the data).

For each city, a large proportion of the variance in the data (log-levels of the 17 isomers measured in fish, sediment, and soil samples) is explained by the first principal component (84.4%, 81.7%, 63.3%, and 63.8% for Bristol, Bridgeport, Hartford, and Wallingford, respectively.) In each case, the first principal component is a linear combination of most isomers with approximately equal loadings. It should be noted that none of the first principal components contains 2,3,7,8-TCDD. Thus 2,3,7,8-TCDD contributes only marginally to the variability in the data; in other words, any stratification in the samples along these isomer levels, if present, cannot be attributed to 2,3,7,8-TCDD.

**Table 15. PCA RESULTS FOR 17 ISOMERS IN FISH, SEDIMENT, AND SOIL SAMPLES COLLECTED NEAR BRISTOL**

Analyte (ordered by % variance)	Analyte loading (correlation)	Variance explained (%)	Cumulative variance (%)
<b>Principal Component 1 (Eigenvalue of 14.3, 84.4% of total variance explained)</b>			
1,2,3,7,8,9-HxCDD	0.98	6.75	6.75
1,2,3,4,6,7,8-HpCDD	0.97	6.58	13.32
2,3,4,6,7,8-HxCDF	0.97	6.57	19.90
1,2,3,6,7,8-HxCDF	0.97	6.55	26.45
1,2,3,4,6,7,8-HpCDF	0.97	6.55	32.99
1,2,3,4,7,8,9-HpCDF	0.96	6.42	39.41
OCDF	0.96	6.38	45.79
1,2,3,4,7,8-HxCDD	0.96	6.37	52.16
OCDD	0.95	6.25	58.41
1,2,3,7,8-PeCDF	0.94	6.14	64.55
1,2,3,4,7,8-HxCDF	0.94	6.13	70.67
1,2,3,6,7,8-HxCDD	0.93	6.05	76.73
<b>Principal Component 2 (Eigenvalue of 0.79, 4.7% of total variance explained)</b>			
2,3,7,8-TCDD	0.54	36.97	36.97
2,3,7,8-TCDF	0.52	33.82	70.79

All concentrations were log-transformed.

Analyte levels in fish are whole weight based.

Analyte levels in sediment and soil are dry weight based.

**Table 16. PCA RESULTS FOR 17 ISOMERS IN SEDIMENT AND SOIL  
SAMPLES COLLECTED NEAR BRIDGEPORT**

Analyte (ordered by % variance)	Analyte loading (correlation)	Variance explained (%)	Cumulative variance (%)
<b>Principal Component 1 (Eigenvalue of 13.9, 81.7% of total variance explained)</b>			
1,2,3,7,8,9-HxCDD	0.98	6.98	6.98
1,2,3,4,7,8-HxCDF	0.97	6.84	13.82
1,2,3,4,7,8,9-HpCDF	0.97	6.82	20.64
2,3,4,6,7,8-HxCDF	0.97	6.80	27.44
1,2,3,7,8-PeCDD	0.97	6.78	34.22
1,2,3,6,7,8-HxCDF	0.97	6.74	40.96
1,2,3,7,8-PeCDF	0.95	6.56	47.52
1,2,3,4,6,7,8-HpCDD	0.94	6.43	53.95
1,2,3,4,6,7,8-HpCDF	0.94	6.34	60.29
1,2,3,6,7,8-HxCDD	0.91	5.97	66.26
2,3,4,7,8-PeCDF	0.91	5.97	72.22
2,3,7,8-TCDF	0.90	5.90	78.12
1,2,3,4,7,8-HxCDD	0.90	5.88	84.01
<b>Principal Component 2 (Eigenvalue of 1.01, 6.0% of total variance explained)</b>			
1,2,3,7,8,9-HxCDF	0.73	51.88	51.88
OCDF	-0.37	13.19	65.07
2,3,7,8-TCDF	0.28	7.54	72.61

All concentrations were log-transformed.  
Analyte levels in sediment and soil are dry weight based.



**Table 17. PCA RESULTS FOR 17 ISOMERS IN FISH, SEDIMENT, AND SOIL  
SAMPLES COLLECTED NEAR HARTFORD**

Analyte (ordered by % variance)	Analyte loading (correlation)	Variance explained (%)	Cumulative variance (%)
<b>Principal Component 1 (Eigenvalue of 10.8, 63.3% of total variance explained)</b>			
1,2,3,7,8,9-HxCDD	0.97	8.70	8.70
1,2,3,4,6,7,8-HpCDD	0.95	8.35	17.06
1,2,3,4,6,7,8-HpCDF	0.94	8.24	25.30
1,2,3,6,7,8-HxCDD	0.92	7.91	33.21
1,2,3,4,7,8,9-HpCDF	0.92	7.87	41.08
OCDF	0.91	7.62	48.70
2,3,4,6,7,8-HxCDF	0.90	7.60	56.30
OCDD	0.88	7.17	63.46
1,2,3,6,7,8-HxCDF	0.88	7.17	70.63
1,2,3,4,7,8-HxCDD	0.84	6.61	77.24
<b>Principal Component 2 (Eigenvalue of 2.28, 13.4% of total variance explained)</b>			
2,3,7,8-TCDD	0.89	34.62	34.62
2,3,4,7,8-PeCDF	0.65	18.47	53.09
1,2,3,7,8-PeCDD	0.63	17.50	70.60
<b>Principal Component 3 (Eigenvalue of 1.21, 7.11% of total variance explained)</b>			
1,2,3,7,8,9-HxCDF	0.69	39.31	39.31
1,2,3,4,7,8-HxCDF	0.52	22.52	61.84
1,2,3,6,7,8-HxCDF	-0.35	10.36	72.19
2,3,4,7,8-PeCDF	-0.29	7.10	79.30

All concentrations were log-transformed.

Analyte levels in fish are whole weight based.

Analyte levels in sediment and soil are dry weight based.

**Table 18. PCA RESULTS FOR 17 ISOMERS IN FISH, SEDIMENT, AND SOIL  
SAMPLES COLLECTED NEAR WALLINGFORD**

Analyte (ordered by % variance)	Analyte loading (correlation)	Variance explained (%)	Cumulative variance (%)
<b>Principal Component 1 (Eigenvalue of 10.9, 63.8% of total variance explained)</b>			
1,2,3,4,7,8-HxCDF	0.96	8.53	8.53
1,2,3,7,8,9-HxCDD	0.96	8.47	17.00
1,2,3,6,7,8-HxCDF	0.95	8.29	25.29
1,2,3,6,7,8-HxCDD	0.94	8.22	33.50
1,2,3,4,6,7,8-HpCDF	0.91	7.63	41.14
1,2,3,7,8-PeCDF	0.90	7.41	48.54
1,2,3,4,6,7,8-HpCDD	0.89	7.28	55.82
1,2,3,4,7,8,9-HpCDF	0.88	7.20	63.02
1,2,3,4,7,8-HxCDD	0.82	6.27	69.29
<b>Principal Component 2 (Eigenvalue of 3.0, 17.6% of total variance explained)</b>			
2,3,7,8-TCDD	0.80	21.12	21.12
OCDF	-0.63	13.43	34.55
OCDD	-0.63	13.21	47.75
2,3,4,7,8-PeCDF	0.61	12.59	60.35
1,2,3,7,8-PeCDD	0.60	12.20	72.55
<b>Principal Component 3 (Eigenvalue of 0.97, 5.7% of total variance explained)</b>			
1,2,3,7,8,9-HxCDF	-0.65	43.83	43.83
2,3,7,8-TCDF	0.56	32.39	76.22
2,3,4,6,7,8-HxCDF	-0.31	9.71	85.93

All concentrations were log-transformed.  
Analyte levels in fish are whole weight based.  
Analyte levels in sediment and soil are dry weight based.

The contribution of the second principal component is considerably lower than that of the first component: 4.7%, 6.0%, 13.4%, and 17.6% for Bristol, Bridgeport, Hartford, and Wallingford, respectively. In three of the four cities, 2,3,7,8-TCDD is the isomer with the highest loading for that principal component. However, as stated above, this isomer contributes only a small proportion to the overall variability (for example, in Bristol—Table 15—the overall contribution of 2,3,7,8-TCDD is 36.97% of 4.7%, or 1.74% overall). The highest, though very low, contribution by 2,3,7,8-TCDD to the overall data variability is found in Hartford at 4.64% (34.62% of 13.4%).

In Hartford, a third principal component was statistically significant (eigenvalue of 1.21), explaining an additional 7.11% of the total variance of the data (Table 17). It is mainly a combination of three hexa-furans. The third principal component was also listed for Wallingford since its eigenvalue of 0.97 fell slightly short of the cut-off point of 1.

The information provided by each of the four tables is shown more readily by means of principal component plots. For each city, the statistically significant principal components are used to compute factor scores for each sample included in the analysis. These standardized scores were calculated by using the respective loadings (correlations) as the coefficients in the linear combinations of the standardized log levels of the analytes in each principal component. Plotting these scores in the space spanned by the first two principal components provides a means of detecting visible patterns, groupings, and/or trends in the samples, should they exist. The values of any other relevant variable associated with the samples can then be used to encode the points on the plot when looking for patterns with respect to that variable. The variable of most interest here is the number of years the RRF has been in operation at the time of sampling. Thus, the points on the principal component plots will be

encoded as 0, 1, 2, or 3, depending on the year during which the sample was collected.

While generating the principal component plots using the number of years of operation of each RRF as the encoding variable, the separation of the data points by matrix type became apparent. Thus, the same plots were also encoded by matrix (fish—F, sediment—S, soil—T) to highlight this obvious matrix separation.

Figures 7 through 14 show eight selected principal component plots. In each plot, the first principal component, explaining the highest proportion of variance in a given pair, is always plotted on the X-axis. The second principal component is plotted on the Y-axis. A plot of the two principal components will therefore display the sum of the variation in the data explained by the two principal components.

Each set of two plots corresponds to a city. The top plot on each page is encoded by the number of years the RRF has been in operation at the time of sampling. The bottom plot on each page shows the same data, using matrix type as the encoding variable, thus explaining the apparent separation in the top plots, except for Bridgeport (no fish samples were collected in that city). This separation between fish, sediment and soil is consistent throughout the entire base and will be further discussed in subsection 5.2.2.

Figures 7, 9, 11, and 13 (Bristol, Bridgeport, Hartford, and Wallingford, respectively) display the standardized scores of all samples (fish, sediment, and soil) along the first two principal components. The plots are encoded by the number of years each RRF had been in operation at the time of sampling.

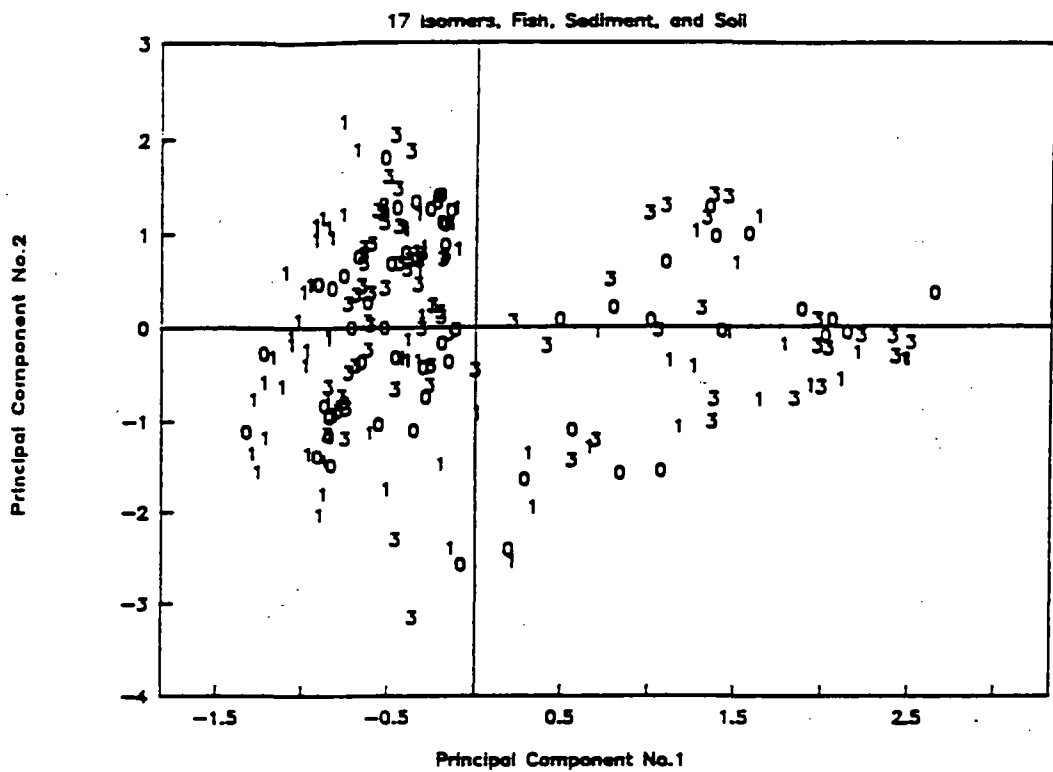


Figure 7. Principal component 2 vs. 1—Years of operation of RRF in Bristol.

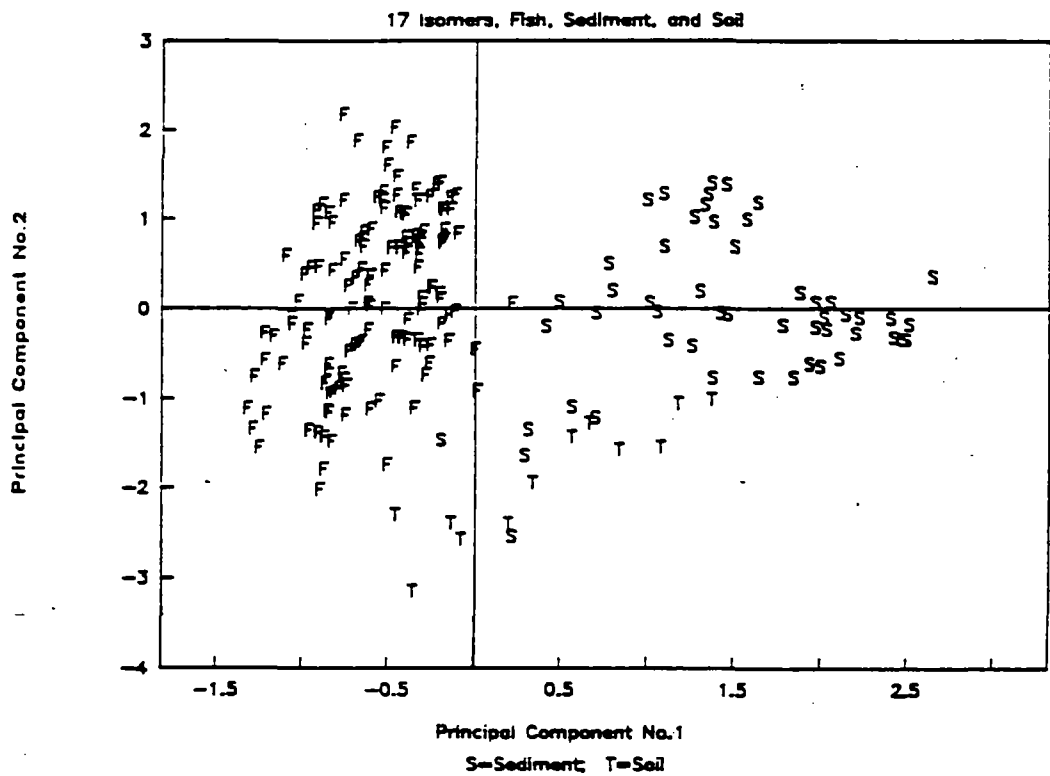


Figure 8. Principal component 2 vs. 1—Matrix effect in Bristol.

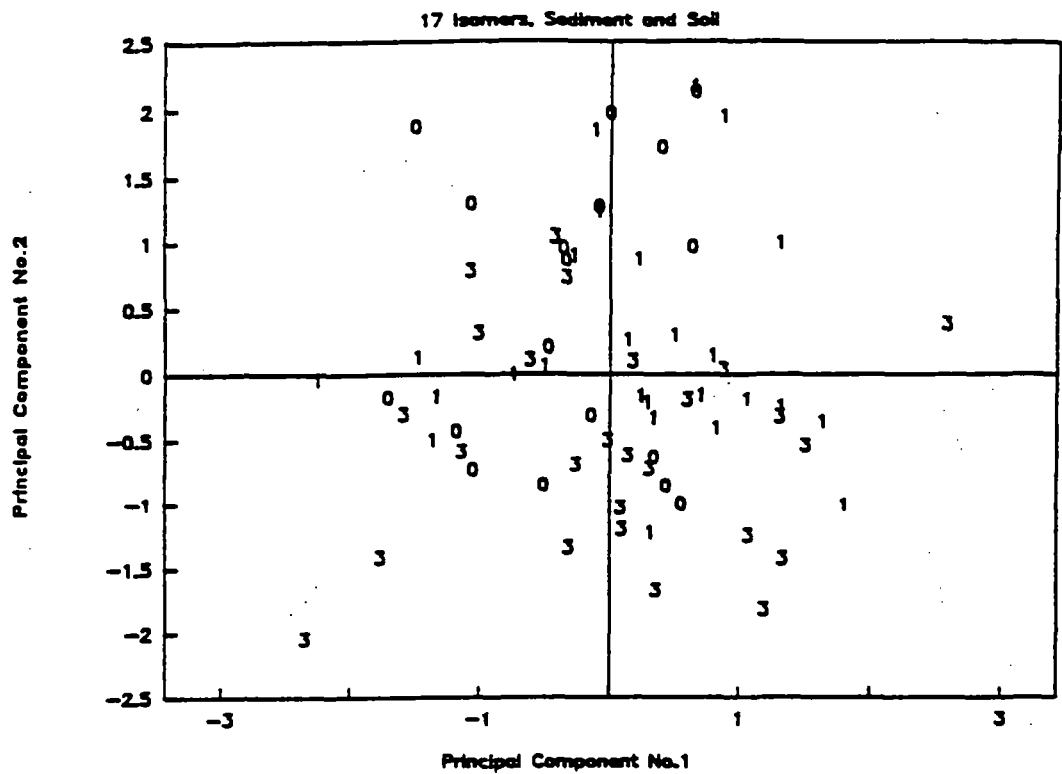


Figure 9. Principal component 2 vs. 1—Years of operation of RRF in Bridgeport.

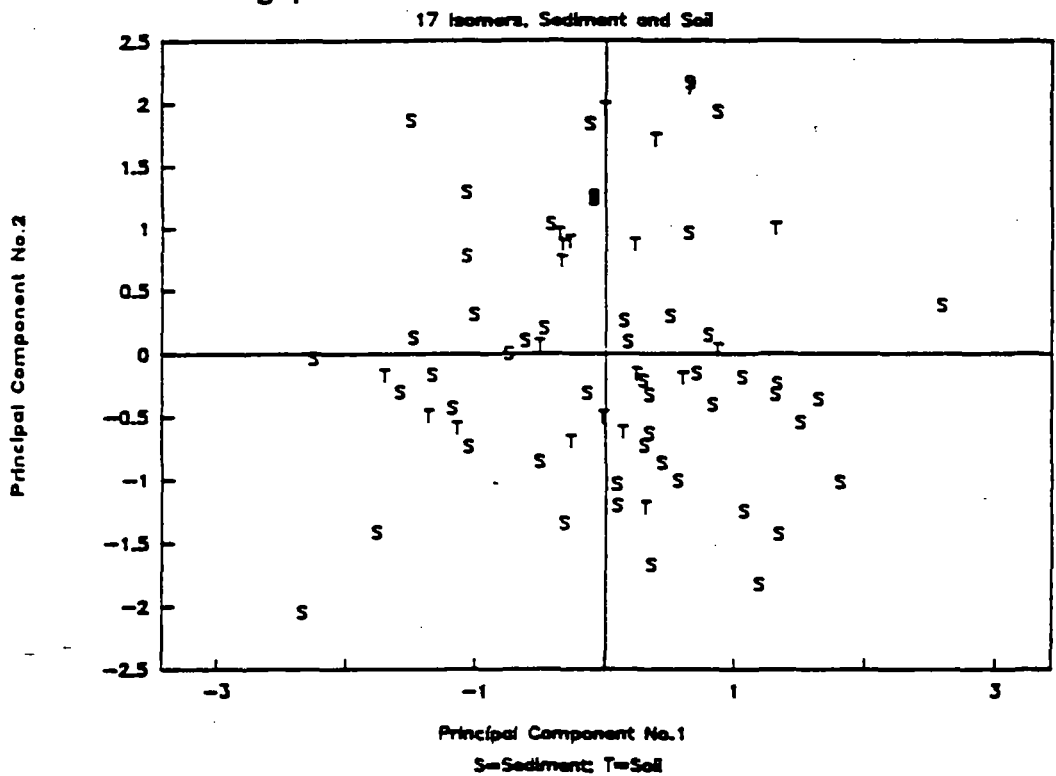


Figure 10. Principal component 2 vs. 1—Matrix type in Bridgeport (no fish samples collected).

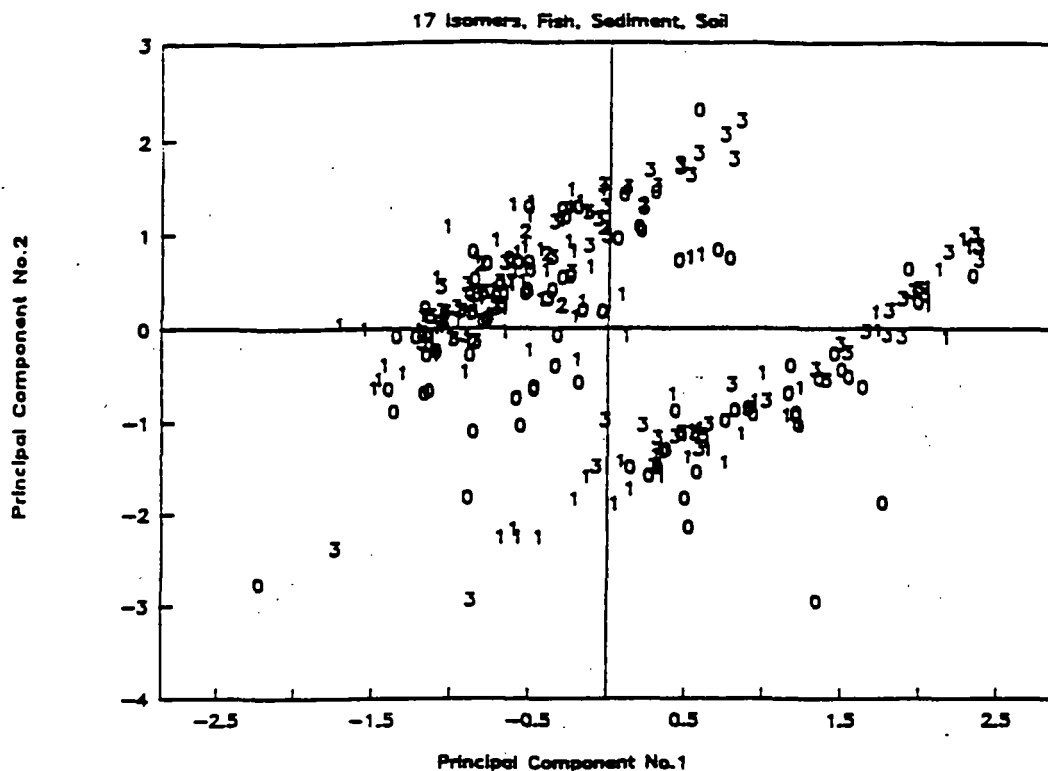


Figure 11. Principal component 2 vs. 1—Years of operation of RRF in Hartford.

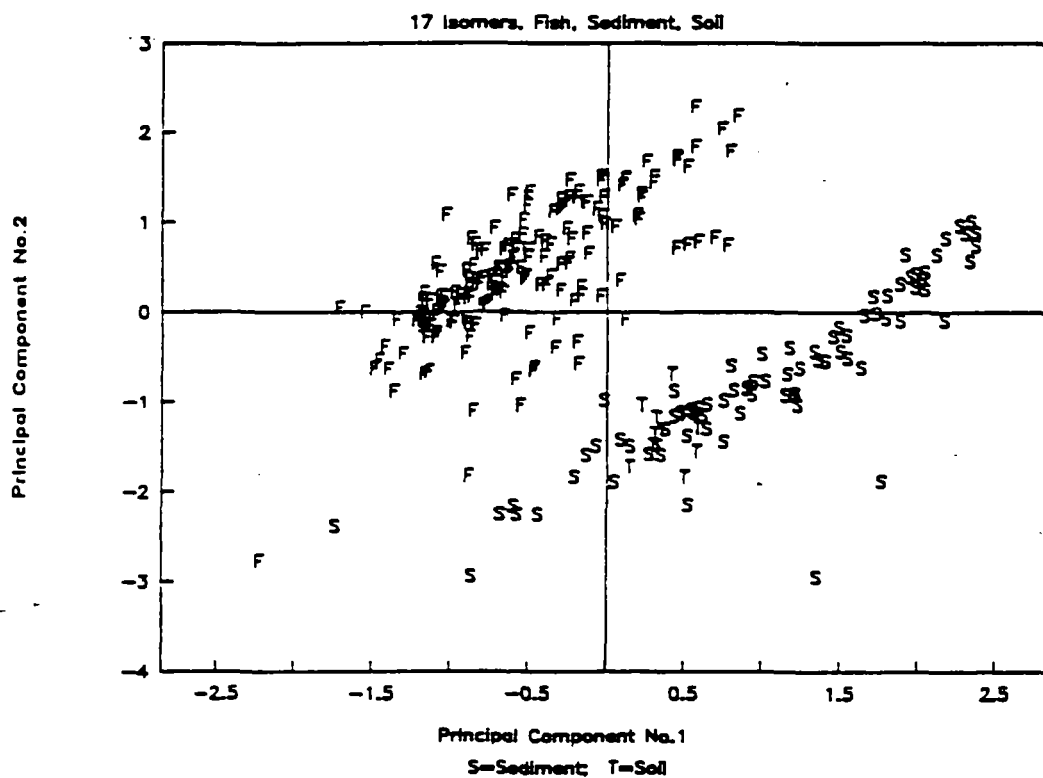


Figure 12. Principal component 2 vs. 1—Matrix effect in Hartford.

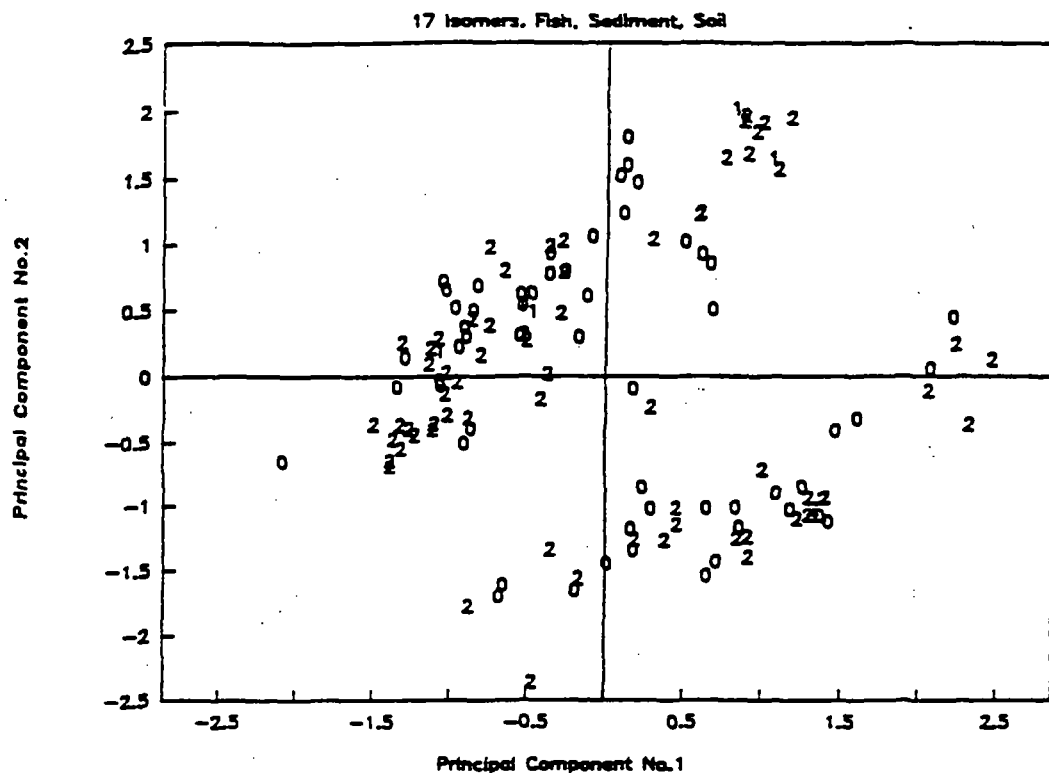


Figure 13. Principal component 2 vs. 1—Years of operation of RRF in Wallingford.

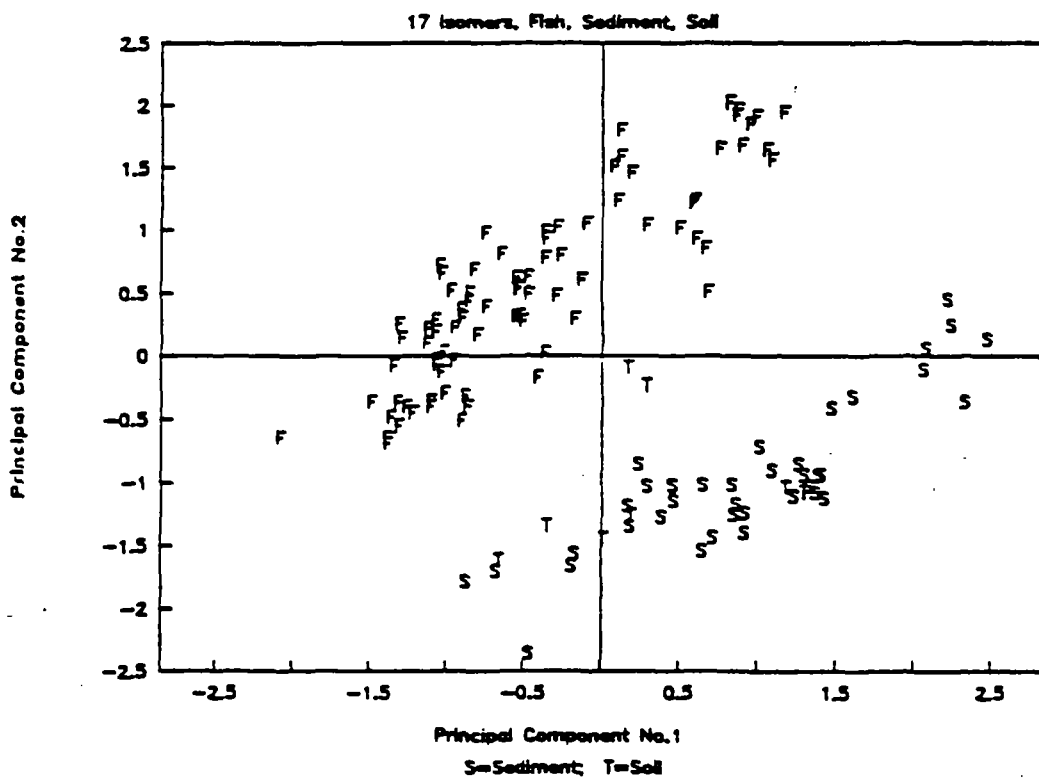


Figure 14. Principal component 2 vs. 1—Matrix effect in Wallingford.



Remembering that most of the data variability is represented along the first principal component (X-axis), patterns found along that axis would be most significant of the RRF's effect on sample levels over time. These two-dimensional plots, however, do not reveal any stratification of the data by years of operation, as shown by the lack of separation of the code levels (0, 1, 2, and 3), indicating that the operation of the RRF has no apparent effect on the levels of these isomers in each of the four cities considered. It should be noted, though, that detecting a potential trend in analyte levels over time may require a minimum of four to five years of operational data. At the time of these analyses, the longest time an RRF had been in operation in any of the four cities was three years. In addition, only a very limited number of data was available from 1989 (19 fish samples), resulting in a one-year gap in the data base.

The stratification of the data on these plots (excluding Figure 9 for Bridgeport where no fish samples were collected) becomes more evident when encoding the data by matrix type as shown in Figures 8, 12, and 14. These results with respect to matrix effect are presented next.

### **5.2.2 Matrix Effect on Analyte Levels**

From the previous PCA, Figures 8, 12, and 14 clearly showed a separation of the samples by matrix. This apparent matrix effect was further investigated using all the data available. A total of 923 samples could be included in a PCA of the log-concentration levels of the 17 isomers in fish, sediment, and soil. The profiles of the two major principal components are presented in Table 19, along with the variable loadings and their individual contributions (percent variance) to the variance explained by each principal component. Again, only the analytes with a variance contribution above average ( $1/17$  or 5.9%, where 17 is the number of analytes included in the PCA) are presented within the two sets.

The first principal component, explaining approximately 78% of the total variance, is a combination of mostly higher-chlorinated dioxins and furans, all with about equal positive loadings. The second principal component, contributing only about 8% to the total variability in the data, consists of 2,3,7,8-TCDD and 1,2,3,7,8-PeCDD and 2,3,4,7,8-PeCDF. Note that OCDF and OCDD contribute to both principal components, though only in a minor way to the second component.

The companion principal component plot to Table 19 is shown in Figure 15. This figure depicts principal component 2 vs. 1 (displaying about 86% of the total variation in the data). Figure 15 shows a distinct separation of analytical results for sediments (S) and soils (T) versus fish samples (F). Furthermore, there is no separation by matrix within the group formed by sediment and soil samples. This grouping is not unexpected, given the distinct differences observed in the raw analytical data for the PCDD and PCDF fingerprints. The PCDDs and PCDFs in fish samples consist primarily of the 2,3,7,8-substituted isomers. These isomers are recognized as the analytes that are also retained by other animal species. In contrast, the 2,3,7,8-substituted isomers are minor contributors to overall concentrations for the total PCDD and PCDF residue levels in sediments and soils. These findings are also supported by the results obtained via cluster analysis of the analytes in samples from the three matrices (Section 5).

### **5.2.3 Comparison of Analyte Levels Across Cities**

The principal component plot shown in Figure 15 was redrawn separately for each city to better compare the data distribution within and among the nine cities included in the analyses. Thus, nine plots were generated and are shown in Figures 16 through 24. These plots are all on the same scale and thus, comparisons can be made across them. The plots pertain to the nine cities as follows:

**Table 19. PCA RESULTS FOR 17 ISOMERS IN FISH, SEDIMENT, AND SOIL SAMPLES—ALL CITIES**

Analyte (ordered by % variance)	Analyte loading (correlation)	Variance explained (%)	Cumulative variance (%)
<b>Principal Component 1 (Eigenvalue of 13.3, 78.4% of total variance explained)</b>			
1,2,3,7,8,9-HxCDD	0.98	7.15	7.16
1,2,3,4,6,7,8-HpCDF	0.96	6.95	14.10
1,2,3,4,6,7,8-HpCDD	0.96	6.89	20.99
1,2,3,6,7,8-HxCDF	0.95	6.81	27.80
1,2,3,6,7,8-HxCDD	0.94	6.63	34.43
2,3,4,6,7,8-HxCDF	0.94	6.59	41.02
1,2,3,7,8-PeCDF	0.93	6.48	47.49
1,2,3,4,7,8,9-HpCDF	0.93	6.44	53.93
1,2,3,4,7,8-HxCDF	0.92	6.37	60.30
1,2,3,4,7,8-HxCDD	0.92	6.32	66.63
OCDF	0.92	6.28	72.91
OCDD	0.90	6.10	79.01
<b>Principal Component 2 (Eigenvalue of 1.34, 7.9% of total variance explained)</b>			
2,3,7,8-TCDD	0.70	36.26	36.26
1,2,3,7,8-PeCDD	0.42	13.46	49.72
2,3,4,7,8-PeCDF	0.37	10.27	59.99
OCDF	-0.35	8.93	68.92
OCDD	-0.30	6.71	75.62

All concentrations were log-transformed.

Analyte levels in fish are whole weight based.

Analyte levels in sediment and soil are dry weight based.

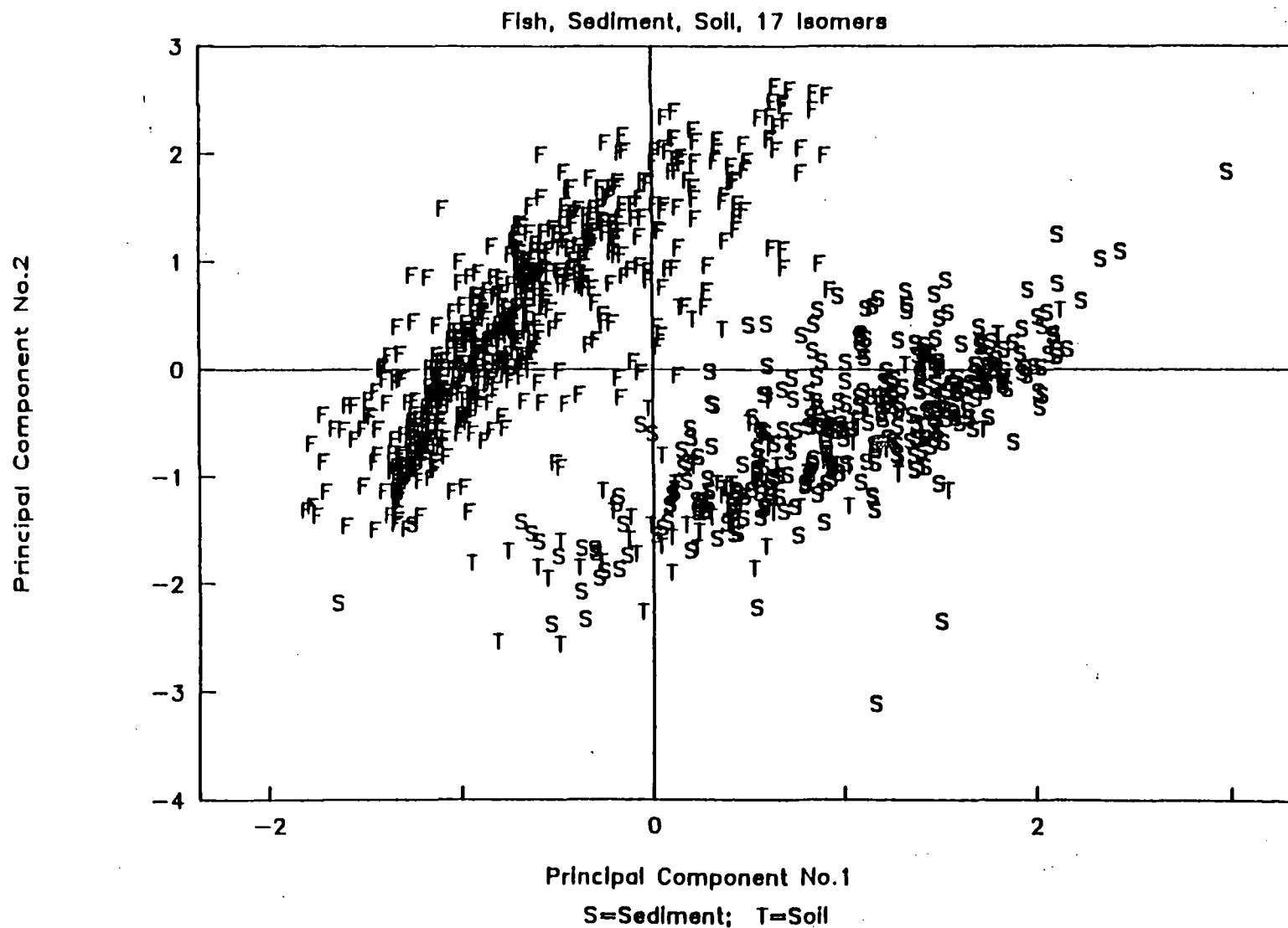


Figure 15. Principal component 2 vs. 1—Matrix effect in all cities.

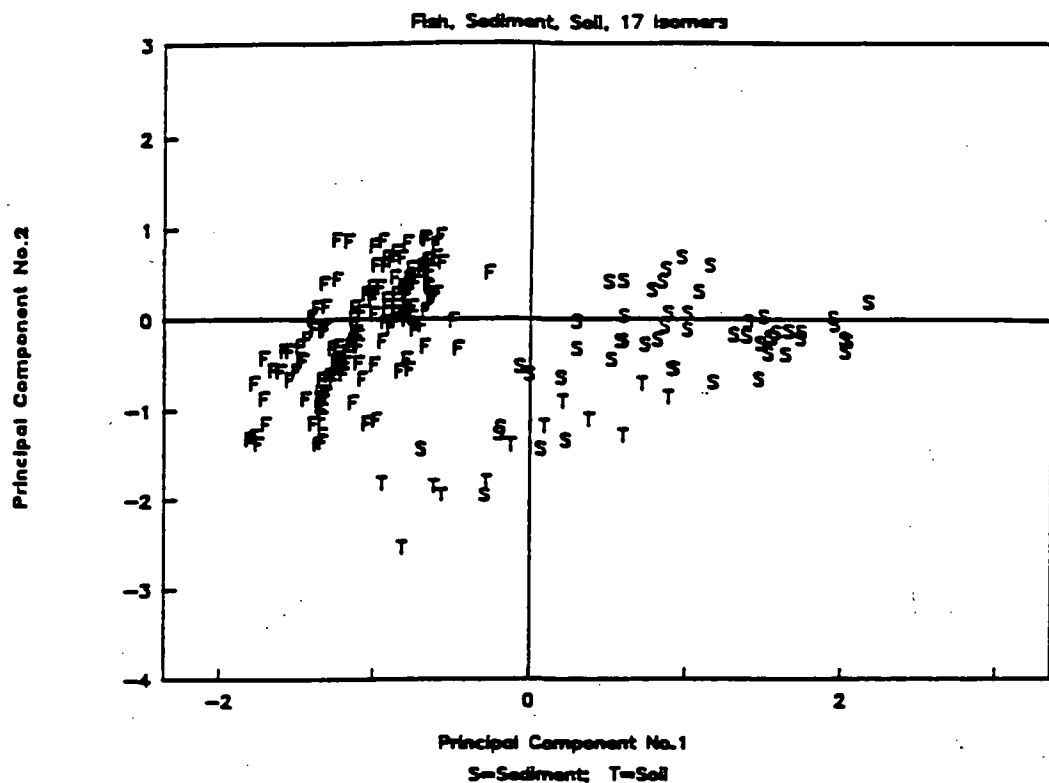


Figure 16. Principal component 2 vs. 1—Matrix Effect (Bristol).

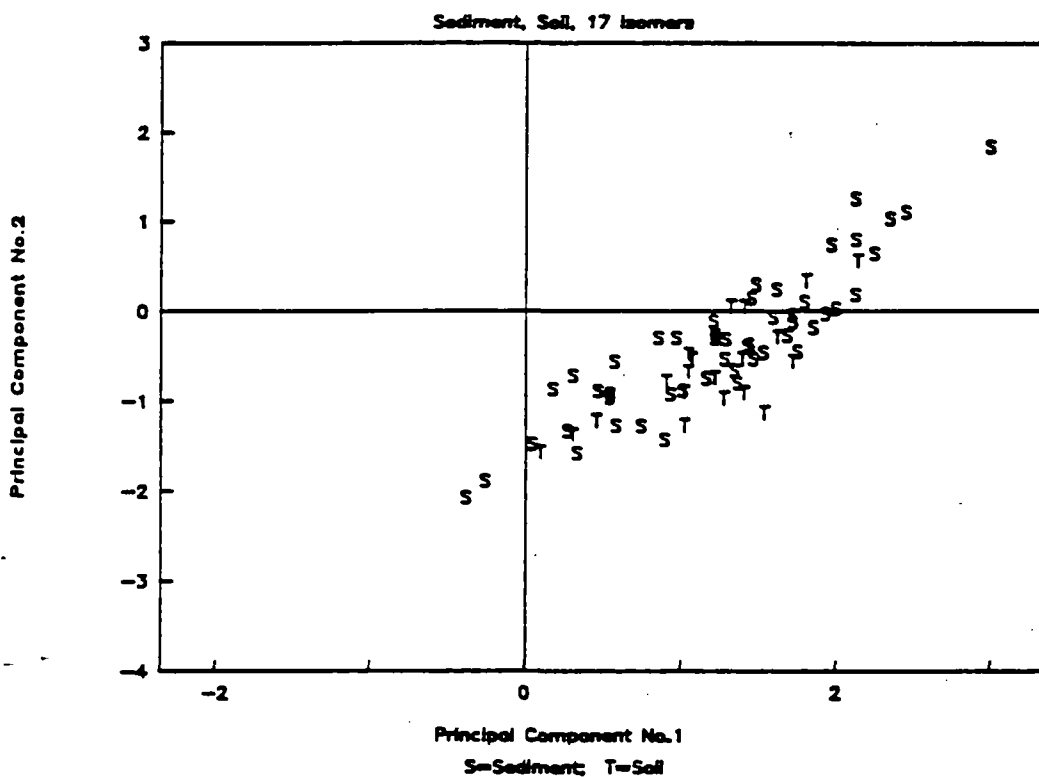


Figure 17. Principal component 2 vs. 1—Matrix Type (no fish samples collected in Bridgeport).

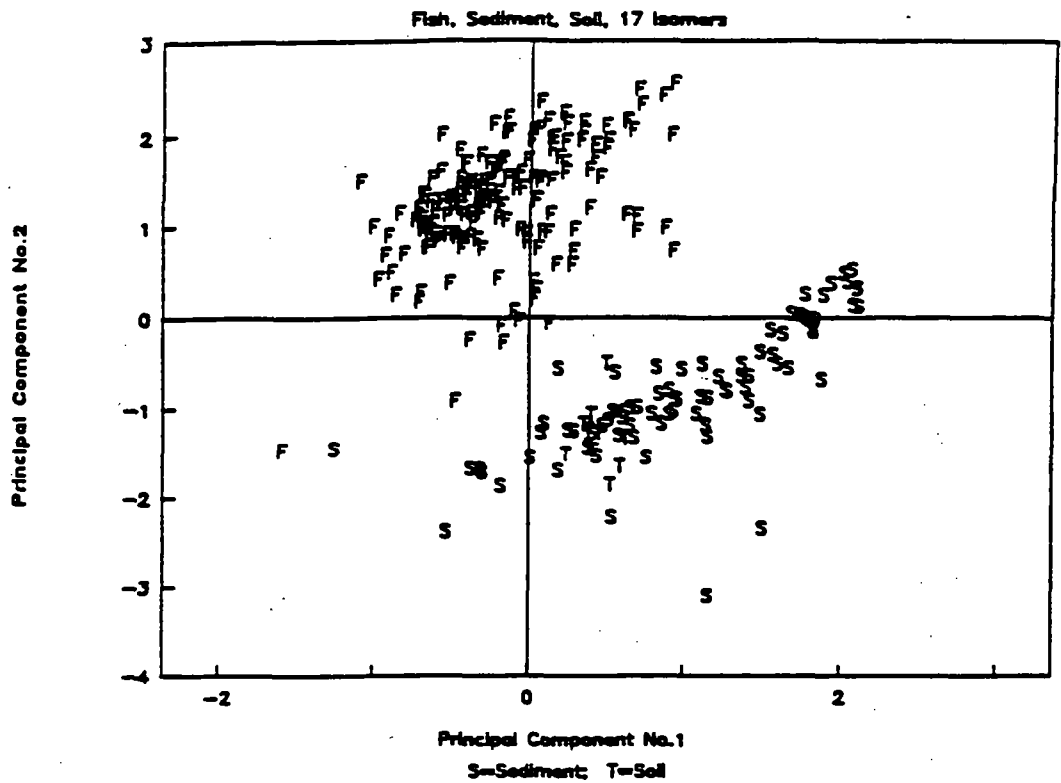


Figure 18. Principal component 2 vs. 1—Matrix Effect (Hartford).

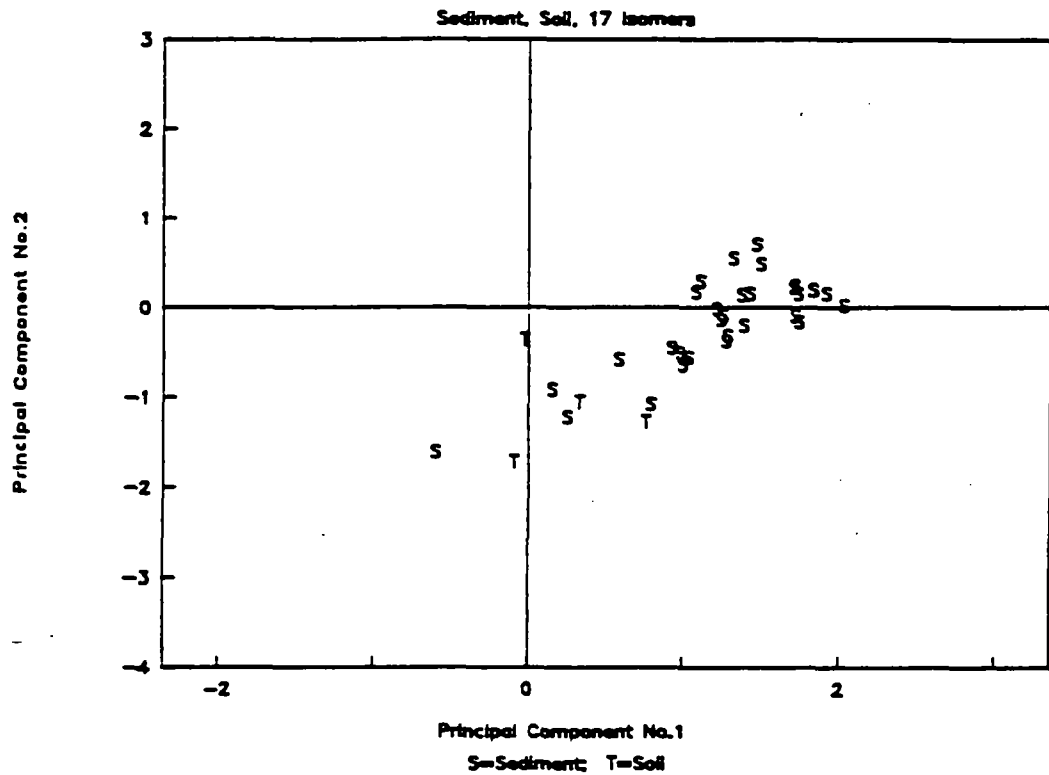


Figure 19. Principal component 2 vs. 1—Matrix Type (no fish samples collected in Preston).

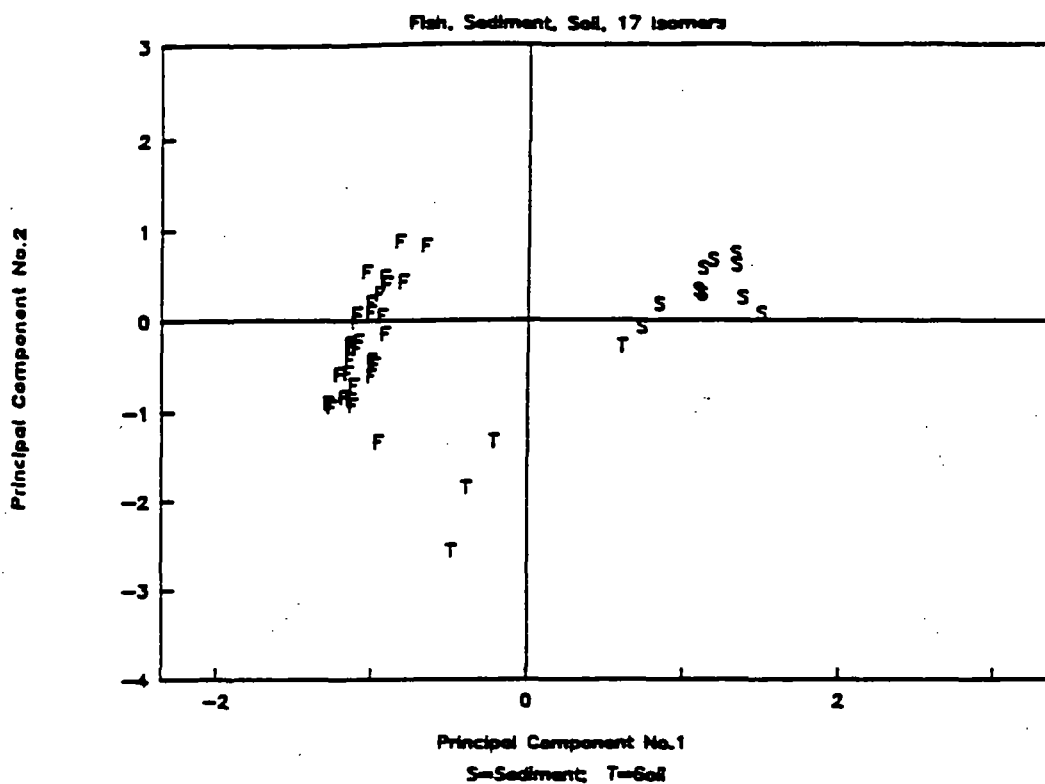


Figure 20. Principal component 2 vs. 1—Matrix Effect (Stafford).

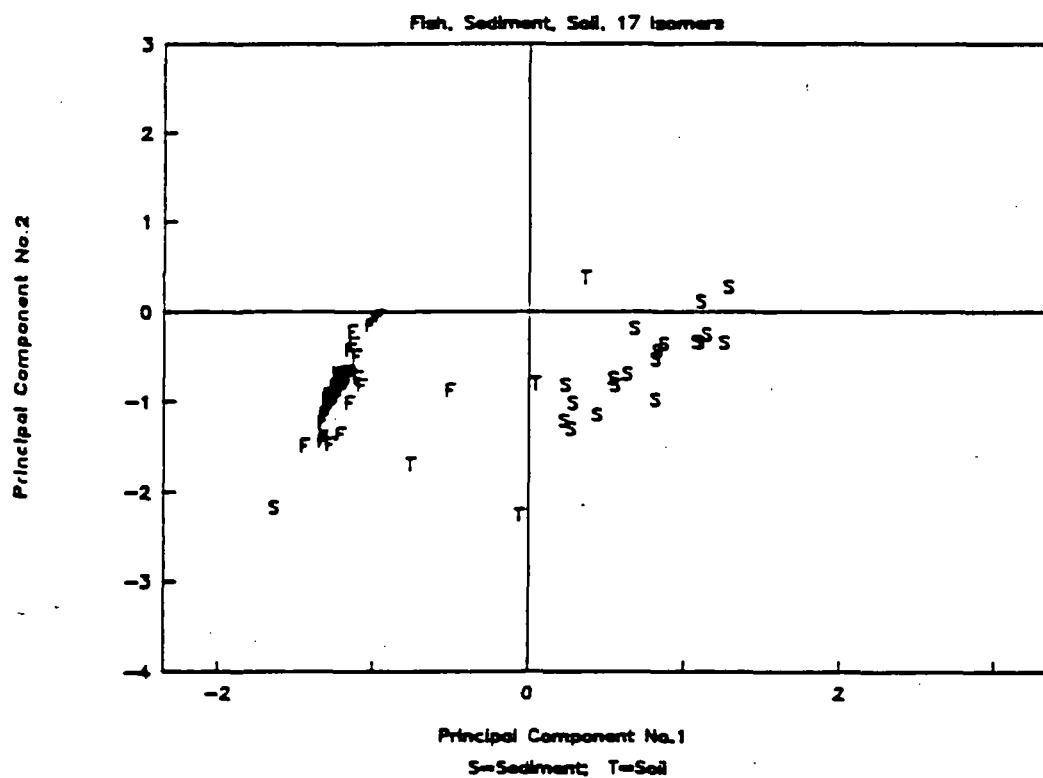


Figure 21. Principal component 2 vs. 1—Matrix Effect (Sterling).

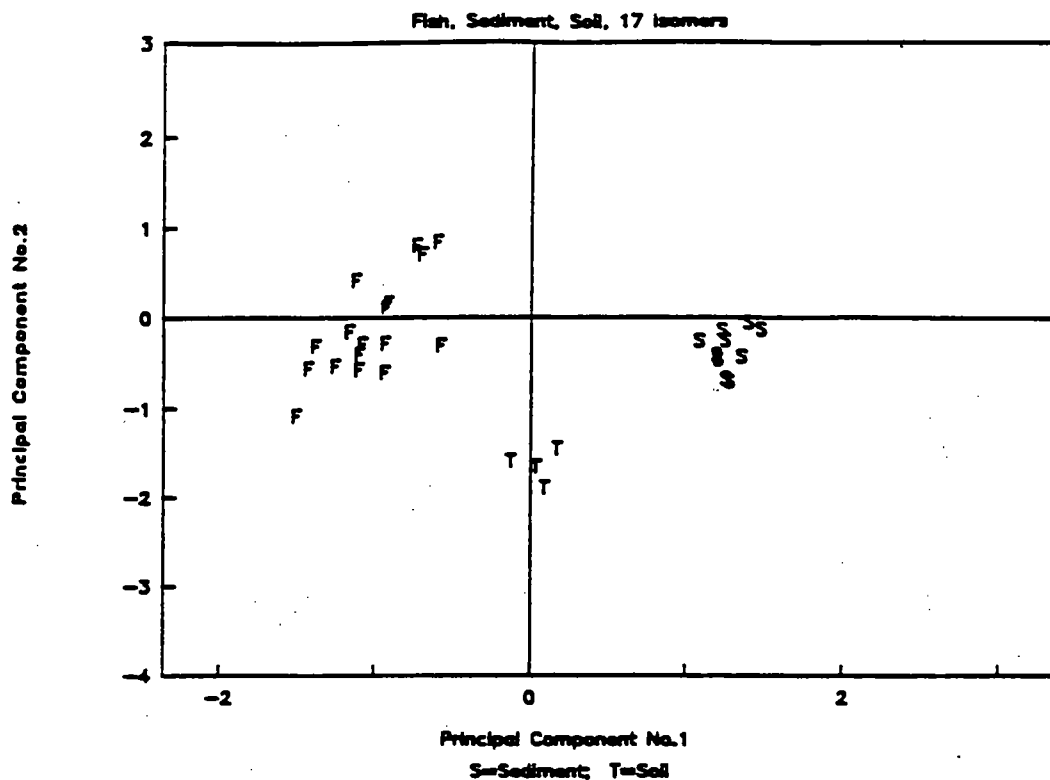


Figure 22. Principal component 2 vs. 1—Matrix Effect (Union).

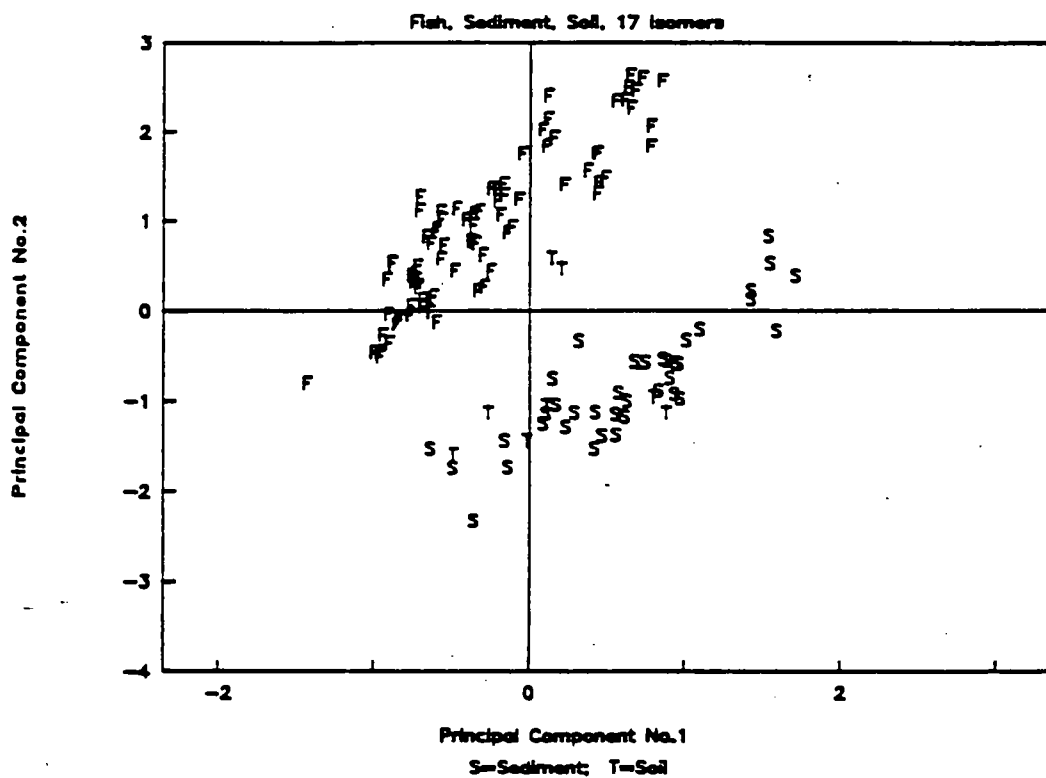


Figure 23. Principal component 2 vs. 1—Matrix Effect (Wallingford).



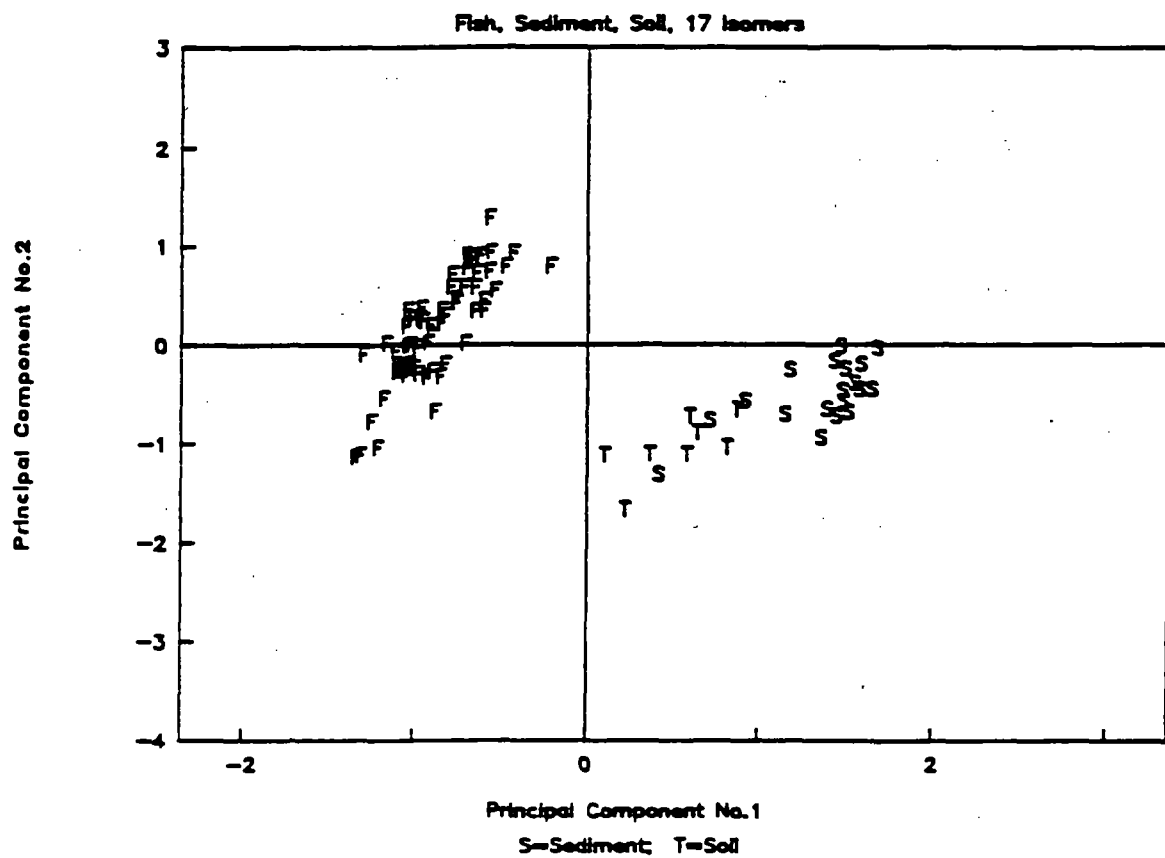


Figure 24. Principal component 2 vs. 1—Matrix Effect (Willimantic).

- Figure 16: Bristol (RRF in operation since 1988)
- Figure 17: Bridgeport (no fish samples collected; RRF in operation since 1988)
- Figure 18: Hartford (RRF in operation since 1988)
- Figure 19: Preston (no fish samples collected)
- Figure 20: Stafford, a control city in 1990
- Figure 21: Sterling
- Figure 22: Union, a control city in 1987
- Figure 23: Wallingford (RRF in operation since 1989)
- Figure 24: Willimantic (RRF in operation since 1982)

The distinct separation of fish from sediment and soil data shown in Figure 15 is maintained in each of the seven city plots (no fish samples were collected in Bridgeport and Preston). Some additional separation between sediment and soil samples is visible in Bristol, Preston, Stafford, Sterling, Union, and Willimantic. In each of these cases, the scores for soil samples (code "T" in plots) are generally lower than those for sediment samples (code "S" on plots) on both principal component axes, reflecting slight differences in "isomer fingerprints" between the two matrices. This is in line with the cluster analysis results (see Figures 5 and 6), although all 25 analytes were included in the cluster analyses while only the 17 2,3,7,8-substituted isomers are considered here.

An interesting observation when comparing the nine plots is that for Hartford and Wallingford, the group of fish samples scores higher on the second principal component axis than for the other seven cities. This axis is mostly driven by 2,3,7,8-TCDD (see second principal component profile in Table 15), giving some indication of higher 2,3,7,8-TCDD levels in those two cities as compared to the other seven cities in the study.

No other city effect (such as low or high outlier groups) is reflected in these plots. Both Stafford and Union, which served as control sites, exhibit the same patterns as the other cities. This is also true for Willimantic, a city where an RRF has been in operation since 1982.

#### **5.2.4 Fish Species Effect on Isomer Levels**

A series of PCAs was performed using various combinations of analytes, matrices, and other available parameters. Based on the observation of the separation between fish and soil and sediment samples, additional PCAs focused on the variables affecting fish residue levels. The effect of fish species on PCDD and PCDF uptake is shown here.

Prior to PCA, the six fish species were categorized into three groups (brown bullhead; yellow perch and white sucker; and white catfish, channel catfish, and carp) by means of analysis of variance of the lipid content (percentage) of each sample (see Subsection 3.3). PCA was performed using the 2,3,7,8-substituted isomer log-concentrations in fish samples only (fish levels on a whole weight basis). Three principal components were significant in explaining the variability in the data. Their profiles are shown in Table 20.

Of the three possible combinations in plotting pairs of principal components, two are shown here to illustrate (a) the higher PCDD and PCDF levels in fish collected in Hartford and Wallingford, and (b) some stratification of fish samples by fish species. Figure 25 illustrates a slight city effect, and Figures 26, 27, and 28 illustrate the fish species effect.

**Table 20. PCA RESULTS FOR 17 ISOMERS IN FISH**

Analyte (ordered by % variance)	Analyte loading (correlation)	Variance explained (%)	Cumulative variance (%)
<b>Principal Component 1 (Eigenvalue of 10.6, 62.1% of total variance explained)</b>			
1,2,3,6,7,8-HxCDD*	0.93	8.21	8.21
1,2,3,7,8,9-HxCDD*	0.92	8.05	16.26
1,2,3,4,6,7,8-HpCDF*	0.91	7.85	24.11
1,2,3,4,6,7,8-HpCDD	0.90	7.66	31.77
1,2,3,7,8-PeCDD	0.90	7.62	39.39
2,3,7,8-TCDD	0.89	7.43	46.81
1,2,3,4,7,8-HxCDD*	0.87	7.11	53.92
1,2,3,7,8-PeCDF	0.85	6.83	60.74
2,3,4,7,8-PeCDF	0.83	6.51	67.25
1,2,3,6,7,8-HxCDF*	0.82	6.31	73.56
2,3,4,6,7,8-HxCDF*	0.80	6.06	79.63
<b>Principal Component 2 (Eigenvalue of 1.64, 9.66% of total variance explained)</b>			
1,2,3,7,8,9-HxCDF*	0.91	50.60	50.60
1,2,3,4,7,8,9-HpCDF*	0.50	15.47	66.07
1,2,3,4,7,8-HxCDF*	0.47	13.19	79.25
<b>Principal Component 3 (Eigenvalue of 1.07, 6.27% of total variance explained)</b>			
2,3,7,8-TCDF	0.66	40.93	40.93
OCDF*	-0.43	17.03	57.96
OCDD	-0.40	14.65	72.61
1,2,3,7,8-PeCDF	0.29	7.74	80.35

- \* Analytes with high occurrences of nondetects (over 25% during either pre-operational or operational status of the RRF).  
 All concentrations were log-transformed.  
 Analyte levels in fish are whole weight based.

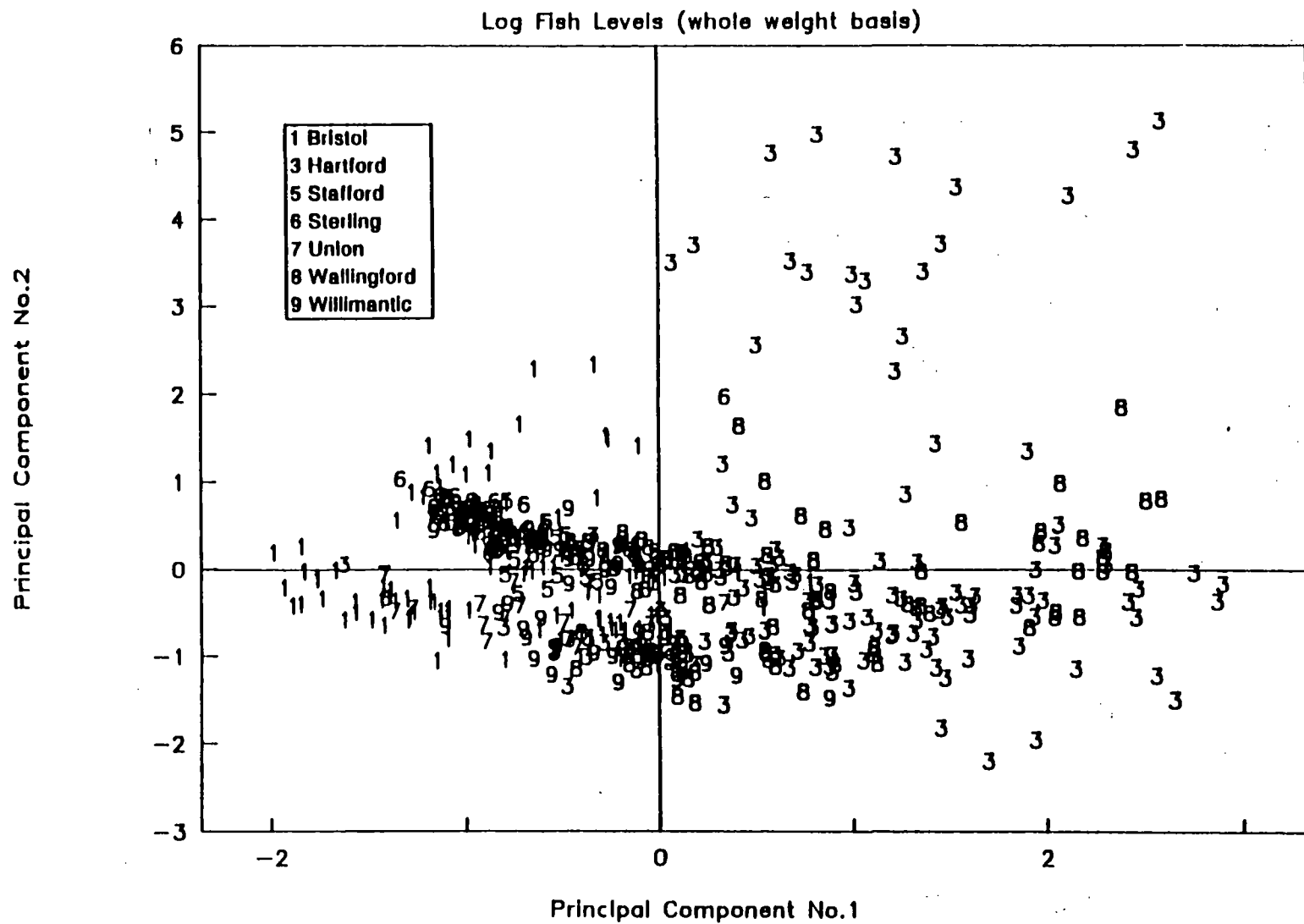


Figure 25. Principal component 2 vs. 1—City effect on fish levels.

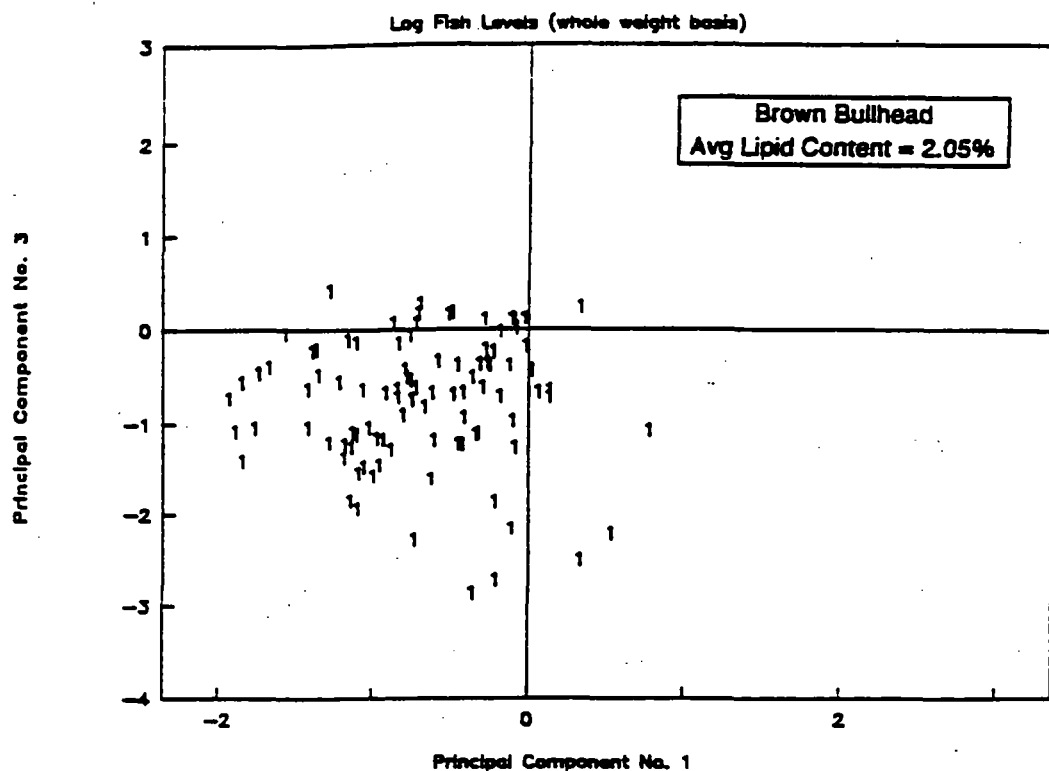


Figure 26. Principal component 3 vs. 1—Fish species effect (fish group 1).

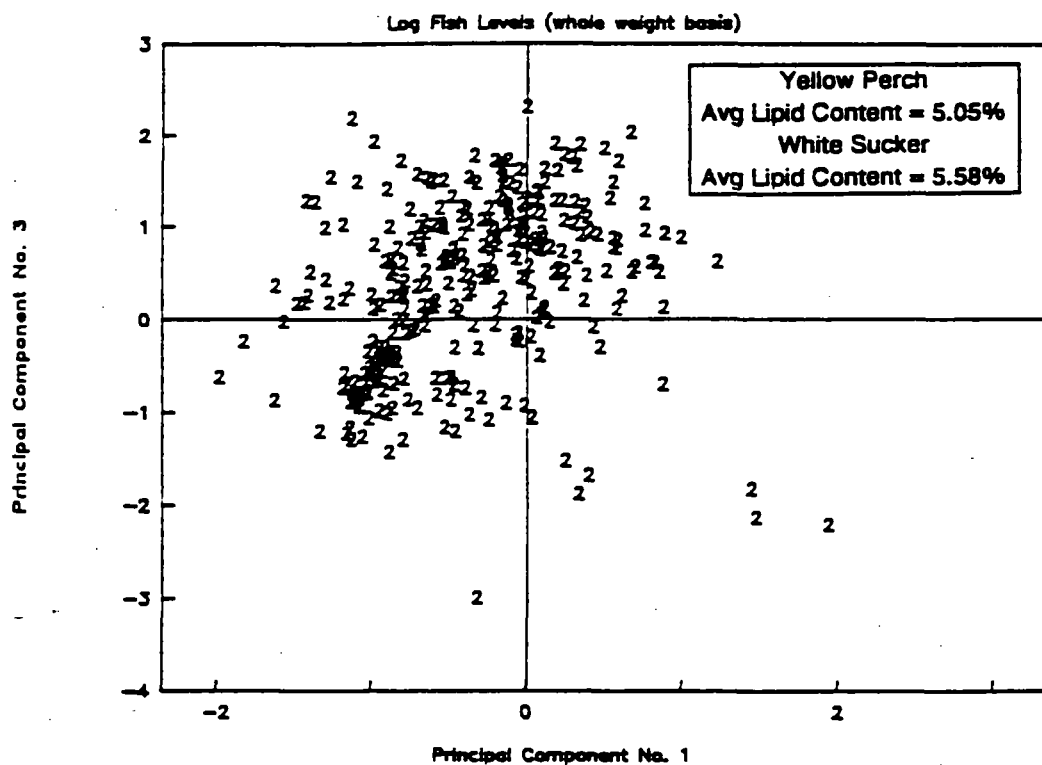


Figure 27. Principal component 3 vs. 1—Fish species effect (fish group 2).

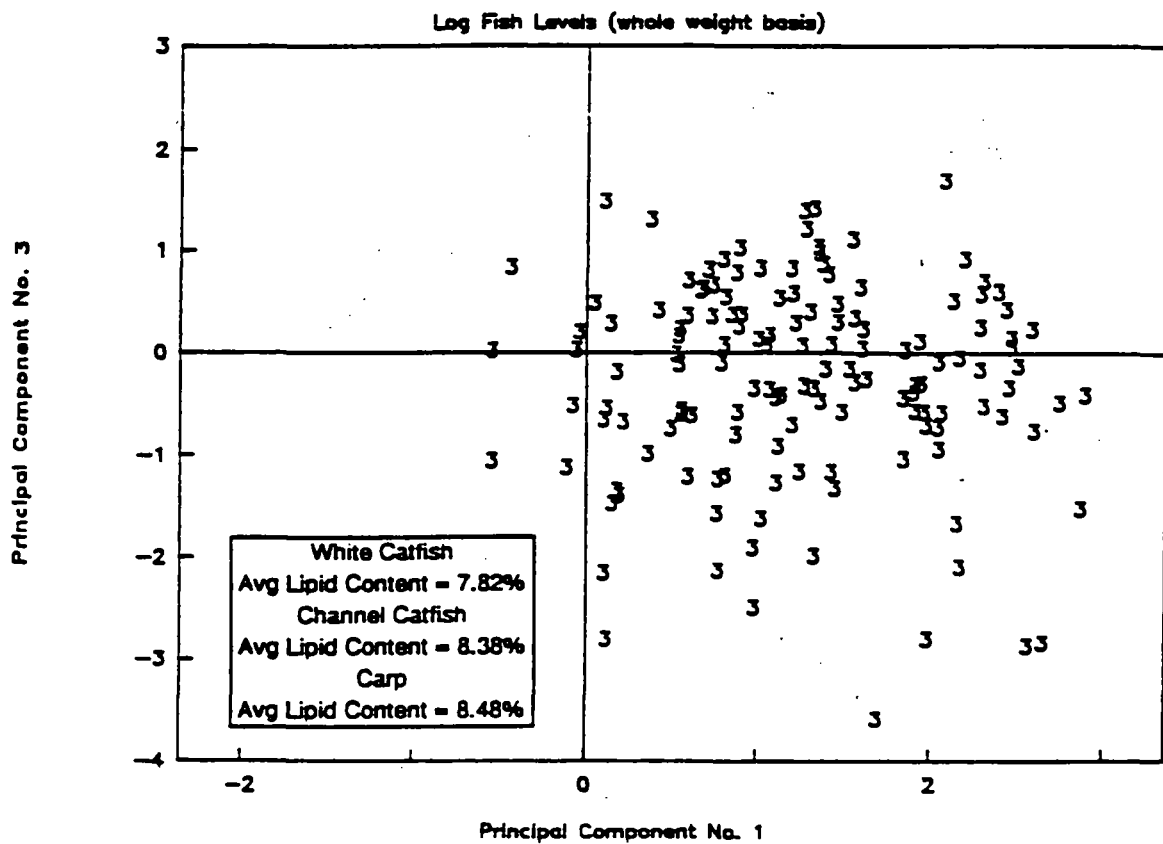


Figure 28. Principal component 3 vs. 1—Fish species effect (fish group 3).

**APPENDIX**

**MANUSCRIPT ACCEPTED AS PART OF THE DIOXIN'91 PROCEEDINGS  
FOR PUBLICATION IN *CHEMOSPHERE***

**Poster No. P114 Presented at Dioxin'91  
Research Triangle Park, North Carolina  
September 23 to 27, 1991**



# MULTIVARIATE STATISTICAL ANALYSES OF PCDD AND PCDF LEVELS IN FISH, SEDIMENT, AND SOIL SAMPLES COLLECTED NEAR RESOURCE RECOVERY FACILITIES

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## ABSTRACT

An environmental monitoring program was established to determine the impact of emissions from resource recovery facilities on the background of PCDDs and PCDFs. Fish, sediment, and soil samples were collected from areas surrounding five resource recovery facilities and one control site. Over 500 samples were analyzed to characterize the environmental levels before and after the resource recovery facilities initiated operations. All samples were analyzed for total tetra- through octa-homologs and 2,3,7,8-substituted isomers. The resulting data base was investigated by multivariate statistical techniques (cluster analysis and principal component analysis—PCA) to determine the relationships of the residue levels between sample types and sample locations. The results of the effort demonstrate distinct differences between the sample matrices. The PCA provided a clear separation of patterns for fish versus soil and sediment. In addition, there were distinct groupings of patterns influenced by fish species.

## INTRODUCTION

The State of Connecticut's Department of Environmental Protection through Public Act 86-332 established an environmental monitoring program in 1987 to assess the potential impact of emissions of PCDDs and PCDFs from resource recovery facilities (RRFs). The act requires both pre- and post-operational monitoring in the vicinity of existing and proposed RRFs. During the initial phase (1987 and 1988) of the environmental monitoring effort, a total of 295 fish, 185 sediments and 42 soil samples were collected for analysis for tetra- through octa- chlorinated PCDDs and PCDFs, with emphasis on the 2,3,7,8-substituted isomers. These samples, collected from six sites (five RRF sites and one control site), represent the pre-operational residue levels and the levels after the first year of operation. PCDDs and PCDFs were detected in all samples. A multivariate statistical analysis was proposed to investigate possible trends in concentration levels and interrelationships of PCDD and PCDF homologs and 2,3,7,8-substituted isomers between sample matrices.

Specific considerations in the application of the cluster analysis and PCA to the data set included:

- Changes in environmental levels and patterns of PCDDs and PCDFs since the initiation of operation of the resource recovery facilities.
- Comparability of environmental levels and patterns between the five operational and control sites.
- Consistency of PCDD and PCDF patterns among fish, sediment, and soil.
- Effects of fish species on the observed PCDD and PCDF patterns.
- Effects of variables such as grain size on observed residue levels in sediments and soils, and of lipid content on residue levels in fish.

## APPROACH

Data were generated for a total of 295 fish, 185 sediment, and 42 soil samples. In a first step, data distributions were examined and univariate and bivariate statistics were computed for all variables. However, due to the magnitude of the data base, pattern recognition—cluster analysis and principal component analysis (PCA)—was proposed as an exploratory tool to address the study objectives. This approach—investigation of potential multidimensional relationships among the analyte levels—was followed by classification of the samples into groups, if groups could be identified. Information on matrix type, fish species, and sampling locations was then used to interpret patterns in the data set.

The objective of the cluster analysis was to uncover possible significant interrelationships among analytes or groups of analytes. All concentrations were log-transformed. This approach was selected to resolve two potential problems: (1) non-normality of the PCDD and PCDF concentration levels and (2) sensitivity of the cluster analysis to extreme concentration values. Absolute correlations were used as the measure of similarity between any two analytes. The BMDP P1M procedure, cluster analysis of variables from the Biomedical Programs software package, was used to perform these analyses.

In PCA, as in cluster analysis, one concentrates on relationships within a set of variables. Whereas cluster analysis is a tool for determining whether groups of analytes with similar behavior exist, PCA is a technique used to reduce the dimensionality of the original set of variables in a different manner. The objective of PCA is to derive a few new components as linear combinations of the original variables which will provide a description of the structure of the data with as little loss of information as possible. The PCA was performed on the correlation matrix; i.e., the variables were autoscaled (standardized) so as to be treated with equal importance. All analyses were performed using the PCA option of the FACTOR procedure, followed by the SCORE procedure from the Statistical Analysis System (SAS) software package.

## DATA BASE DESCRIPTION

The data base contained information on a total of 522 field samples collected over a two-year period (1987 and 1988) around six cities, in and around 15 ponds. Approximately twice as many samples were collected in 1988 than in 1987. Fish from six species—brown bullhead, white sucker, yellow perch, white catfish, carp, and channel catfish—were collected. Various physical properties were measured for these samples—percent lipid for fish samples, and percent moisture, total organic carbon (TOC), and grain size (i.e., percent sand, silt, and clay) for sediment and soil samples.

All fish concentrations were reported on a wet, whole weight basis, while sediment and soil samples were reported on a dry weight basis. Throughout all statistical analyses, half the limit of detection value was used for those samples with levels below detection limits. When the analyte was reported at a trace level, that is, between the limit of detection and the limit of quantitation, the reported concentration was used in the analyses.

In at least one of the two collection years, 13 analytes had levels below detection limits in over 25% of the fish samples. However, only two analytes—2,3,7,8-TCDD and 1,2,3,7,8,9-HxCDF—had levels below detection limits in over 25% of the sediment or soil samples. (An upper limit of 25% for the total number of samples with levels below detection limit for a given analyte was selected as the cutoff point for that analyte to be included in some of the subsequent multivariate analyses.) Basic descriptive statistics were calculated for all 2,3,7,8-substituted isomers and homologs. Table 1 provides distributional statistics for total TCDF, total TCDF, 2,3,7,8-TCDF and 2,3,7,8-TCDD.

**Table 1. CONCENTRATION STATISTICS (pg/g)—TOTAL TCDF/D AND 2,3,7,8 - TCDF/D**

Analyte	Matrix	Year	Sample Size	Mean	Standard deviation	Minimum	Percentiles			
							25th	Median	75th	Maximum
Total TCDF	Fish	1987	90	5.26	4.12	0.10	1.61	4.36	7.48	17.50
		1988	205	5.46	5.56	0.07	1.55	4.20	7.75	45.70
	Sediment	1987	52	69.92	61.00	2.42	30.57	43.75	109.5	290.0
		1988	105	81.09	72.23	0.55	15.55	71.50	135.0	299.0
	Soil	1987	14	47.45	58.94	1.90	12.87	20.60	62.35	202.0
		1988	27	40.86	57.46	2.26	6.75	14.50	45.20	210.0
2,3,7,8-TCDF	Fish	1987	90	2.86	2.54	0.08	0.59	2.22	4.26	11.10
		1988	205	2.84	2.43	0.07	0.76	2.42	4.15	11.30
	Sediment	1987	52	17.34	18.72	0.69	6.32	9.97	23.13	101.0
		1988	105	18.31	19.71	0.35	2.82	13.80	26.75	85.00
	Soil	1987	14	9.39	13.39	0.48	2.32	3.20	11.67	46.00
		1988	27	7.58	13.53	0.36	1.26	2.58	5.37	61.60
Total TCDD	Fish	1987	90	1.81	1.99	0.05	0.45	0.90	2.45	8.37
		1988	198	1.17	1.25	0.05	0.36	0.70	1.46	6.22
	Sediment	1987	72	12.47	11.10	0.22	4.61	8.12	17.80	48.70
		1988	107	15.66	19.23	0.28	2.31	7.82	22.20	95.50
	Soil	1987	14	6.07	8.91	0.15	1.36	3.20	5.15	33.30
		1988	27	9.58	12.07	0.24	1.49	4.48	12.00	49.70
2,3,7,8-TCDD	Fish	1987	90	1.43	1.62	0.05	0.28	0.72	2.28	6.15
		1988	198	0.97	1.17	0.05	0.22	0.39	1.23	6.12
	Sediment	1987	72	3.14	4.79	0.09	0.59	1.38	2.66	23.10
		1988	107	1.59	2.47	0.18	0.45	0.88	1.75	18.00
	Soil	1987	14	0.20	0.09	0.11	0.14	0.17	0.23	0.42
		1988	27	0.64	1.37	0.13	0.15	0.21	0.37	6.00

#### CLUSTER ANALYSIS RESULTS FOR ALL ANALYTES

Cluster analysis was performed on the log-transformed concentrations of all 25 analytes (2,3,7,8-substituted isomers and homologs) separately for fish, sediment, and soil samples. Overall, the results showed that:

- PCDDs and PCDFs exhibit very different patterns in fish samples than in sediment and soil samples;
- In fish, only a few strongly correlated groups exist (group correlation coefficient above 0.80), with most of them containing only a small number of analytes;
- In sediment, all but five analytes (total TCDD, 1,2,3,7,8-PeCDF, 2,3,4,7,8-PeCDF, 1,2,3,7,8,9-HxCDF, and 2,3,7,8-TCDF) form a group with a group correlation coefficient of 0.91; and
- In soil, all but three analytes (1,2,3,4,6,7,8,9-OCDD, 1,2,3,7,8,9-HxCDF, and 2,3,7,8-TCDD) form a group with a group correlation coefficient of 0.90.

#### PRINCIPAL COMPONENT ANALYSIS RESULTS FOR 17 ANALYTES MEASURED IN ALL THREE MATRICES

A principal component analysis was performed using all three matrices and data from all 2,3,7,8-substituted isomers. The major PCA results are listed below:

- Matrix type provides a clear means of stratification of the samples into two groups: a) fish and b) sediment and soil.
- There is no separation by matrix within the group formed by soil and sediment samples.
- A large proportion (84.2%) of the variability in the data is explained by the combination of two principal components:
  - The first and predominant principal component is a combination of 12 analytes, with each having about equal and positive contribution (loadings) to this component. This component explains approximately 76% of the total variance in the data set.

- The second component (8.33 % of total variance) consists of 2,3,7,8-TCDD, 1,2,3,7,8-PeCDD, 2,3,4,7,8-PeCDF, OCDF, and OCDD. The first two analytes alone represent 55.3 % of that component: 2,3,7,8-TCDD with 40.2% and 1,2,3,7,8-PeCDD with 15.1%.

The profiles of the two major principal components are presented in Table 2, along with the variable loadings and their individual contributions (percent variance) to the variance explained by each principal component. The analytes with a variance contribution above average (1/17 or 5.9%, where 17 is the number of analytes included in the PCA) are shaded within each set.

Figure 1 depicts principal component 2 vs. 1 (displaying about 84.2% of the total variation in the data). Figure 1 shows a distinct separation of analytical results for sediments (S) and soils (T) versus fish samples (F). This grouping is not unexpected given the distinct differences observed in the raw analytical data for the PCDD and PCDF fingerprints. The PCDDs and PCDFs in fish samples consist primarily of the 2,3,7,8-substituted isomers. These isomers are recognized as the analytes that are also retained by other animal species. In contrast, the 2,3,7,8-substituted isomers are minor contributors to overall concentrations for the total PCDD and PCDF residue levels in sediments and soils. These findings are also supported by the results obtained via cluster analysis of the analytes in samples from the three matrices.

**Table 2. PRINCIPAL COMPONENT ANALYSIS RESULTS  
2,3,7,8-SUBSTITUTED ISOMER LEVELS  
IN ALL THREE MATRICES  
(281 FISH, 153 SEDIMENT, AND 41 SOIL SAMPLES)**

Analyte (Ordered by % variance)	Loading	Variance explained (%)	Cumulative variance (%)
<b>First Principal Component (75.9% of total variance explained) "Higher Chlorinated PCDDs and PCDFs"</b>			
1,2,3,7,8,9-HxCDD	0.98	7.4	7.4
1,2,3,4,6,7,8-HpCDF	0.96	7.1	14.5
1,2,3,4,6,7,8-HpCDD	0.95	7.0	21.6
1,2,3,4,7,8-HxCDD	0.95	7.0	28.6
1,2,3,4,7,8,9-HpCDF	0.94	6.8	35.4
1,2,3,6,7,8-HxCDF	0.93	6.7	42.2
2,3,4,6,7,8-HxCDF	0.93	6.7	48.9
1,2,3,6,7,8-HxCDD	0.93	6.7	55.6
1,2,3,4,6,7,8,9-OCDF	0.92	6.6	62.1
1,2,3,7,8-PeCDF	0.90	6.2	68.3
1,2,3,4,6,7,8,9-OCDD	0.89	6.2	74.5
1,2,3,4,7,8-HxCDF	0.87	5.9	80.4
1,2,3,7,8-PeCDD	0.78	4.7	85.1
2,3,7,8-TCDF	0.77	4.6	89.7
2,3,4,7,8-PeCDF	0.77	4.6	94.2
1,2,3,7,8,9-HxCDF	0.73	4.1	98.4
2,3,7,8-TCDD	0.46	1.6	100
<b>Second Principal Component (8.33% of total variance explained) "2,3,7,8-TCDD and 1,2,3,7,8-PeCDD"</b>			
2,3,7,8-TCDD	0.75	40.2	40.2
1,2,3,7,8-PeCDD	0.46	15.1	55.3
2,3,4,7,8-PeCDF	0.34	8.3	63.6
1,2,3,4,6,7,8,9-OCDF	-0.32	7.4	71.1
1,2,3,4,6,7,8,9-OCDD	-0.31	6.8	77.9

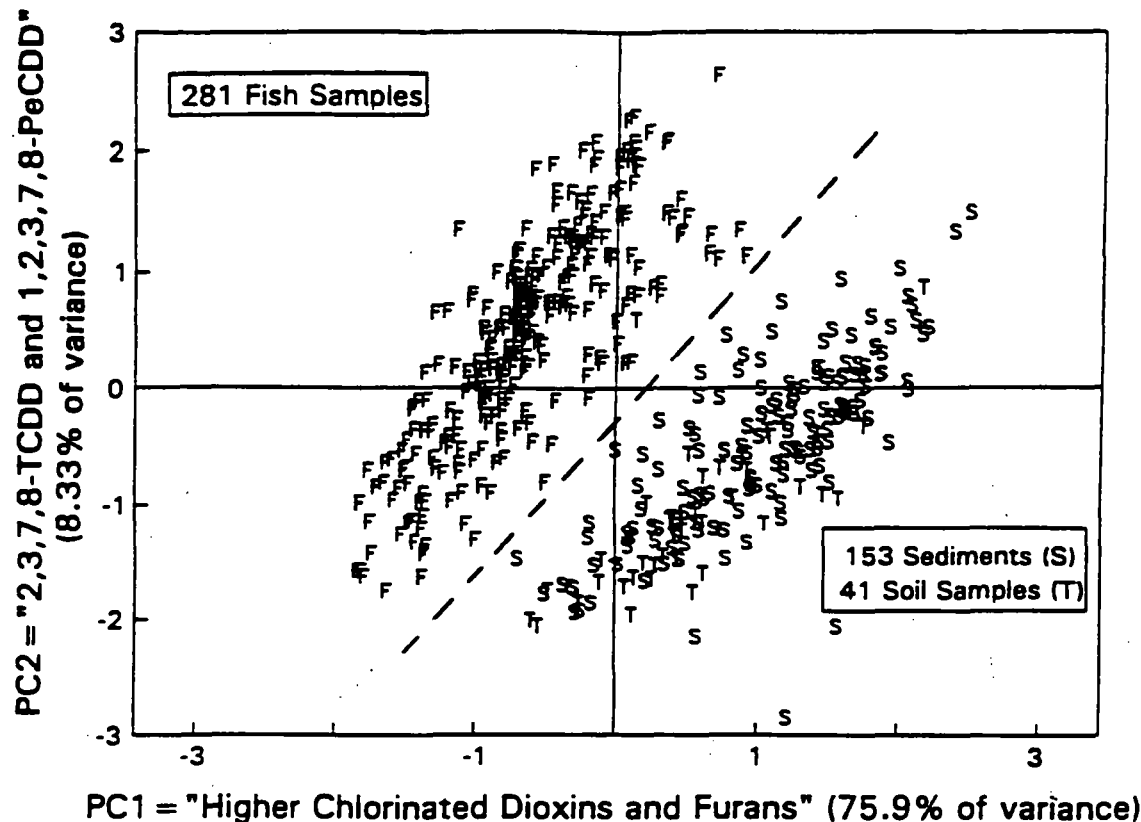


Figure 1. Plot of principal components 2 vs. 1 for fish, sediment, and soil log-transformed concentrations of 2,3,7,8-substituted isomers (fish levels on wet, whole weight basis, sediment and soil levels on dry weight basis).

#### PRINCIPAL COMPONENT ANALYSIS RESULTS FOR 17 ANALYTES MEASURED IN FISH SAMPLES

A series of PCA were performed using various combinations of analytes, matrices, and other available parameters. Based on the observation of the separation between fish and soil and sediment samples, additional PCA focused on the variables affecting fish residue levels. The effect of fish species on PCDD and PCDF uptake is shown here. Prior to PCA, the six fish species were categorized into three groups (brown bullhead; white sucker and yellow perch; and white catfish, carp, and channel catfish) by means of analysis of variance of the lipid content (percentage) of each sample. PCA was performed using the 2,3,7,8-substituted isomer concentrations in fish samples only (fish levels on a whole weight basis). Major PCA results are listed below:

- A relatively large proportion (69.4%) of the variability in the data is explained by the combination of two principal components:
  - The first and predominant principal component (57.8% of the variance) is a combination of hexa-, hepta-, penta-, and tetra-PCDDs, with each analyte having about equal and positive contribution to this component.
  - The second component (11.6 %) consists of analytes that were not detected in a large proportion of the fish samples. The component comprises hexa-, hepta-, penta-PCDFs and OCDD. Two analytes, 1,2,3,7,8,9-HxCDF and 1,2,3,4,7,8-HxCDF alone, contribute 60.7% to the variance explained by this component.
- The species provides some stratification of the fish samples in this principal component plot. There is almost complete separation of brown bullheads from white catfish, carps, and channel catfish. The middle group, consisting of white suckers and yellow perch, overlaps in the middle of the X-axis with both groups. As percent lipid increases, so do PCDD and PCDF residue levels as measured by the first principal component. The separation along the second principal component is mainly due to geographical location of the sample collection.

The profiles of the two major principal components are presented in Table 3, along with the variable loadings and their individual contributions (percent variance) to the variance explained by the given principal component. The analytes with a variance contribution above average (1/17 or 5.9%, where 17 is the number of analytes included in the PCA) are shaded within each set. Figure 2 depicts principal component 2 vs. 1 (displaying about 69.4% of the total variation in the data).

**Table 3. PRINCIPAL COMPONENT ANALYSIS RESULTS  
2,3,7,8-SUBSTITUTED ISOMER LEVELS  
IN 281 FISH SAMPLES**

Analyte (Ordered by % variance)	Loading	Variance explained (%)	Cumulative variance (%)
<b>First Principal Component (57.8% of total variance explained) "Hexa-, Hepta-, Penta-, Tetra-PCDDs"</b>			
1,2,3,7,8,9-HxCDD	0.92	8.7	8.7
1,2,3,6,7,8-HxCDD	0.92	8.5	17.2
1,2,3,4,7,8-HxCDD	0.88	7.9	25.1
1,2,3,4,6,7,8-HpCDD	0.87	7.8	32.9
1,2,3,4,6,7,8-HpCDF	0.87	7.7	40.6
1,2,3,7,8-PeCDD	0.86	7.6	48.2
2,3,7,8-TCDD	0.85	7.3	55.5
1,2,3,7,8-PeCDF	0.80	6.5	62.0
2,3,4,6,7,8-HxCDF	0.75	5.8	67.8
1,2,3,4,6,7,8,9-OCDF	0.73	5.4	73.2
2,3,4,7,8-PeCDF	0.72	5.3	78.5
1,2,3,6,7,8-HxCDF	0.72	5.2	83.7
1,2,3,4,7,8,9-HpCDF	0.68	4.8	88.5
1,2,3,4,6,7,8,9-OCDD	0.66	4.5	93.0
1,2,3,4,7,8-HxCDF	0.62	3.9	96.9
2,3,7,8-TCDF	0.46	2.2	99.0
1,2,3,7,8,9-HxCDF	0.31	1.0	100
<b>Second Principal Component (11.6% of total variance explained) "Hexa-, Hepta-, Penta-PCDFs, and OCDD"</b>			
1,2,3,7,8,9-HxCDF	0.89	40.0	40.0
1,2,3,4,7,8-HxCDF	0.64	20.8	60.7
1,2,3,6,7,8-HxCDF	-0.40	8.2	68.9
1,2,3,4,7,8,9-HpCDF	0.35	6.3	75.2
2,3,4,7,8-PeCDF	-0.35	6.2	81.5
1,2,3,4,6,7,8,9-OCDD	-0.35	6.1	87.6

## CONCLUSIONS

Principal component analysis and cluster analysis provide patterns of PCDDs and PCDFs in fish that are very different from those in soil and sediment, but there is no distinction in patterns between soils and sediments. Principal component analysis also shows distinct groupings between levels of PCDDs and PCDFs by fish species. These results hold whether using lipid-adjusted or whole weight values. The correlations between PCDD and PCDF levels in fish and those in sediments should be further investigated, with the total organic carbon-adjusted levels in sediments and lipid-adjusted levels in fish being used as the basis for these future analyses.

The short time period (one year) during which the resource recovery facilities were in operation does not warrant comparisons of PCDD and PCDF levels over time. At least four to five years of data may be required to detect a trend in PCDD and PCDF levels that could be attributed to the operation of resource recovery facilities. Further analyses are necessary to investigate whether patterns observed in fish, sediment, and soil samples are indicative of environmental contributions other than combustion.

## ACKNOWLEDGMENT

This work was supported by the State of Connecticut, Department of Environmental Protection, through Personal Services Agreement No. 91-1526

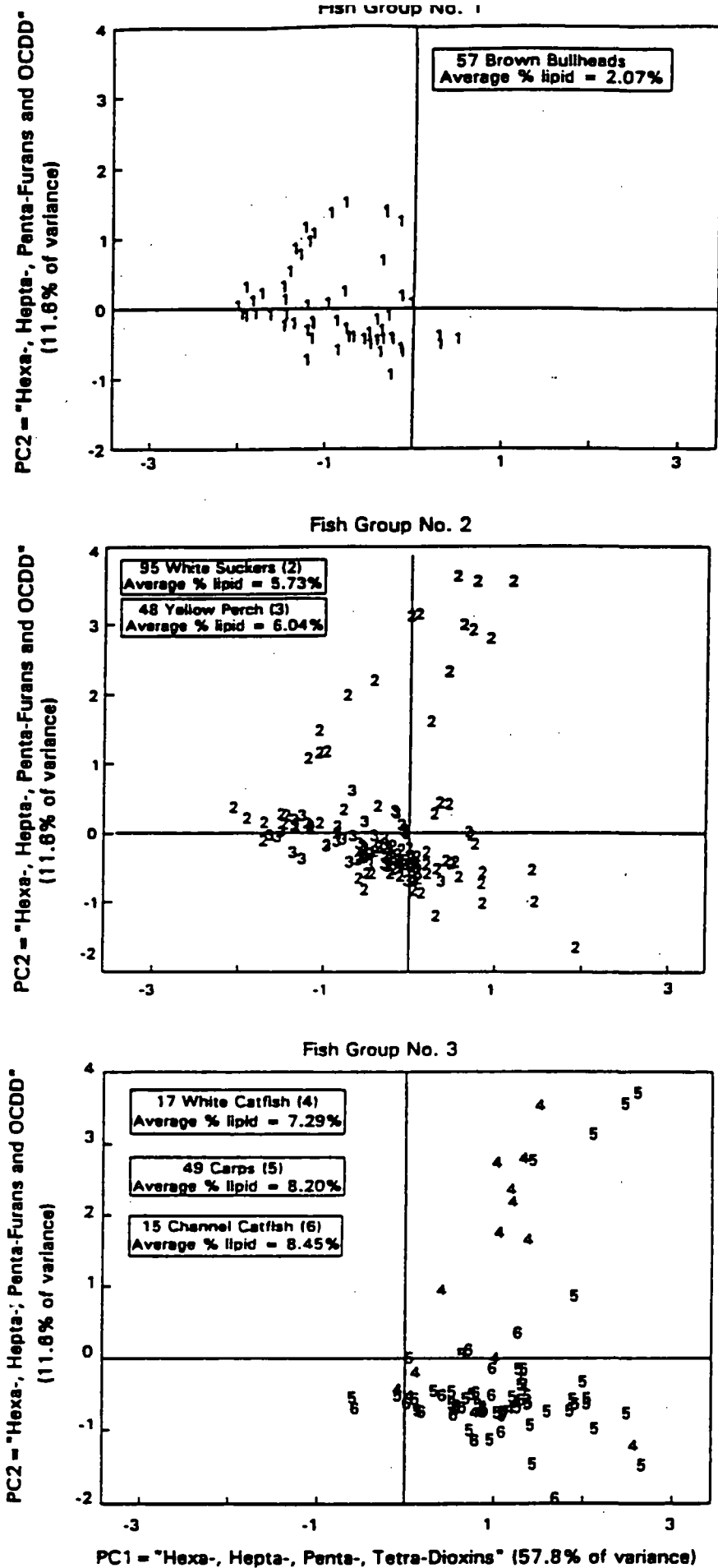


Figure 2. Plot of principal components 2 vs. 1 for log-transformed concentrations of 2,3,7,8-substituted isomers in fish (concentrations on wet, whole weight basis).



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# INVOICES

August 20, 1993

Mr. Richard Desrosiers  
Environmental Laboratories  
142 Temple Street  
New Haven, CT 06510

Subject: Review of P  
Site, MRI Project

Raymark Industries, Inc.,

Dear Mr. Desrosiers:

Midwest Research Institute has reviewed the analytical data and background information pertaining to the Raymark Industries, Inc. We have reviewed the data with respect to assisting Environmental Laboratories, Inc. (ELI), in assessing the origination of PCDD/PCDF residues on site. ELI has provided to MRI a number of items which include historical data and perspective of production efforts at the Raymark Industries site, data tables (hardcopy and Lotus 1-2-3 format) for the targeted analytes, and hardcopy results for each of the samples for the PCDDs and PCDFs to allow review of the chromatographic patterns.

In approaching this effort, MRI has reviewed all information supplied by ELI pertaining to the site history, confirmed the calculations for the determination of TEF, assessed the potential for statistical evaluation of the results, reviewed the chromatographic patterns, reviewed available literature, and contacted a number of other scientists with experience in the area of PCDDs, PCDFs, and PCBs.

Our conclusions based on the data received to date reflect that the levels of PCDDs and PCDFs detected in the samples from the Raymark Industries site reflect contribution from a number of sources, which may include the background contribution from general atmospheric transport, residues from Aroclor 1268, and some of the phenoxy herbicides (2,4-D, 2,4,5-T, and 2,4,5-TP) detected in the field samples.

## PCDD/PCDF BACKGROUND LEVELS IN THE STATE OF CONNECTICUT

MRI has previously worked with the State of Connecticut to determine background levels of PCDDs and PCDFs in soil and sediment from several locations throughout the state to develop a baseline and evaluation of the impact of municipal waste incineration. These data have demonstrated a background of PCDDs and PCDFs in



the state. Table 1 presents a summary of data from sites located in Bridgeport, Connecticut. The data base from which this information was extracted contains data on up to 971 samples (77 soils, 344 sediments, and 550 fish) collected from 1987 to 1990 from areas within or adjacent to nine different municipalities. MRI has contacted the Connecticut Department of Environmental Protection to obtain approval for the release of our final report, "Multivariate Statistical Analysis of Dioxin and Furan Levels in Fish, Sediment and Soil Collected near Resource Recovery Facilities" (Final Report for PSA Contract 91-1526).

A comparison of the general Connecticut background dioxins and furans and the ELI results illustrates the difference between these data. Figure 1 shows the average level dioxins and furans in the ELI expressed as a percent of the total dioxin and furan level. In this figure, the average level of OCDD is more than 40% of the total of all dioxins and furans. Also the average level of furans in these samples is nearly 20% for HxCDF. Figure 2 shows a similar treatment of the samples which MRI has analyzed from Bridgeport, Connecticut. In these samples, the average level of OCDD is over 60% of the total of all dioxins and furans. This figure also illustrates the fact that the overall level of furans in the Bridgeport results is lower than in the ELI data. This higher level of furans may be result of the presence of Aroclor 1268.

#### **PCDDS/PCDFS FROM AROCLORS**

As per our past telephone discussions, the Aroclor 1268 may be an important contributor to the levels of PCDFs observed in the sample. The chromatographic patterns observed for the TCDFs, PeCDFs, and HxCDFs reflect some similarities with other Aroclors that have been studied in round robin investigations by the Electric Power Research Institute (EPRI) and in investigations of thermal degradation products from dielectric fluids conducted by MRI. Unfortunately, the samples in both cases focused on Aroclors that were typically used in transformers. Aroclor 1268 was not used for the same purpose as it is a material of higher molecular weight and hence was not suitable as a fluid. At this time we have not been able to identify any research efforts or investigations pertaining to the levels of PCDFs in Aroclor 1268.

Some examples of chromatographic patterns for Aroclor 1254 that has been thermally degraded are presented in the series of Figures presented as attachments to this letter. These data were generated by MRI as an investigation of the thermal degradation of dielectric fluids ("Thermal Degradation Products from Dielectric Fluids," M. D. Erickson, et al., EPA-560/5-84-009, December 1984). The chromatographic patterns for the tetra, penta, and hexachlorinated dibenzofurans demonstrate some similarities with the data in the soil samples showing positive results. The other striking factor is the relative difference in the PCDF to PCDD ratios. The PCDFs are greater than the PCDDs by factors of up to 20 or more in some of the field samples from the Raymark Industries site.

Additional data on PCDFs and PCDDs in Aroclor mixtures were obtained from the EPRI report on PCDFs and PCDDs in Utility Transformers and Capacitors (EPRI EL/EA-4858, December 1986). MRI has spoken with the EPRI project officer on that work, Mr. Gil Addis, and two of the principal investigators from IIT Research Institute and Battelle Columbus Laboratories. The effort on this work focused on Aroclors used within the utility industry. Some of the pertinent data from that effort follow.

**Aroclor 1016.** Aroclor 1016 did not exhibit any PCDDs or PCDFs in the samples analyzed in this study. Approximate detection limits appear to be on the order of 10 ng/g of sample.

**Aroclor 1242.** Aroclor 1242 demonstrated responses to the TCDF, PeCDF, HxCDF, and OCDF. The PCDDs were not detected in this particular matrix. The total PCDF values ranged from approximately 130 ng/g for the OCDF up to 870 ng/g for the TCDFs. The TCDFs were the predominant responses for this mixture.

**Aroclor 1260.** Aroclor 1260 also demonstrated responses to the PCDFs, but not the PCDDs. The PCDF homolog concentrations were as follows OCDF (4150 ng/g) > HpCDF (1985 ng/g) > HxCDF (1400 ng/g) > PeCDF (1040 ng/g) > TCDF (600 ng/g).

**Askarel Load Center Network Transformer.** This sample contained 70% Aroclor 1260, 29% trichlorobenzenes, and 1% tetrachlorobenzene from a transformer of over 30 years' service. PCDDs were not detected in the sample to any significant extent. The data demonstrated a different order of predominance of the PCDFs: HxCDF (2300 ng/g) > HpCDF (1590 ng/g) > PeCDF (1530 ng/g) > TCDF (760 ng/g) > OCDF (560 ng/g).

Some effort was made to correlate the Aroclor 1268 concentrations to the total PCDDs and PCDFs using the I-TEF values. However, a graphical representation of the data did not result in any clear or coincident relationship of these values. The correlation of data may be affected by the homogeneity of the matrices analyzed.

Using the information obtained from the EPRI round robin study on various dielectric fluids an estimate of the contaminant levels of PCDFs in the soil samples resulting from the Aroclor 1268 was developed as follows.

If one assumes that the TCDF concentration of 8.1 ng/g in field sample 92-1949 is attributed to the Aroclor 1268 (determined at - 30 µg/g of soil), the contaminant level of the TCDF in the Aroclor would originally be 270 µg/g or

$$(8.1 \times 10^{-9} / 30 \times 10^{-6}) = 0.27 \times 10^{-3} \text{ g/g or } 270 \text{ µg/g}$$

This contaminant level is considerably higher than found in any other PCB mixture reported in the EPRI study. Some data on the thermal degradation products of PCBs indicate that conversion efficiencies of up to 2% can be achieved. Hence without information on the residual levels of PCDFs and PCDDs in the Aroclor 1268, it must be considered that some thermal degradation of the product may have occurred under chemical processing conditions or that the contribution is from other sources outside the manufacturing processes and procedures.

## **PCDDS/PCDFS FROM PHENOXY HERBICIDES**

Our review of the data tables indicates some potential association of the PCDD/PCDF levels with the presence of phenoxy herbicides in a number of the samples. However, as with the PCB data, there does not appear to be a strict statistical correlation with any one of the herbicide residues pertaining to the overall TCDD TEA values. Attachment 2 to this letter summarizes available published literature through approximately 1984 ("Guidelines for Determination of Halogenated Dibenzo-*p*-dioxins and Dibenzofurans in Commercial Products," EPA-560/5-87/007, September 1987).

Other sources of information in commercial products, particularly the journal Chemosphere, have been reviewed. While additional information has been published regarding PCDD/PCDF levels, the information is consistent with citations from the late 1970s and early 1980s demonstrating the major contaminants are PCDDs. In many products, particularly 2,4,5-T, the primary contaminant is 2,3,7,8-TCDD which was not present in the field samples.

## **TOXICITY EQUIVALENTS**

Toxic equivalence is an attempt to estimate the total cumulative toxic effect of the variety of dioxin and furan isomers which are detected in a sample. Using this approach, the quantitated result for the various dioxins and furans can be reduced to a single number (TEF). At best, this cumulative dioxin/furan result is an estimate and it should be applied with caution. The purpose of this approach is to provide a basis for comparison of the dioxin/furan results from different samples. As such it has limited usefulness when comparing measured results from different samples. This technique has very little value when it is calculated from estimated results such as detection limits.

The majority of the samples from ELI reviewed for this report had no detectable dioxins other than OCDD (the least toxic congener). If these congeners are not detected, they would not be expected to contribute to the overall estimate of toxicity as calculated using the TEF formula.

However, there are several approaches to the use of detection limits in the calculation of TEF. In the calculation of TEF, a congener which is not detected can have a value of zero, or half the analytical detection limit or the detection limit. When the detection limit is used in the calculation of TEF, a comparison of results or a regulatory decision can be influenced by the sensitivity of the analytical method and not by the actual amount of dioxin or furan in the sample. Therefore, the approach used for non-detects can have a significant impact on the estimated TEF value.

If there are any questions regarding this information, please call. A hardcopy of this letter and contents will be forwarded under separate cover.

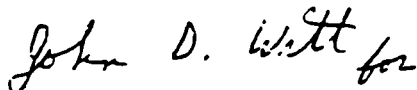
Sincerely,

MIDWEST RESEARCH INSTITUTE



John S. Stanley, Ph.D.  
Section Head  
Analytical Chemistry

Approved:



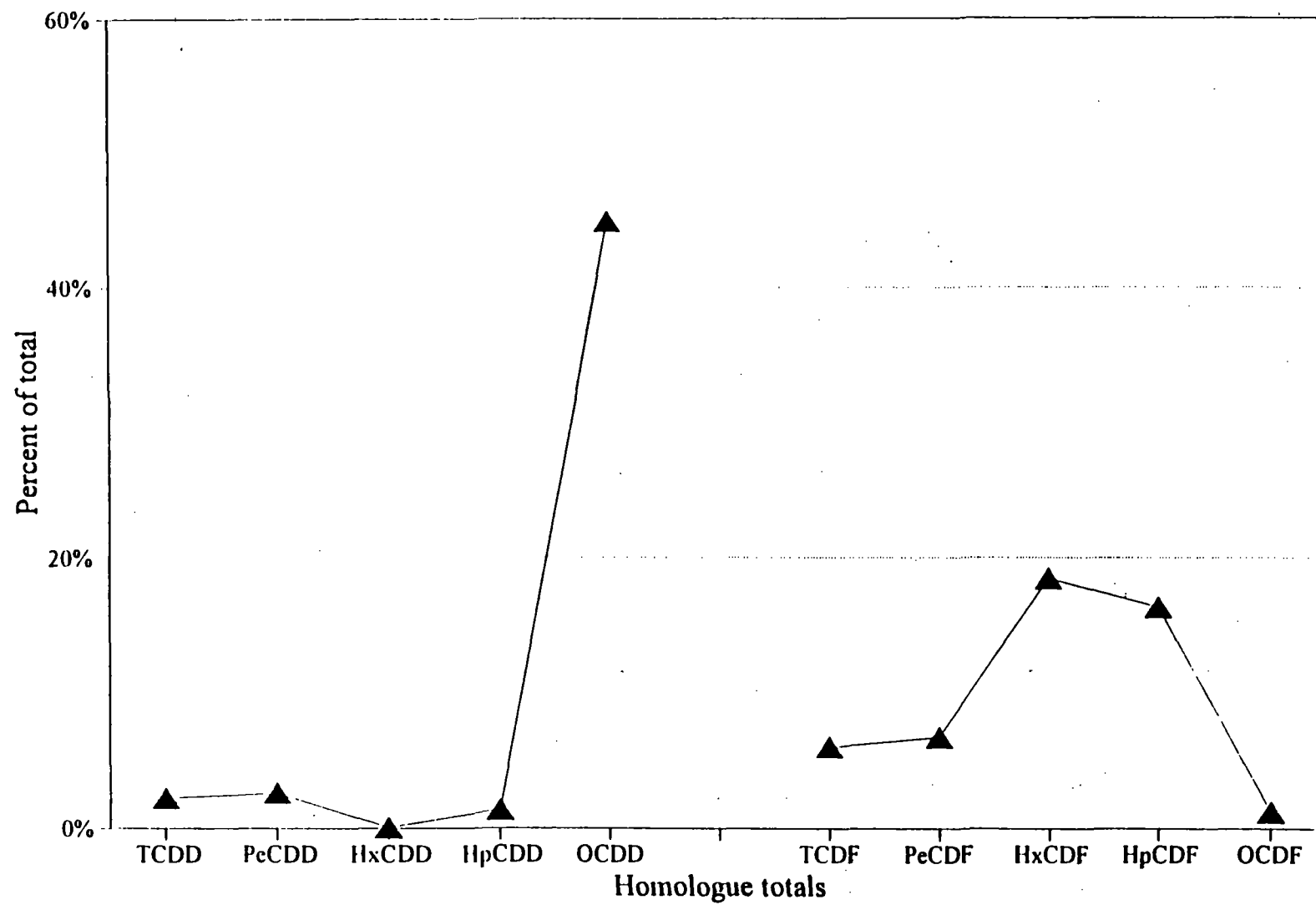
Don D. Gay, Ph.D.  
Director  
Chemical Sciences Department

**ATTACHMENT 1**

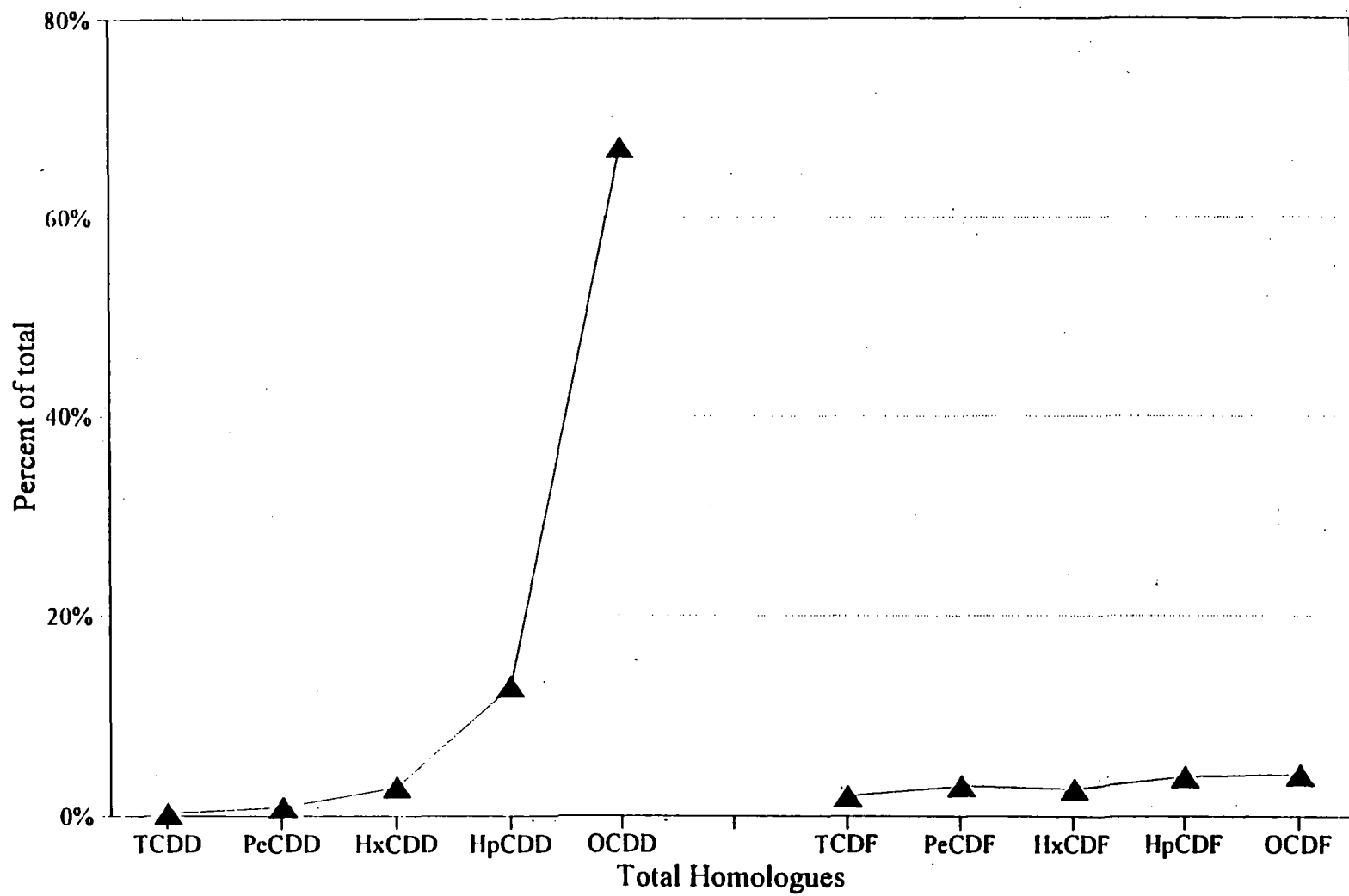
**Background Levels of PCDDs/PCDFs  
in the  
Bridgeport, Connecticut, Area**

# ELI Dioxins/Furans

Totals



# Bridgeport Dioxins/Furans Totals



BRIDGEPORT SUMMARY (pg/g, dry weight basis)

Analyte	Lab ID	30264	30265	30266	30267	30268	30271	30272	30273
TCDF		238	929	404	29.5	218	59.1	39.5	68
TCDD		49.9	288	115	5.63	38.5	5.57	5.86	11.2
PeCDF		236	1330	470	30.9	405	90.5	56.6	105.4
PeCDD		168	718	151	5.96	79.4	22.4	10.8	20.2
HxCDF		373	1610	370	24.5	365	108	57.6	97.2
HxCDD		430	1670	434	25.9	380	118	84.4	146
HpCDF		552	1990	574	40.6	635	214	160	234
HpCDD		1843	5587	1340	95.6	2039	599	323	476
2378TCDF		47.7	206	65.8	5.08	39.7	15.6	10.8	18.3
2378TCDD		13.9	51.5	6.23	ND( .193 mpc)	ND( 1.24 mpc)	0.762	ND( .412)	ND( .383 mpc)
12378PeCDF	ND( 22.7 mpc)		82.9	22	1.24	13.4	5.16	3.95	6.95
23478PeCDF		20.1	96.9	27.2	1.77	18.3	6.51	4.88	8.42
12378PeCDD		19.6	87.8	13.3	0.78	7.38	2.77	1.4	2.25
123478HxCDF		59.2	277	46.4	3.04	25.2	11.6	5.95	9.77
123678HxCDF		18.4	94.4	18.1	1.26	14.5	5.15	2.56	4.33
234678HxCDF		28.6	134	28.9	2.24	26.7	8.49	4.34	7.71
123789HxCDF	ND( .488)		ND( 9.13 cdl)	ND( 1.72)	ND( .578 mpc)	ND( .647)	ND( .325)	ND( .203)	ND( .964 mpc)
123478HxCDD	ND( 10.3 mpc)		42	11.1	ND( .581 mpc)	10.4	2.25	2.08	2.98
123678HxCDD		41.4	150	35.6	1.99	36.4	8.62	5.99	8.82
123789HxCDD		36.3	146	32.8	2.15	30.6	8.15	5.33	8.5
1234678HpCDF		253	1100	273	18.7	252	93	67.6	101
1234789HpCDF		21	70.5	17	ND( 1.19 mpc)	15.1	3.92	2.36	4.04
1234678HpCDD		773	2330	565	42	831	187	122	192
12346789OCDF		478	1480	665	70.5	772	300	259	380
12346789OCDD		7160	16700	5210	364	6650	1720	1220	1860
I-TE		86.7	356	80.1	5.50	60.6	20.3	13.6	21.9
TOC (mg/kg)		31,900	87,500	43,700	5,120	50,100	34,000	23,000	38,000
Grain Size:									
Sand (%)		45	55	28	82	34	74	60	15
Silt (%)		45	36	58	8	52	21	35	75
Clay (%)		10	9	14	10	14	5	2	10



**BRIDGEPORT SUMMARY (pg/g, dry weight basis)**

Analyte	Lab ID	30274	30275	30276	30277	30278	30279	30280	30281
TCDF		70	64.1	60	56.8	14.6	26.6	60.6	90.6
TCDD		9.12	10.9	1.08	1.61	2.7	2.86	4.86	10.8
PeCDF		150	93.4	103	71.9	15.4	35.3	71.9	110
PeCDD		43.9	37.9	30.9	23.9	4.91	7.83	20.7	34.6
HxCDF		354	78	122	89.6	4.11	22.2	22	52
HxCDD		365	182	140	102	13.4	21.6	36.4	45.6
HpCDF		743	261	155	121	9.24	16.8	24.9	30
HpCDD		2540	757	723	557	39.7	85.5	89.3	126
2378TCDF		22.2	17.2	17.4	14.7	4.2	6.55	20.3	28.4
2378TCDD		2.18	1.66	ND( .483 mpc)	ND( .736)	ND( .631)	ND( .459)	ND( .754)	ND( .434 mpc)
12378PeCDF		12.3	5.87	4.92	4.2	0.98	1.78	4.38	5.41
23478PeCDF		9.02	5.69	10.1	6.07	1.57	2.71	5.05	7.89
12378PeCDD		11.1	5.94	ND( 3.92 mpc)	3.2	ND( .299 mpc)	0.718	1.28	2.01
123478HxCDF		30.7	14.6	11.5	9.77	1.78	3.22	5.58	7.3
123678HxCDF		17.5	7.01	5.62	4	0.78	0.211	1.91	3.55
234678HxCDF		25.6	11.2	9.08	7.35	1.68	3.07	4.55	5.74
123789HxCDF		ND( .592)	ND( .259)	ND( .546)	ND( .355)	ND( .0844 mpc)	0.537	ND( .303)	ND( .51 mpc)
123478HxCDD		12.8	ND( 1.96 mpc)	5.93	ND( 3.23 mpc)	ND( .146 mpc)	1.08	1.22	ND( .727)
123678HxCDD		58.7	21.2	13.6	14.6	ND( .465 mpc)	1.4	2.56	2.16
123789HxCDD		47.3	16.2	13.2	11.5	1.47	2.5	3.57	4.34
1234678HpCDF		318	109	69.6	57.3	4.74	9.73	15.4	17.7
1234789HpCDF		22.2	4.85	5.28	4.29	0.757	0.999	ND( 1.53 mpc)	1.72
1234678HpCDD		1310	319	338	260	17.7	35.1	37.2	53.4
12346789OCDF		603	236	120	98	9.44	10.7	36.7	18.5
12346789OCDD		10600	2580	4530	3560	519	1210	799	1970
I-TE		67.1	27.1	28.5	22.0	4.30	7.42	12.1	17.8
TOC (mg/kg)		130,000	93,000	100,000	88,000	35,000	150,000	220,000	240,000
Grain Size:									
Sand (%)		45	46	28	64	90	99	94	90
Silt (%)		53	53	72	36	8	1	6	10
Clay(%)		2	1	0	0	0	0	0	0

## BRIDGEPORT SUMMARY (pg/g, dry weight basis)

Analyte	Lab ID	30282	30283	32586	32587	32588	32589	32590	32591
TCDF		2.04	1.58	40.9	272	8.67	37.7	57.1	159
TCDD		ND( .255)	ND( .233)	4.02	46.9	0.516	2.97	3.13	13.8
PeCDF		8.6	6.83	73.5	399	17.2	91.2	173	479
PeCDD		2.11	ND( .338)	27.6	121	6.03	23.5	29.3	55
HxCDF		10.9	7.57	73.7	213	15.6	64.9	140	370
HxCDD		12.9	4.93	59.3	233	23.6	48.8	62.4	181
HpCDF		17.5	13.1	80.2	255	16.9	40	149	198
HpCDD		65.9	44.1	303	909	129	265	286	846
2378TCDF		0.943	ND( .323 mpc)	8.87	57.4	2.21	8.01	11.1	18.2
2378TCDD		ND( .255)	ND( .233)	ND( .298 mpc)	1.74	ND( .43)	ND( .386 mpc)	ND( .343 mpc)	0.955
12378PeCDF		0.493	ND( .118 mpc)	3.55	23.4	0.798	3.22	4.51	6.64
23478PeCDF		0.535	0.324	4.74	25.8	ND( 1.35 mpc)	4.54	6.63	10.5
12378PeCDD		0.366	ND( .0952 mpc)	2	6.65	0.614	1.36	2.07	5.53
123478HxCDF		0.952	ND( .314 mpc)	7.9	ND( 61.3 mpc)	2.76	7.13	11.6	17.5
123678HxCDF		ND( .567 mpc)	0.274	3.9	ND( 19.2 mpc)	1.37	3.19	5.43	13.6
234678HxCDF		0.859	0.679	5.77	30.1	1.92	5.33	9.63	25.8
123789HxCDF		ND( .0677 mpc)	ND( .0597 mpc)	ND( .35)	ND( 1.02)	ND( .18 mpc)	ND( 2.36 cdl)	ND( .565)	ND( 1.68)
123478HxCDD		ND( .223 mpc)	ND( .0191 mpc)	2.04	ND( 1.33)	0.824	1.86	ND( 2.34 mpc)	ND( 2.05 mpc)
123678HxCDD		2.05	1.3	5.8	26.9	1.9	3.17	6.84	23.5
123789HxCDD		1.02	0.744	5.36	20.6	2.22	4.28	ND( 5.92 mpc)	19.1
1234678HpCDF		6.79	5.13	37.9	138	9.11	20.5	71.6	85.8
1234789HpCDF		0.562	ND( .437 mpc)	2.16	14.3	0.761	1.88	4.87	6.62
1234678HpCDD		33.4	23.6	142	377	53.6	100	137	394
12346789OCDF		15.5	12.1	48.9	86.2	13.1	45	110	142
12346789OCDD		448	436	3420	4790	3130	4450	2520	6150
I-TE		2.87	2.04	15.8	60.3	6.84	15.2	18.2	38.2
TOC (mg/kg)		3,600	16,000	21,000	75,000	84,000	64,000	53,000	74,000
Grain Size:									
Sand (%)		90	100	53	- (I)	- (I)	44	56	48
Silt (%)		1	0	41	-	-	50	42	48
Clay (%)		0	0	6	-	-	6	2	4

**BRIDGEPORT SUMMARY (pg/g, dry weight basis)**

Analyte	Lab ID	32592	31460		
TCDF		101	151	ND(0.5);	1.58 - 929
TCDD		8.41	29.5	ND(0.5);	0.516 - 288
PeCDF		211	119	ND(0.5);	6.83 - 1330
PeCDD		41.4	166	ND(0.5);	2.11 - 718
HxCDF		143	524	ND(1.25);	4.11 - 1610
HxCDD		86.6	525	ND(1.25);	4.93 - 1670
HpCDF		97.4	745	ND(1.25);	9.24 - 1990
HpCDD		386	3610	ND(1.25);	39.7 - 5587
2378TCDF		25.3	44	ND(0.5);	0.943 - 206
2378TCDD	ND( .878 mpc)		12.8	ND(0.5);	0.762 - 51.5
12378PeCDF	11.1	ND( 25.6 mpc)		ND(0.5);	0.493 - 82.9
23478PeCDF	9.1	7.63		ND(0.5);	0.324 - 96.9
12378PeCDD	3	19.9		ND(0.5);	0.366 - 87.8
123478HxCDF	16.4	72.4		ND(1.25);	0.952 - 277
123678HxCDF	6.95	28.5		ND(1.25);	0.211 - 94.4
234678HxCDF	11.8	41.8		ND(1.25);	0.679 - 134
123789HxCDF	ND( .297)	1.84		ND(1.25);	0.537 - 1.84
123478HxCDD	ND( .969 mpc)	15.5		ND(1.25);	0.824 - 42
123678HxCDD	9.66	58.4		ND(1.25);	1.300 - 150
123789HxCDD	8.16	42.9		ND(1.25);	0.744 - 146
1234678HpCDF	49.7	317		ND(1.25);	4.74 - 1100
1234789HpCDF	3.6	23.5		ND(1.25);	0.562 - 70.5
1234678HpCDD	168	1560		ND(1.25);	17.7 - 2330
12346789OCDF	53.9	638		ND(2.5);	9.440 - 1480
12346789OCDD	3850	> 9700		ND(2.5);	364 - 16700
I-TE		26.1	87.1	0.83 (i);	2.040 - 356
TOC (mg/kg)		71,000	49,500	ND(1,040)	3600 - 240000
Grain Size:					
Sand (%)		71	47	-	15 - 100
Silt (%)		28	43	-	0 - 75
Clay(%)		0	10	-	0 - 14

## **ATTACHMENT 2**

### **Example Chromatograms from Thermally Degraded PCB Mixtures**

**(Taken from EPA 560/5-84-009, December 1984,  
Thermal Degradation Products from Dielectric Fluids)**

MID MASS CHROMATOGRAMS

DATA: 8201H27R6 #1

SCANS 900 TO 1100

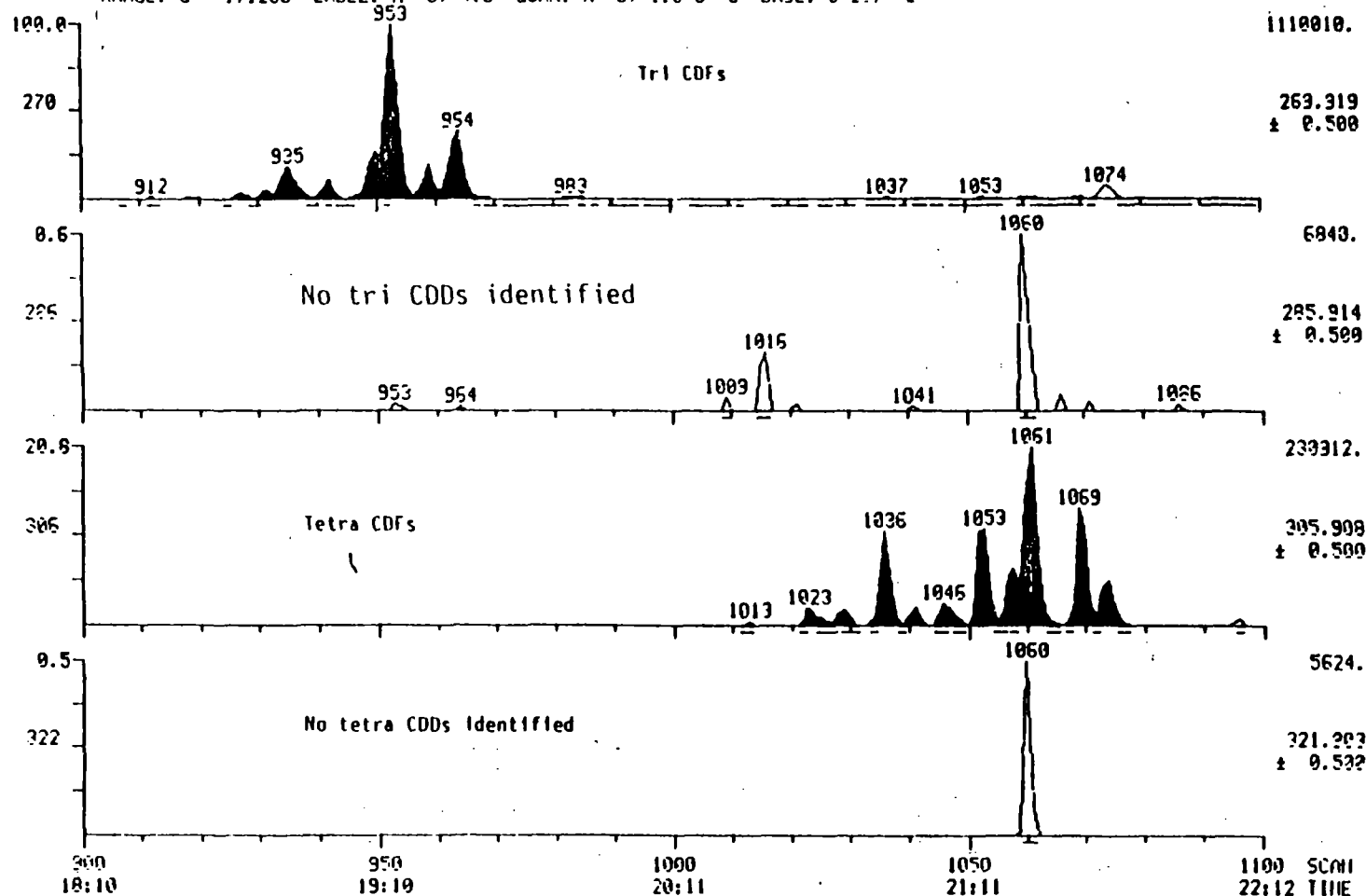
08/27/84 11:27:00

CAL1: MID250H21 #2

SAMPLE: 8201A23-RUN 8-22-52-S500 IUL INJ

COND.S: -200000V 70EV 11A 0E5-30M-80-2H-320-10/

RANGE: G 1,1200 LABEL: H 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3



Tri and tetra CDFs and CDDs in sample 8-22-52-S500,  
silicone oil at 500 µg/g PCB.

MID MASS CHROMATOGRAMS

03/12/84 17:16:00

SAMPLE: 8-22-52-S500 IUL PC50/F/BN CL5-E

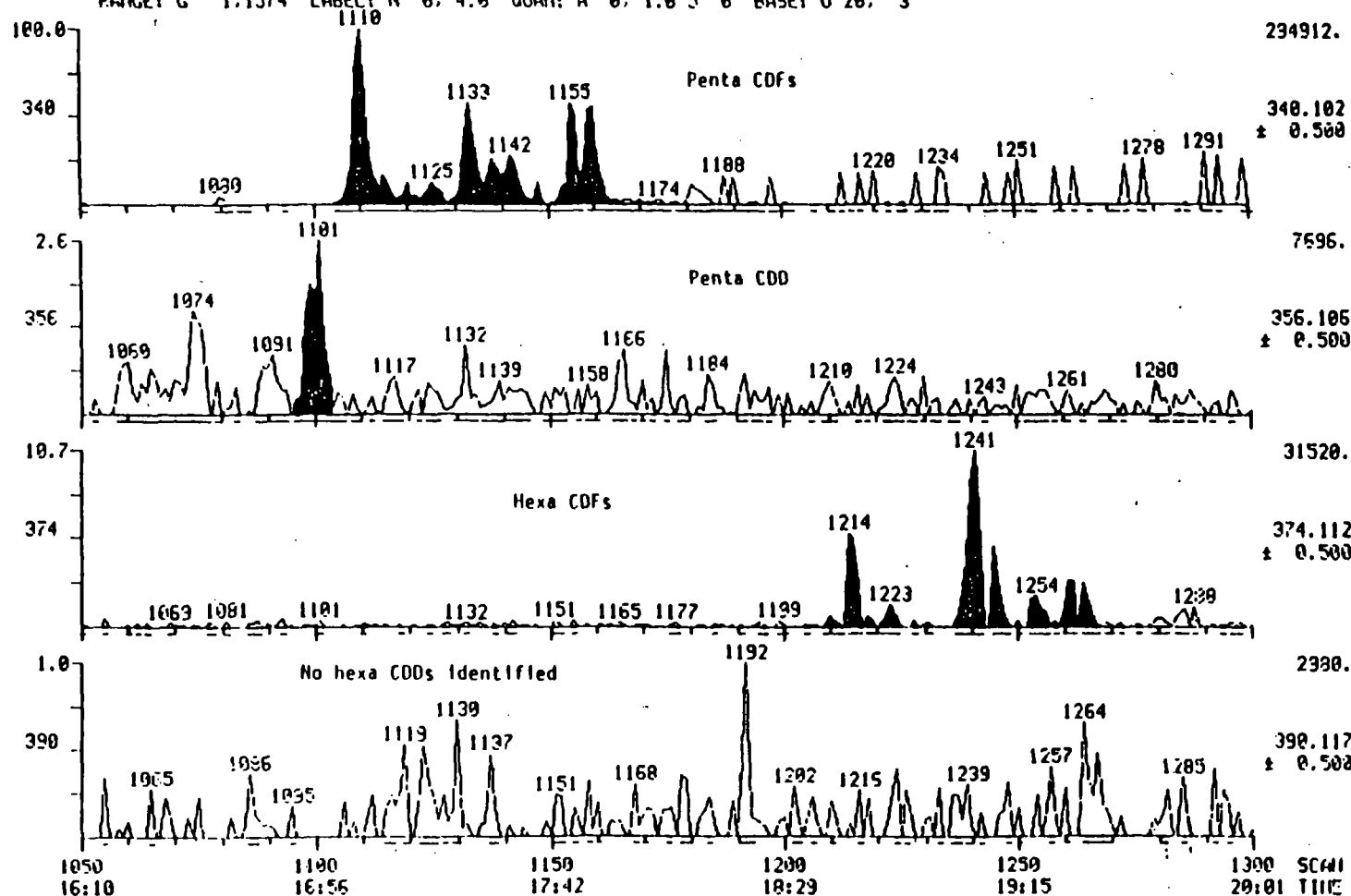
CONVS.: M3=3.5 70EV PC=3 OF5-15M 100-1H-325-10/ 45 SEC SPLTL

RANGE: G 1.1574 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

DATA: 8201112X4 #1

SCANS 1050 TO 1390

CAL: 110315112X1 #3



Penta and hexa CDFs and CDDs in sample 8-22-52-S500,  
silicone oil at 500 µg/g PCB.

MID MASS CHROMATOGRAMS

DATA: 8201115R5 M1

SCANS 850 TO 1040

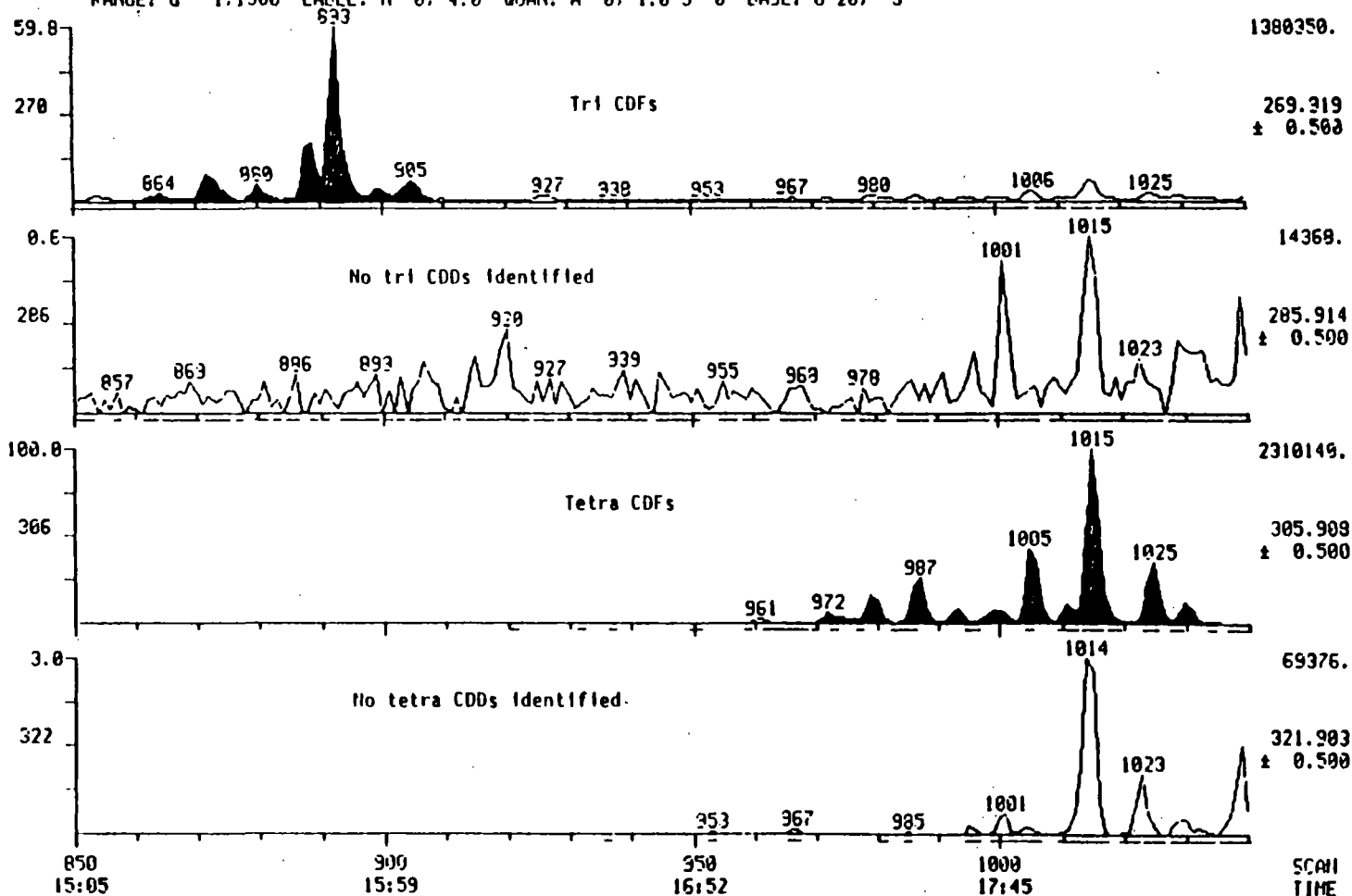
09/15/84 16:34:00

CAL1: 1110250H21 M2

SAMPLE: 8201A23 RUI 8-30-62 ASKL 1/100 DIL 1UL 11UJ

COND3.: -2200EMU 70EV 1MA 065-3PM 100 1H-320-10/

RANGE: G 1.1300 LABEL: H 0. 4.0 QUAN: A 0. 1.0 J 0 BASE: U 20, 3



Tri and tetra CDFs and CDDs in sample 8-30-62-ASKL,  
Askarel sample.

MID MASS CHROMATOGRAMS

09/15/84 0:35:00

SAMPLE: 8-30-62-ASKL 1/100DIL PCDD/F/8PH IUL CL5-6

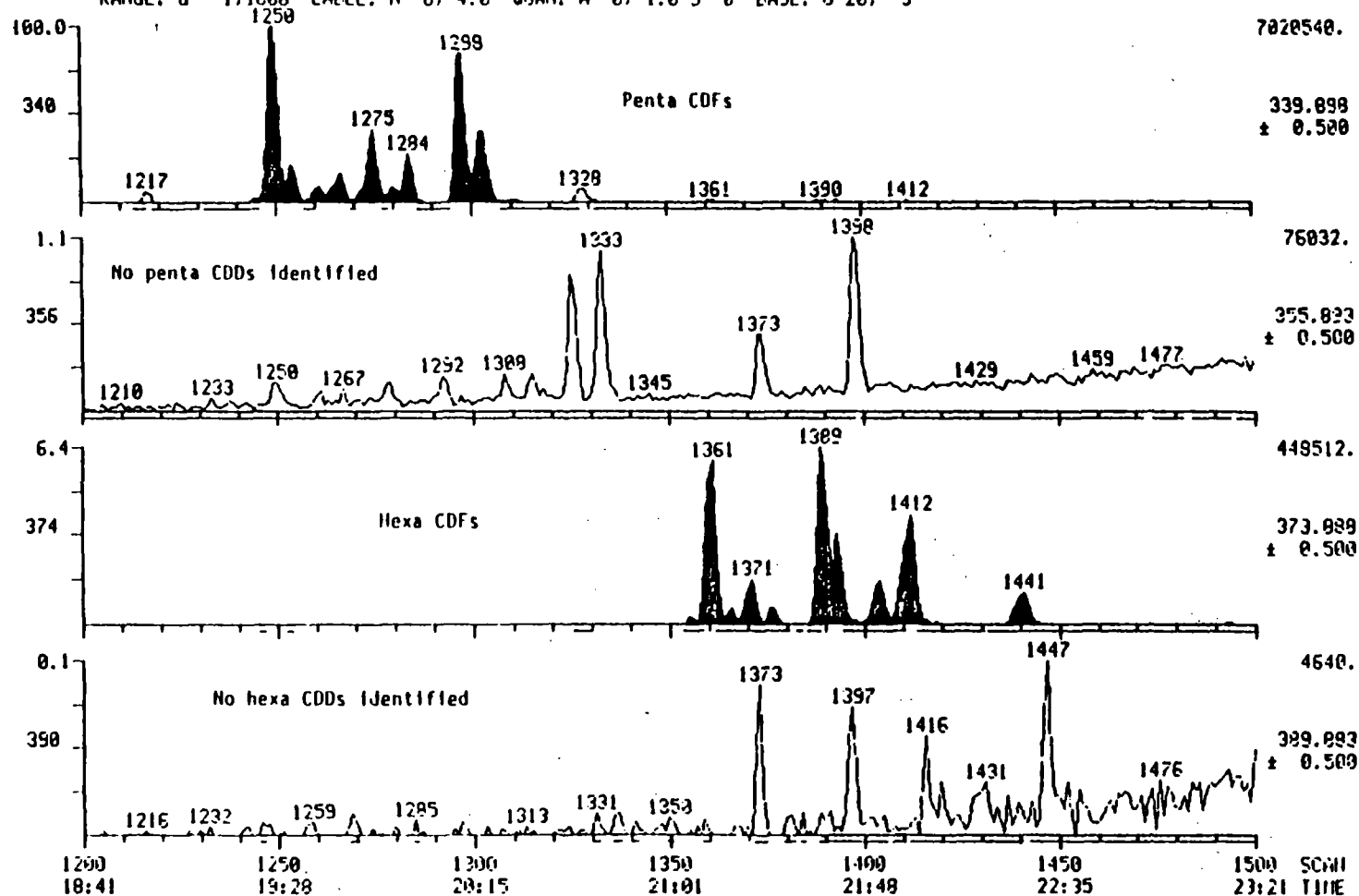
COMPS.: -220RENU 70EV INA DBS-30M 100-1H-320-10/

RANGE: G 1.1E00 LABEL: N 0, 4.0 QUANT: A 0, 1.0 J 0 BASE: U 20, 3

DATA: 820111SR1 #1

SCANS 1200 TO 1500

CAL: MID315114R1 #3



Penta and hexa CDFs and CDDs in sample 8-30-62-ASKL,  
Askarel sample.



MSD MASS CHROMATOGRAMS

DATA: 8201111R12 #1

SCANS 580 TO 850

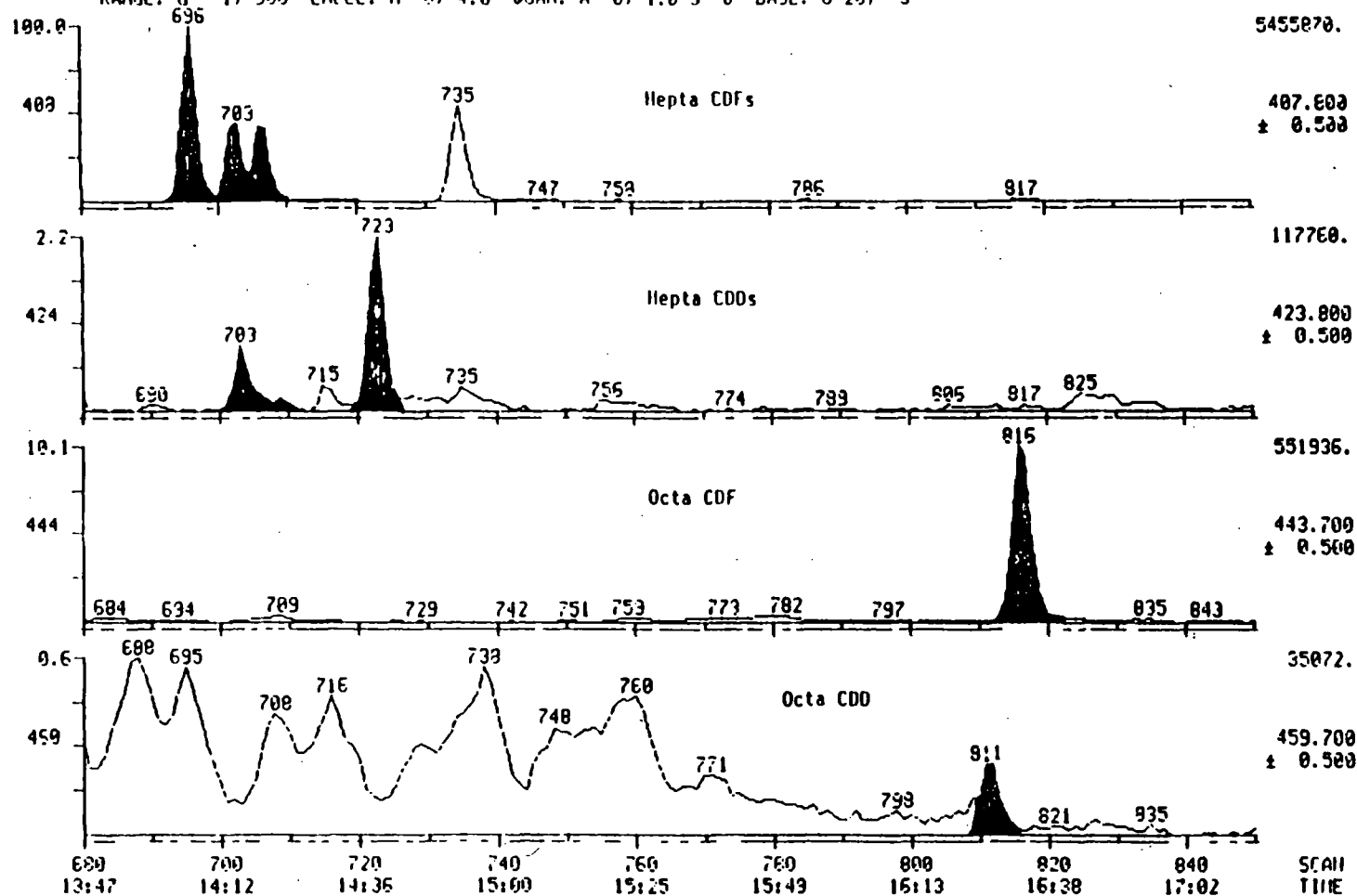
03-11-84 16:50:00

CAL1: MID390111 #2

SAMPLE: 8-30-62-ASKL (D/F) JUL PCDD/F/BPH CL7-8

COND5: -2000EMU 70EV 1MA 065-30M-200-2H-320-10/

RANGE: C 1, 990 LABEL: H 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3



Hepta and octa CDFs and CDDs in sample 8-30-62-ASKL,  
Askarel sample.

MID MASS CHROMATOGRAMS

DATA: 8201111X5 P1

SCANS 780 TO 1020

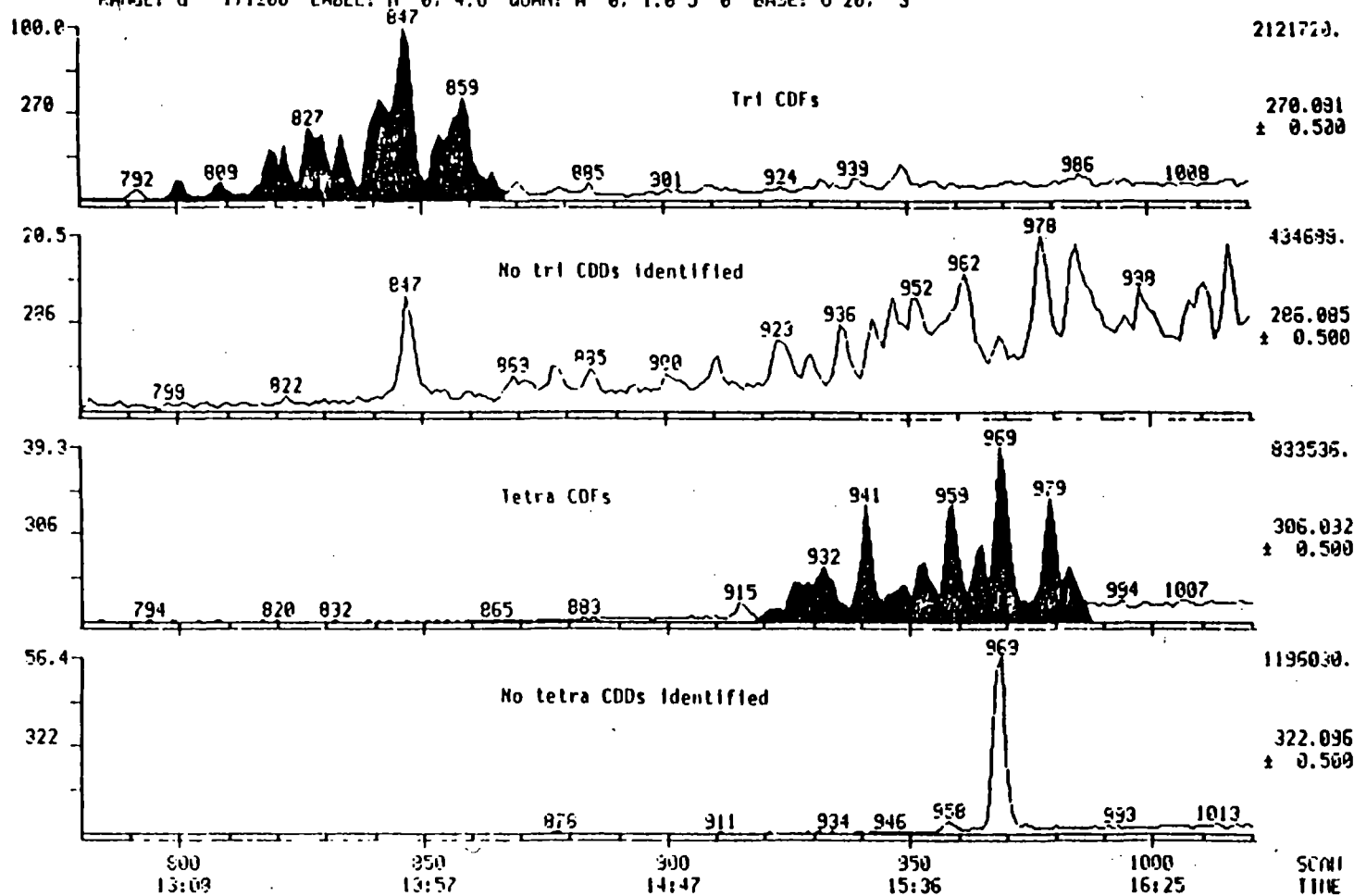
09/11/84 11:22:00

CALI: 1110250110M1 M3

SAMPLE: 8201A23 RUN 8-15-43-M5 10L INJ

COND5.: MG=3.5 70EV 8C=3 065-15M 100-1H-325-10/

RANGE: G 1.1200 LABEL: 11 0. 4.0 QUAN: A 0. 1.0 J 0 BASE: U 20. 3



Tri and tetra CDFs and CDDs in sample 8-15-43-M5,  
mineral oil at 5 µg/g PCB.

MIO MASS CHROMATOGRAMS

DATA: 820111X3 01

SCANS 750 TO 1050

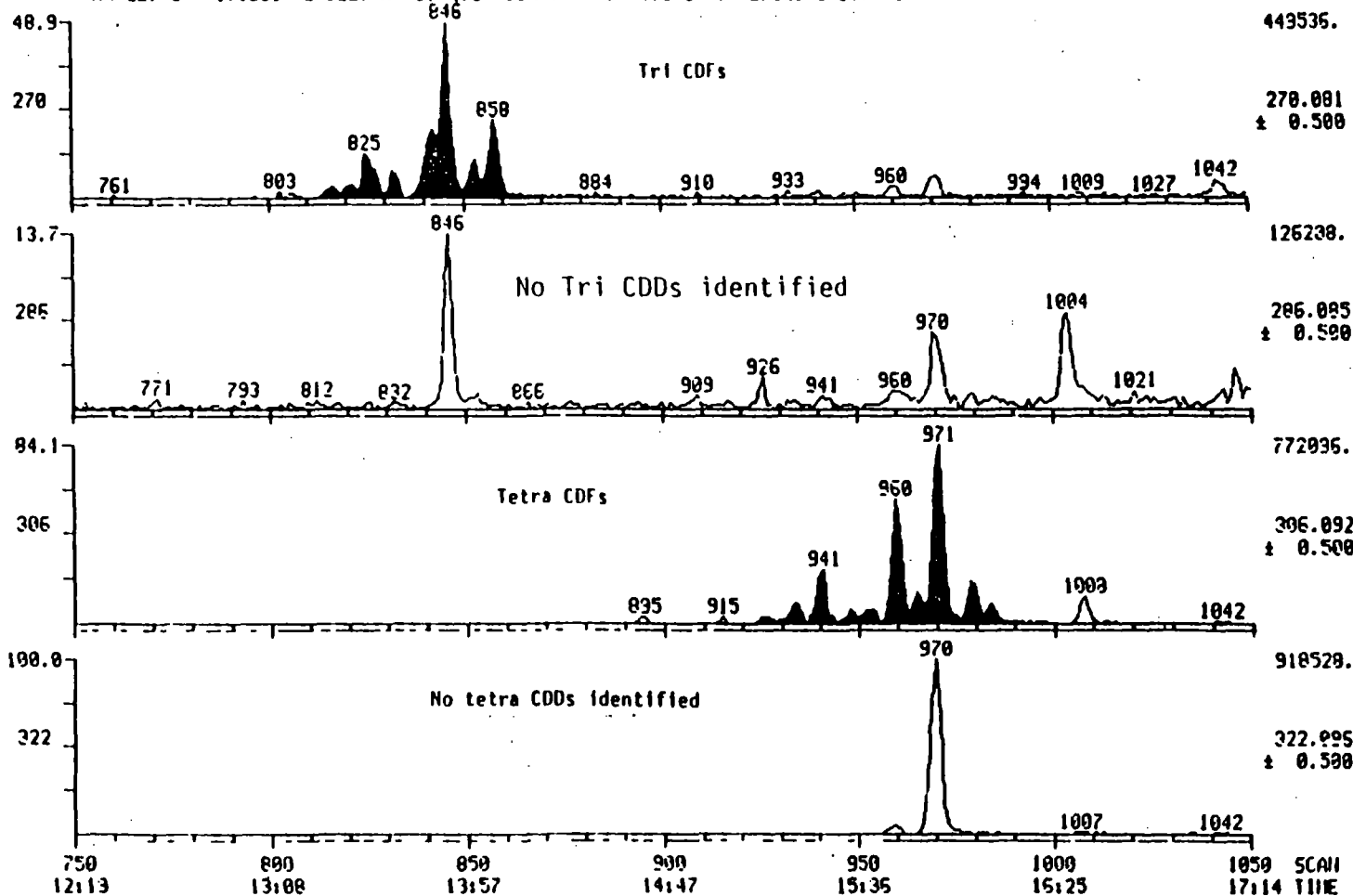
03/11/84 10:10:00

CAL: MID250110X1 03

SAMPLE: 8201A23 RUN 8-17-47 S5 XAD 1UL INJ

COND5.: MG=3.5 70EV 6C=3 065-15M 100-1H-325-10/

RANGE: G 1.1200 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3



Tri and tetra CDFs and CDDs in sample 8-17-47-S5,  
silicone oil at 5 µg/g PCB.

MID MASS CHROMATOGRAMS

03/10/84 15:45:00

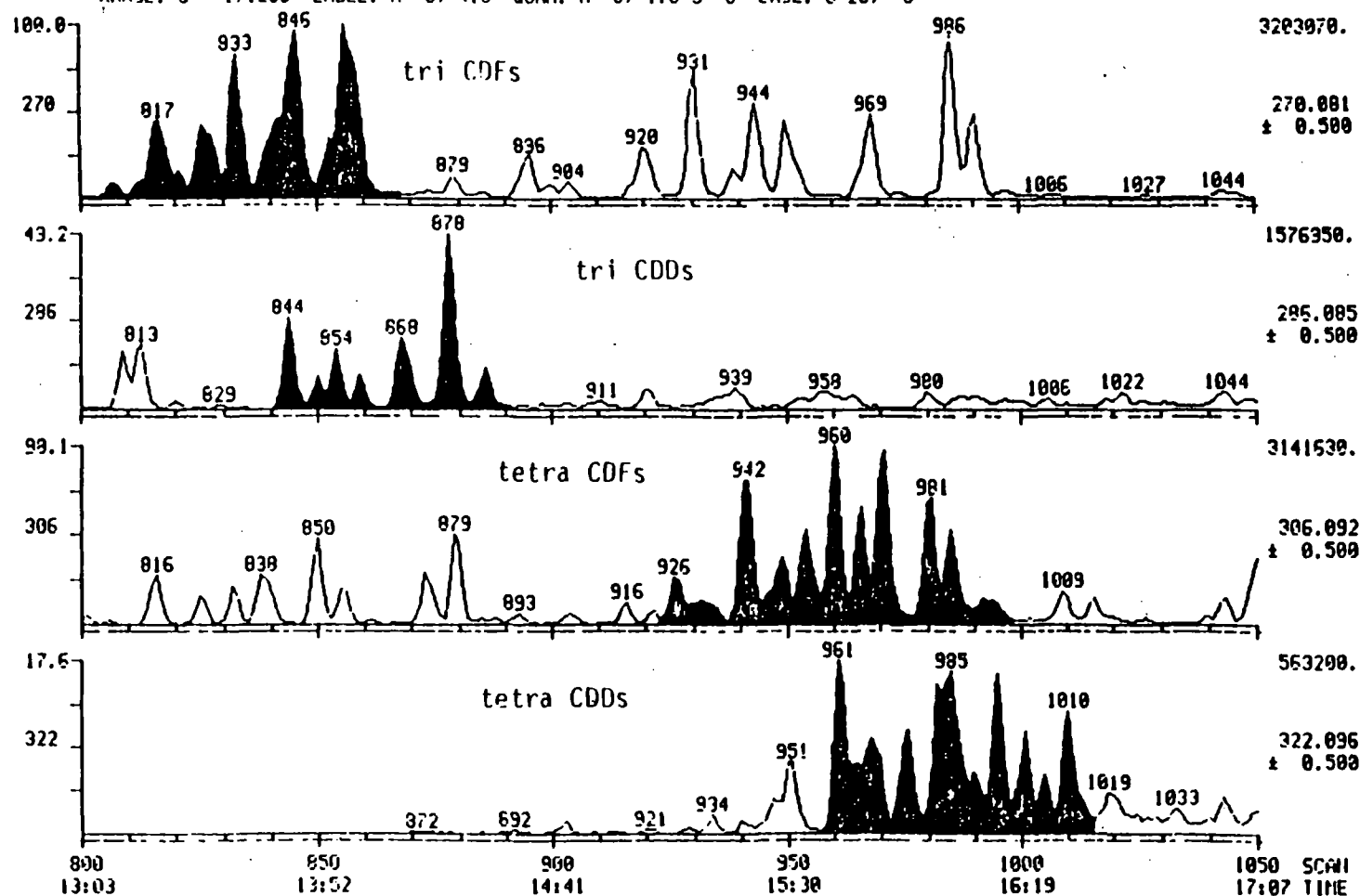
SAMPLE: 8201A23 RUN 8-28-57 IUL INJ

COND5.: MG=3.5 70EV BC=3 DBS-15M 100-1H-325-10/

RANGE: C 1.1250 LABEL: N 0. 4.0 QUAN: A 0. 1.0 J 0 BASE: U 20. 3

DATA: 8201110X2 #1  
CALI: MID250110X1 #3

SCANS 809 TO 1050



Tri and tetra CDFs and CDDs in sample 8-28-57-CLBZ,  
chlorobenzene sample.

**ATTACHMENT 3**

**PCDD/PCDF Residue Levels  
in  
Phenoxy Herbicides**

**A SUMMARY OF THE REPORTED ANALYTICAL LIMITS OF DETECTION FOR POLYCHLORODIBENZO-*p*-DIOXINS (PCDDs)  
AND POLYCHLORODIBENZOFURANS (PCDFs) IN PHENOXYALKANOIC ACID HERBICIDES\***

Reference	Applicability	Compounds detected	Limit of detection (mg kg <sup>-1</sup> of sample)
Storherr et al.	2,4,5-T	TCDD	0.05
Brenner et al. (1972)	2,4-D, dichlorprop, mecoprop, and 2,4,5-T	TCDD	0.01
Vogel (1976)	2,4,5-T	TCDD	0.03
Ramstad et al. (1977)	Butoxypropyl esters of 2,4,5-T and of fenoprop	TCDD	0.005
Elvidge (1971)	2,4,5-T, ethylhexyl ester of 2,4,5-T, and formulations containing the ethylhexyl ester of 2,4,5-T	TCDD	0.05 (on 2,4,5-T content)
Woolson et al. (1972)	2,4-D, 2,4-DB, dichlorprop, 2,4,5-T, fenoprop, and dicamba	Di-, tri-, tetra-, penta-, hexa-, hepta-, and octa-CDDs	0.5 (for any one of the compounds detected)
Edmunds et al. (1973)	2,4,5-T, the butyl and octyl esters of 2,4,5-T, and formulations of the butyl ester of 2,4,5-T (50% in mineral oil)	TCDD	0.05
Crummett and Stehl (1973)	Esters of 2,4,5-T and of fenoprop, Herbicide Orange	TCDD	0.05
Buser et al. (1974)	2,4,5-T, formulations of esters of 2,4,5-T and of amine salts of 2,4,5-T	TCDD	0.001 (on a standard solution)
Huckins et al. (1978)	Herbicide Orange	TCDD and penta-CDD	0.02
Rappe et al. (1978)	Formulations of esters of 2,4,5-T, Herbicide Orange	Di-, tri-, tetra-, penta-, and hexa-CDDs, di-, tri-, tetra-, penta-, and hexa-CDFs	0.01-0.05
Polzhofer (1979)	2,4,5-T, alkyl esters of 2,4,5-T and formulations	TCDD	0.005 (on 2,4,5-T content)
Carello et al.	Fenoprop	TCDD	0.01

\* Baker, P. G., R. H. Hoodless, and J. C. Tyler, *Pestic. Sci.*, 12, 297-304.

**ATTACHMENT 4**

**Literature and Patent Search  
for Applications of  
Aroclor 1268**

Monday, August 9, 1993  
ONLINE LITERATURE SEARCH FOR  
BRIAN BIRD -- AROCLOR 1268

PROJ/OVERHEAD #: 3387

L1 ANSWER 1 OF 1 COPYRIGHT 1993 ACS  
RN 11100-14-4 REGISTRY  
CN \*\*\*Aroclor 1268 (9CI)\*\*\* (CA INDEX NAME)  
MF Unspecified  
CI MAN  
LC BIOBUSINESS, BIOSIS, CA, CAPREVIEWS, CHEMLIST, CJACS, CSCHEM,  
IFICDB, IFIPAT, IFIUDB, RTECS

\*\*\* STRUCTURE DIAGRAM IS NOT AVAILABLE \*\*\*

1 REFERENCES IN FILE CAPREVIEWS  
57 REFERENCES IN FILE CA (1967 TO DATE)  
1 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA



FILE COVERS 1950 TO DATE.

FILE LAST UPDATED: 2 Aug 1993 (930802/ED) PATENT PUBLICATION: 27 Jul 1993

HIGHEST PATENT NUMBER: 5231699 - INDEXING IS AVAILABLE IN THE IFIUDB

FILE AND IS CURRENT THROUGH 30 MAR 1993 (930731/UP)

FILE 'IFIPAT' ENTERED AT 10:07:55 ON 09 AUG 93

L4 10 S L2

L4 ANSWER 1 OF 10

AN 1158491 IFIPAT;IFIUDB;IFICDB

TI FLAME RETARDANT, NON-DRIPPING COMPOSITIONS OF POLYPHENYLENE ETHER  
AND ACRYLONITRILE-BUTADIENE-STYRENE; HALOGENATED COMPOUND,  
POLYTETRAFLUOROETHYLENE

INF Haaf, William Robert, Voorhesville, NY

Reinhard, Donald Lewis, Lenox, MA

IN HAAF WILLIAM ROBERT; REINHARD DONALD LEWIS

PAF General Electric, Pittsfield, MA

PA GENERAL ELECTRIC CO (33808)

EXNAM Ziegler, J

AG Morgan, Finnegan, Pine, Foley & Lee

PI US 4107232 15 Aug 1978 (CITED IN 008 LATER PATENTS)

AI US 75-642981 22 Dec 1975

RLI US 74-530373 6 Dec 1974 DIVISION

FI US 4107232 15 Aug 1978

DT UTILITY

FS CHEMICAL

OS CA 90:55799

AB The present invention provides novel flame retardant, nondripping  
thermoplastic molding compositions of polyphenylene ether resins or  
acrylonitrile-butadiene-styrene copolymers which include a flame  
retardant agent, and polytetrafluoroethylene.

L4 ANSWER 2 OF 10

AN 1084847 IFIPAT;IFIUDB;IFICDB

I FLAME-RETARDANT RESIN COMPOSITIONS; COPPER CLAD LAMINATES,  
POLYHALOGENATED AROMATIC HYDROCARBONS, POLYPHENYLENE ETHERS, COATED  
ELECTRICAL DEVICES

INF Beacham, Harry Hoyt, Severna Park, MD

Wright, Carl Leonard, Pasadena, MD

IN BEACHAM HARRY HOYT; WRIGHT CARL LEONARD

PAF FMC Corporation, Philadelphia, PA

PA FMC CORP (31248)

EXNAM Lesmes, George F

EXNAM Thibodeau, P J

AG Fellows, Charles C

Ianno, Frank

PI US 4034136 5 Jul 1977 (CITED IN 010 LATER PATENTS)

AI US 75-575515 8 May 1975

RLI US 69-837911 30 Jun 1969 CONTINUATION-IN-PART ABANDONED

US 70-95418 4 Dec 1970 DIVISION 3936414

FI US 4034136 5 Jul 1977

US 3936414

DT UTILITY; REASSIGNED

FS CHEMICAL

OS CA 87:69143

AB This specification discloses that certain polychlorinated or  
polybrominated aromatic hydrocarbon compounds can be incorporated  
in thermosetting polymer compositions to make the thermosetting  
compositions flame retardant without injuring the desirable  
physical and electrical properties of the thermosetting  
compositions when in the thermoset state. These polyhalogenated  
aromatic hydrocarbons are apparently caused to react into the  
thermosetting composition, provided there is a minimum of 1 mol of  
a polyunsaturated monomer containing carbon to carbon unsaturation  
for each 3 mols of polychlorinated aromatic hydrocarbons. The  
thermosetting compositions generally contain a polyunsaturated  
monomer, a polyunsaturated polymer with carbon to carbon double  
bond unsaturation or a polyphenylene ether polymer which may be

used in place of some or all of the polyunsaturated polymer, and a polyhalogenated aromatic hydrocarbon, containing at least 50% by weight combined chlorine or bromine and whose molecular weight is greater than 200. These thermosetting compositions can be heat cured or thermoset by a free radical initiator at a temperature of at least 130° C. and can contain pigments, reinforcing fibers, release agents, coupling agents, and other incidental amounts of additives commonly used in thermosetting resin compositions.

L4 ANSWER 3 OF 10  
AN 0947133 IFIPAT;IFIUDB;IFICDB  
TI POLYAMIDE-IMIDES  
INF Brode, George Lewis, Somerville, NJ  
Kawakami, James Hajime, Piscataway, NJ  
IN BRODE GEORGE LEWIS; KAWAKAMI JAMES HAJIME  
PAF Union Carbide Corporation, New York, NY  
PA UNION CARBIDE CORP (87136)  
EXNAM Shaver, Paul F  
AG Crowe, B F  
PI US 3897497 29 Jul 1975 (CITED IN 003 LATER PATENTS)  
AI US 74-456805 1 Apr 1974  
RLI US 70-50968 29 Jun 1970 CONTINUATION-IN-PART ABANDONED  
US 71-182526 21 Sep 1971 DIVISION 3817921  
FI US 3897497 29 Jul 1975  
US 3817921  
DT UTILITY; REASSIGNED  
FS CHEMICAL  
OS CA 84:5655  
AB A series of polyamide-imides which are both tractable and soluble in polar solvents as well as stable at high temperatures has been prepared by the condensation polymerization of aromatic diamines or oligomers containing oxygen, sulfone, and optionally alkylidene linkages with trimellitic acid, trimellitic acid anhydride or trimellitic anhydride acid chloride.

L4 ANSWER 4 OF 10  
AN 0888552 IFIPAT;IFIUDB;IFICDB  
TI POLYARYLIMIDES; GLASS CLOTH PREPEGS, MOLDING, ADHESIVES  
INF Brode, George L, Somerville, NJ  
Kwiatkowski, George T, Greenbrook, NJ  
IN BRODE G; KWIATKOWSKI G  
PAF Union Carbide Corporation, New York, NY  
PA UNION CARBIDE CORP (87136)  
EXNAM Lee, Lester L  
AG Crowe, Bernard Francis  
PI US 3839287 1 Oct 1974 (CITED IN 014 LATER PATENTS)  
AI US 73-362287 21 May 1973  
RLI US 71-182527 21 Sep 1971 CONTINUATION-IN-PART ABANDONED  
FI US 3839287 1 Oct 1974  
DE 2246026  
FR 2153331  
GB 1408933  
DT UTILITY; REASSIGNED  
FS CHEMICAL  
OS CA 78:160446  
AB Arylimides have been prepared from oligomer diamines and polyamines containing halogenated phenylene, oxygen, alkylidene and/or sulfone moieties and maleic anhydride. The resultant arylimides, in the form of B-stage resins can be used for the preparation of glass cloth prepreps, molding materials and adhesives which can be thermally converted to the thermoset state.

L4 ANSWER 5 OF 10  
AN 0839576 IFIPAT;IFIUDB;IFICDB  
TI MICROBIOCIDAL NITROGEN DERIVATIVES OF HALOGENATED BIPHENYLS  
IN ADAMS P; MERIANOS J; PETROCCI A; SHAY E  
PA MILLMASTER ONYX CORP (55656)  
PI US 3790678 5 Feb 1974  
I US 71-111681 1 Feb 1971  
RLI US 69-883636 9 Dec 1969 DIVISION 3663620

FI US 3790678 5 Feb 1974  
US 3663620  
DT UTILITY; REASSIGNED  
FS CHEMICAL  
OS CA 80:129250  
AB A METHOD OF INHIBITING MICROORGANISMS WHICH COMPRISES APPLYING TO THE ENVIRONMENT IN WHICH SUCH MICROORGANISMS ARE FOUND AMMONIA AND AMINOSUBSTITUTED HALOGENATED BIPHENYLS, AS WELL AS THE NITROGEN DERIVATIVES THEREOF SUCH AS QUATERNARY AMMONIUM COMPOUNDS AMINE OXIDES, IMIDAZOLINES, AMIDES, ENAMINES, AMPHOLYTES, AND THE LIKE. THE AMMONIA AND AMINO HALOBIPHENYLS ARE PREPARED BY REACTING THE AMMONIA OR THE AMINE AND THE HALOBIPHENYL REACTANTS IN SUCH A MANNER THAT THE RATIO OF AMINE TO HALOBIPHENYL IS PREFERABLY LIMITED TO BETWEEN TWO AND FIVE MOLS OF AMINE TO EACH MOL OF HALOBIPHENYL.

L4 ANSWER 6 OF 10  
AN 0811931 IFIPAT;IFIUDB;IFICDB  
TI N-(PENTACHLOROBIPHENYL) ETHYLENE DIAMINE; MICROBIOCIDES  
IN ADAMS P; MERIANOS J; PETROCCI A; SHAY E  
PA MILLMASTER ONYX CORP (55656)  
PI US 3763238 2 Oct 1973  
AI US 72-234987 15 Mar 1972  
RLI US 69-883636 9 Dec 1969 DIVISION 3663620  
FI US 3763238 2 Oct 1973  
US 3663620  
DT UTILITY; REASSIGNED  
FS CHEMICAL  
OS CA 79:146165  
AB MICROBIOCIDAL AMMONIA AND AMINO SUBSTITUTED HALOGENATED BIPHENYLS, AS WELL AS THE NITROGEN DERIVATIVES THEREOF SUCH AS QUATERNARY AMMONIUM COMPOUNDS, AMINE OXIDES, IMIDAZOLINES, AMIDES, ENAMINES, AMPHOLYTES, AND THE LIKE. THE AMMONIA AND AMINO HALOBIPHENYLS ARE PREPARED BY REACTING THE AMMONIA OR THE AMINE AND THE HALOBIPHENYL REACTANTS IN SUCH A MANNER THAT THE RATIO OF AMINE TO HALOBIPHENYL IS PREFERABLY LIMITED TO BETWEEN TWO AND FIVE MOLS OF AMINE TO EACH MOL OF HALOBIPHENYL.

L4 ANSWER 7 OF 10  
AN 0808677 IFIPAT;IFIUDB;IFICDB  
TI N-(NONACHLOROBIPHENYL) AMINOPROPYL-N'-DIMETHYLAMINE; ANTISEPTICS, PRESERVATIVES, MICROBIOCIDES  
INF Adams, Phillip, Murray Hill, NJ  
Merianos, John J, Jersey City, NJ  
Petrocci, Alfonso N, Glen Rock, NJ  
Shay, Edward Griffin, Suffern, NY  
PAF Millmaster Onyx Corporation, New York, NY  
PA MILLMASTER ONYX CORP (55656)  
EXNAM Hines, Robert V  
AG Jacobs, Arthur A  
PI US 3759995 18 Sep 1973  
AI US 72-235064 15 Mar 1972  
RLI US 69-883636 9 Dec 1969 DIVISION 3663620  
FI US 3759995 18 Sep 1973  
US 3663620  
DT UTILITY; REASSIGNED  
FS CHEMICAL  
OS CA 79:126057  
AB Microbiocidal ammonia and amino substituted halogenated biphenyls, as well as the nitrogen derivatives thereof such as quaternary ammonium compounds, amine oxides, imidazoles, amides, enamines, ampholytes, and the like. The ammonia and amino halobiphenyls are prepared by reacting the ammonia or the amine and the halobiphenyl reactants in such a manner that the ratio of amine to halobiphenyl is preferably limited to between two and five mols of amine to each mol of halobiphenyl.

L4 ANSWER 8 OF 10  
N 0804103 IFIPAT;IFIUDB;IFICDB  
TI N-(PENTACHLOROBIPHENYL)-DIETHYLENE TRIAMINE; MICROBIOCIDES  
IN ADAMS P; MERIANOS J; PETROCCI A; SHAY E

PA MILLMASTER ONYX CORP (55656)  
PI US 3755448 28 Aug 1973 (CITED IN 001 LATER PATENTS)  
AI US 72-235092 15 Mar 1972  
RLI US 69-883636 9 Dec 1969 DIVISION 3663620  
FI US 3755448 28 Aug 1973  
US 3663620  
T UTILITY; REASSIGNED  
FS CHEMICAL  
OS CA 79:104918  
AB MICROBIOCIDAL AMMONIA AND AMINO SUBSTITUTED HALOGENATED BIPHENYLS,  
AS WELL AS THE NITROGEN DERIVATIVES THEREOF SUCH AS QUATERNARY  
AMMONIUM COMPOUNDS, AMINE OXIDES, IMIDAZOLINES, AMIDES, ENAMINES,  
AMPHOLYTES, AND THE LIKE. THE AMMONIA AND AMINO HALOBIPHENYLS ARE  
PREPARED BY REACTING THE AMMONIA OR THE AMINE AND THE HALOBIPHENYL  
REACTANTS IN SUCH A MANNER THAT THE RATIO OF AMINE TO HALOBIPHENYL  
IS PREFERABLY LIMITED TO BETWEEN TWO AND FIVE MOLS OF AMINE TO EACH  
MOL OF HALOBIPHENYL.

L4 ANSWER 9 OF 10  
AN 0781923 IFIPAT;IFIUDB;IFICDB  
TI MICROBIOCIDAL NITROGEN DERIVATIVES OF HALOGENATED BIPHENYLS  
IN ADAMS P; MERIANOS J; PETROCCI A; SHAY E  
PA MILLMASTER ONYX CORP (55656)  
PI US 3733421 15 May 1973 (CITED IN 003 LATER PATENTS)  
AI US 70-38513 18 May 1970  
RLI US 69-883636 9 Dec 1969 CONTINUATION-IN-PART 3663620  
FI US 3733421 15 May 1973  
US 3663620  
DT UTILITY; REASSIGNED  
FS CHEMICAL  
OS CA 79:42132  
AB MICROBIOCIDAL BIS-AMINO SUBSTITUTED HALOGENATED BIPHENYLS, AS WELL  
AS THE NITROGEN DERIVATIVES THEREOF SUCH AS QUATERNARY AMMONIUM  
COMPOUNDS, AMINE OXIDES, IMIDAZOLINES, AMIDES, ENAMINES,  
AMPHOLYTES, AND THE LIKE.

4 ANSWER 10 OF 10  
N 0711662 IFIPAT;IFIUDB;IFICDB  
TI N-(HALOGENATED BIPHENYL)-DIETHYLENE TRIAMINES  
IN ADAMS PHILLIP; MERIANOS JOHN J; PETROCCI ALFONSO N; SHAY EDWARD  
GRIFFIN  
PA MILLMASTER ONYX CORP (55656)  
PI US 3663620 16 May 1972 (CITED IN 007 LATER PATENTS)  
AI US 69-883636 9 Dec 1969  
FI US 3663620 16 May 1972  
DT UTILITY; REASSIGNED  
FS CHEMICAL  
OS CA 77:48033  
AB MICROBIOCIDAL AMMONIA AND AMINO SUBSTITUTED HALOGENATED BISPHENYLS,  
AS WELL AS THE NITROGEN DERIVATIVES THEREOF SUCH AS QUATERNARY  
AMMONIUM COMPOUNDS, AMINE OXIDES, IMIDAZOLINES, AMIDES, ENAMINES,  
AMPHOLYTES, AND THE LIKE. THE AMMONIA AND AMINO HALOBIPHENYLS ARE  
PREPARED BY REACTING THE AMMONIA OR THE AMINE AND THE HALOBIPHENYL  
REACTANTS IN SUCH A MANNER THAT THE RATIO OF AMINE TO HALOBIPHENYL  
IS PREFERABLY LIMITED TO BETWEEN TWO AND FIVE MOLS OF AMINE TO EACH  
MOL OF HALOBIPHENYL.

L1 57 S 11100-14-4  
L2 5 S L1 AND (USE OR USES OR APPLICATION!)  
L3 6 S L1 AND (USE OR USES OR APPLICATION!)/AB  
L4 6 S L3 NOT L2

## L2 ANSWER 1 OF 5 COPYRIGHT 1993 ACS

CA118(8):65794m On the \*\*\*use\*\*\* of tree leaves as bioindicators of the contamination of air by organochlorines in France. Granier, Laurent; Chevreuil, Marc (Inst. Hydrol. Climatol., Univ. Pierre et Maris Curie, Paris 75252, Fr.). Water, Air, Soil Pollut., 64(3-4), 575-84 (Eng) 1992. CODEN: WAPLAC. ISSN: 0049-6979.

AB The possibility of using leaves of plane trees (*Platanus vulgaris*) as bioindicators of air pollution by organochlorines was studied in France. Inter- and intraspecific variations by a factor of 3 in the concns. of polychlorinated biphenyls were obsd. This type of biomonitoring appears appear to be capable of discerning, on a continental scale, variations of a factor 10 in atm. concns. Air/leaf bioconc. factors are .apprx.105 for PCBs and 104 for lindane.

## L2 ANSWER 2 OF 5 COPYRIGHT 1993 ACS

CA109(16):134468m Degradation and dehalogenation of polychlorobiphenyls and halogenated aromatic molecules by superoxide ion and by electrolytic reduction. Sugimoto, Hiroshi; Matsumoto, Shigenobu; Sawyer, Donald T. (Dep. Chem., Texas A and M Univ., College Station, TX 77843, USA). Environ. Sci. Technol., 22(10), 1182-6 (Eng) 1988. CODEN: ESTHAG. ISSN: 0013-936X. OTHER SOURCES: CJACS.

AB Polyhalogenated arom. hydrocarbons (e.g., PCB's and hexachlorobenzene) are rapidly degraded by O<sub>2</sub><sup>-</sup> in DMF to give CO<sub>3</sub><sup>2-</sup> and halide ions. The efficient destruction of such materials is accomplished via the in-situ electrolytic redn. of dissolved O to generate O<sub>2</sub><sup>-</sup>, which reacts with polyhalo aroms. by nucleophilic substitution. The reaction stoichiometries were detd. by cyclic voltammetric measurements, and the reactant/produced profiles were assayed by capillary gas chromatog. and potentiometric titrns. Complete destruction by O<sub>2</sub><sup>-</sup> occurs for PCB's that contain .gtoreq.3 Cl atoms per arom. ring. Another means to the dehalogenation of halo arom. hydrocarbons is their electrolytic redn. to the parent hydrocarbon in O-free DMF solns. Electrochem. studies confirm that all PCB's can be dehalogenated via anaerobic electrolysis.

## L2 ANSWER 3 OF 5 COPYRIGHT 1993 ACS

CA107(12):108523q The \*\*\*use\*\*\* of simplex optimization in evaluating complex chromatograms of mixtures. Tomas, X.; Sabate, L. G. (Dep. Chemometrics, Inst. Quim. Sarria, Barcelona 08017, Spain). Anal. Chim. Acta, 191, 439-43 (Eng) 1986. CODEN: ACACAM. ISSN: 0003-2670.

AB The application of simplex optimization to the qual. and quant. evaluation of chromatograms of complex mixts. is outlined. The chromatogram is considered as a linear combination of pure component chromatograms (assuming no interactions are present); if suitable components are selected and the coeffs. of the linear combinations are properly evaluated, the exptl. chromatogram can be reproduced numerically. The simplex method is then used to evaluate the soln. with minimal error. The quantitation of polychlorobiphenyls from Aroclor mixts. in marine sediments and fish, and the identification of essential oils in a perfume base ext. are outlined. Errors of .ltoreq.10% (.25% in environmental samples) between the exptl. and reconstructed chromatograms were achieved.

## L2 ANSWER 4 OF 5 COPYRIGHT 1993 ACS

CA90(8):55799f Flame retardant, nondripping compositions of polyphenylene ether and acrylonitrile-butadiene-styrene. Haaf, William Robert; Reinhard, Donald Lewis (General Electric Co., USA). U.S. US 4107232 15 Aug 1978, 6 pp. (Eng). CODEN: USXXAM. CLASS: IC: C03L051-00. NCL: 260876000R. APPLICATION: US 74-530373 6 Dec 1974.

B The addn. of PTFE [9002-84-0] to a poly(2,6-dimethyl-1,4-phenylene) ether (I) [24938-67-8] compn. or a ABS polymer [9003-56-9] compn., each contg. a fireproofing agent, increased to the non-dripping

flame-resistant properties. Thus, a compn. contg. 1 20, SBR rubber 80, polyethylene [9002-88-4] 1.5, tetrabromobisphenol A [79-94-7] 8, triphenyl phosphate [115-86-6] 1, Sb2O3 3, and PTFE 0.25 part was molded into self-extinguishing non-dripping sheets cf. to self-extinguishing but dripping sheets when PTFE was omitted.

L2 ANSWER 5 OF 5 COPYRIGHT 1993 ACS

CA89(16):134737s Separation of chlorinated dibenzo-p-dioxins from chlorinated congeners. Dolphin, R. J.; Willmott, F. W. (Philips Res. Lab., Redhill/Surrey, Engl.). J. Chromatogr., 149, 161-8 (Eng) 1978. CODEN: JOCRAM. ISSN: 0021-9673.

AB Chlorinated dibenzo-p-dioxins, e.g. 2,3,7,8-tetrachlorodibenzo-p-dioxin [1746-01-6] and octachlorodibenzo-p-dioxin [3268-87-9], were sepd. from polychlorinated biphenyls, polychlorinated naphthalenes, polychlorinated dibenzofurans, and organochlorine pesticides by high-performance liq.-chromatog. on microparticulate Al2O3 column.

L4 ANSWER 1 OF 6 COPYRIGHT 1993 ACS

CA117(6):61928q Barycentric coordinates and the orientation of the classical mixture surface. Rayens, William S. (Dep. Stat., Univ. Kentucky, Lexington, KY 40506, USA). J. Math. Chem., 9(2), 147-60 (Eng) 1992. CODEN: JMCHEG. ISSN: 0259-9791.~Q

AB The signs of the barycentric coordinates of a point exterior to a nondegenerate k-simplex in IR<sup>p</sup> contain useful information about how that point is positioned relative to the vertices of that simplex. This relationship is certainly not newly obsd., with some of the first ideas dating back to Moebius in 1827. However, this article presents some new geometrical results which further quantify the relationship and focuses on applying these new results to help solve the problem of finding the point on a simplex that is closest to a given exterior point. In particular, it is shown that the signs of the barycentrics can be used to immediately identify a potentially large set of facets that could not contain this closest point. Such results have immediate \*\*\*applications\*\*\* to the problem of identifying the components in a chem. linear mixt. Real polychlorobiphenyl mixts. are employed to illustrate the new ideas.

L4 ANSWER 2 OF 6 COPYRIGHT 1993 ACS

CA115(14):141483q Evidence of an unusual pattern of polychlorinated biphenyls in the serum of some residents and canines in Paoli, Pennsylvania. Burse, Virlyn W.; Groce, Donald F.; Korver, Margaret P.; Caudill, Samuel P.; McClure, Patricia C.; Lapeza, Chester R., Jr.; Head, Susan L.; Schilling, Rebecca J.; Farrar, Jeffrey A.; et al. (Cent. Environ. Health Injury Control, Cent. Dis. Control, Atlanta, GA 30333, USA). J. - Assoc. Off. Anal. Chem., 74(4), 577-86 (Eng) 1991. CODEN: JANCA2. ISSN: 0004-5756. OTHER SOURCES: CJAOC.

AB The present study \*\*\*uses\*\*\* gas chromatog. with electron capture detection with packed and capillary columns to detect polychlorinated biphenyls (PCBs) in serum samples from people living near the elec. car repair and maintenance facility of the Southeastern Pennsylvania Transit Authority in Paoli, Pennsylvania. Most of the cohort surveyed had serum patterns similar to patterns for Aroclor 1260 (AR 1260); a small portion (3 of 89) had patterns indicative of an AR with higher chlorination (e.g., AR 1268). In addn. to analyzing serum samples from humans, serum samples from canines (pet of some of the subjects) were analyzed. In general, the serum pattern for canines was less descriptive for AR 1260 than the pattern for humans; however, the pattern for several canines (9 of 16) was that of the higher chlorinated PCBs (e.g., AR 1268). By using mass spectrometry and capillary column gas chromatog., the presence of high-mol.-wt. polychlorinated congeners in both human and animal samples was confirmed. A statistically significant relationship was not shown between serum patterns of PCBs in canines and their owners or between canines and certain behavioral traits (e.g., runs free, retrieves, hours outside, hours inside). However, the correlation between PCBs quantified as AR 1266 and canines' residence time was statistically significant.

CA115(10):105183h Determination of congeners of polychlorinated biphenyls in reference materials. Anderson, Jerry W. (Robert S. Kerr Environ. Res. Lab., NSI Technol. Serv., Inc., Ada, OK 74820, USA). J. High Resolut. Chromatogr., 14(6), 369-72 (Eng) 1991. CODEN: JHRCE7. ISSN: 0935-6304.

AB Aroclor ref. materials from the United States Environmental Protection Agency repository are proposed for \*\*\*use\*\*\* as secondary stds. for the congener-specific anal. for polychlorinated biphenyls in environmental samples. Representative samples of the materials have been analyzed using a gas chromatog. equipped with photoionization and electron capture detectors. The elution pattern of the congeners was detd. for a DB-17 capillary column.

CA114(20):191848n Speciation and quantitation of Aroclors in hazardous wastes based on PCB congener data. Draper, William M.; Wijekoon, Donald; Stephens, Robert D. (Hazard. Mater. Lab., State of California Dep. Health Serv., Berkeley, CA 94704, USA). Chemosphere, 22(1-2), 147-63 (Eng) 1991. CODEN: CMSHAF. ISSN: 0045-6535.

AB Selected chlorobiphenyl congener data were used to accurately est. the Aroclor content of hazardous wastes. Only 12 chlorobiphenyl congeners are detd., nominally, IUPAC Nos. 15, 18, 31, 87, 105, 110, 118, 170, 180, 183, 203, and 206, and from these marker compds. Aroclors 1016, 1254, 1260 and 1268 are estd. The remaining regulated Aroclor mixts. also are measured by this technique making it suitable for enforcement of existing California and US regulations. An addnl. 34 chlorobiphenyl congeners are routinely analyzed allowing detn. of PCBs in highly weathered and/or treated wastes. Included are the major constituents of the com. Aroclors and the predominant congeners in the environment. The sum of PCB congener concns., s-PCB, is generally .apprx.70% of the total Aroclor content. The \*\*\*use\*\*\* of capillary column sepns. and data redn. procedures described has a no. of advantages over packed column GC procedures: (1) data interpretation does not require analyst judgement and can be automated; (2) the technique makes full \*\*\*use\*\*\* of the sepn. power of capillary columns to minimize interference by pesticides and other sample components; and (3) the technique accurately measures PCBs in samples with >1 Aroclor. Application of this technique to the detn. of Aroclors in auto shredder waste is described and results compared to a packed-column procedure, U.S. EPA method 8080.

CA103(8):58345c Optimization of perchlorination conditions for some representative polychlorinated biphenyls. Lin, Jia Ming; Hee, Shane S. Que (Med. Cent., Univ. Cincinnati, Cincinnati, OH 45267-0056, USA). Anal. Chem., 57(11), 2130-4 (Eng) 1985. CODEN: ANCHAM. ISSN: 0003-2700. OTHER SOURCES: CJACS.

AB The yield of decachlorobiphenyl (I) [2051-24-3] from representative Aroclors (50 to 6000 ng) using SbCl<sub>5</sub> as perchlorinating agent was extremely temp.-dependent at <236 for a reaction period of 2 h. At 288.degree., a 35-min reaction period was sufficient to obtain I yields of >80% for Aroclor 1016 [12674-11-2], 1242 [53469-21-9], 1254 [11097-69-1], and 1268 [11100-14-4]. The perchlorination process was shown to be 1st-order. The temp. dependence of the reaction times below 236.degree. was largely responsible for the inconsistent perchlorination yields reported previously in the literature. The extn. of I with hexane from a HCl-acidified perchlorinated soln., and the subsequent column chromatog. on silica gel were also essential after the perchlorination to quantitate the I by 63Ni electron capture/gas chromatog. The structures of the 2 nonachlorobiphenyls which were the penultimate stable intermediates before I, were found. The methodol. to \*\*\*use\*\*\* the technique for air and blood samples was described.

CA83(19):158697f Occurrence of polychlorinated biphenyls in humans. Price, Harold A.; Welch, Robert L. (Michigan Dep. Public Health Pestic., Lansing, Mich., USA). Environ. Health Perspect., 1, 73-8 (Eng) 1972. CODEN: EVHPAZ.

AB Anal. of human adipose tissue, collected through the human monitoring survey program (Division of Pesticide Community Studies, E. P. A.) revealed that 41-5% of the general population have levels of .gtoreq.1.0 ppm (wet wt.) polychlorinated biphenyls, with isomers from Aroclors 1254 [11097-69-1], 1260 [11096-82-5], 1262 [37324-23-5], and 1268 [11100-14-4]. The presence of polychlorinated biphenyls ranging from pentachlorobiphenyls to decachlorobiphenyls was confirmed in selected samples by the \*\*\*use\*\*\* of combined gas chromatog.-mass spectrometry. The possible interference of these compds. with the anal. of organochlorine pesticide residues is discussed.



3/4/1 (Item 1 from file: 653)

01255385 DIALOG File 653: U.S. Patents-Full Text (1980-89)

Utility

FLAME RETARDANT, 'NON-DRIPPING COMPOSITIONS OF POLYPHENYLENE ETHER AND ACRYLONITRILE-BUTADIENE-STYRENE

PATENT NO.: 4,355,126

ISSUED: October 19, 1982 (19821019)

INVENTOR(s): Haaf, William R., Voorhesville, NY (New York), US (United States of America)  
Reinhard, Donald L., Lenox, MA (Massachusetts), US (United States of America)

ASSIGNEE(s): General Electric Company, (A U.S. Company or Corporation), Pittsfield, MA (Massachusetts), US (United States of America)

APPL. NO.: 6-15,103

FILED: February 26, 1979 (19790226)

This is a continuation of application Ser. No. 530,373 filed Dec. 6, 1974, abandoned.

FULL TEXT: 585 lines

ABSTRACT

The present invention provides novel flame retardant, non-dripping thermoplastic molding compositions of polyphenylene ether resins or acrylonitrile-butadiene-styrene copolymers which include a flame retardant agent, and polytetrafluoroethylene.

...sup 1

lene HBB sup 1

TPP sup 2

AO sup 3

PTFE sup 4

Aroclor 1268 sup 5

Times (sec...)

3/4/2

(Item 1 from file: 652)

01043890 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility

FLAME-RETARDANT RESIN COMPOSITIONS

PATENT NO.: 4,159,371

ISSUED: June 26, 1979 (19790626)

INVENTOR(s): Wright, Carl L., Pasadena, MD (Maryland), US (United States of America)

Beacham, Harry H., Severna Park, MD (Maryland), US (United States of America)

ASSIGNEE(s): FMC Corporation, (A U.S. Company or Corporation), Philadelphia, PA (Pennsylvania), US (United States of America)

APPL. NO.: 5-783,533

FILED: April 01, 1977 (19770401)

CROSS REFERENCE TO RELATED APPLICATION

This is a division, of application Ser. No. 575,515, filed May 8, 1975, and now U.S. Pat. No. 4,034,436 which is a division of application Ser. No. 95,418, now U.S. Pat. No. 3,936,414, filed Dec. 4, 1970, which is a continuation-in-part of Ser. No. 837,911, filed June 30, 1969, now abandoned.

FULL TEXT: 1575 lines

ABSTRACT

This specification discloses that certain polychlorinated or polybrominated aromatic hydrocarbon compounds can be incorporated in thermosetting polymer

compositions to make the thermosetting compositions flame retardant without injuring the desirable physical and electrical properties of the thermosetting compositions when in the thermoset state. These polyhalogenated aromatic hydrocarbons are apparently caused to reset into the thermosetting composition, provided there is a minimum of 1 mol of a polyunsaturated monomer containing carbon to carbon unsaturation for each 3 ols of polychlorinated aromatic hydrocarbons. The thermosetting compositions generally contain a polyunsaturated monomer, a polyunsaturated polymer with carbon to carbon double bond unsaturation or a polyphenylene ether polymer which may be used in place of some or all of the polyunsaturated polymer, and a polyhalogenated aromatic hydrocarbon, containing at least 50% by weight combined chlorine or bromine and whose molecular weight is greater than 200. These thermosetting compositions can be heat cured or thermoset by a free radical initiator at a temperature of at least 130 degree(s) C., and can contain pigments, reinforcing fibers, release agents, coupling agents, and other incidental amounts of additives commonly used in thermosetting resin compositions.

...5460)

50 45 40 60 65 60 -- -- 40 74 87 78

Chlorinated biphenyl,  
68% chlorine (Aroclor 1268 )

-- -- -- -- -- -- 62 62 -- -- -- --

Diallyl phthalate

-- -- -- -- -- 40 -- 38 -- -- -- --

Diallyl isophthalate

50 55 60 40 35 -- 38 -- -- -- --

Diallyl

3/4/3

(Item 2 from file: 652)

00988079 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility

FLAME RETARDANT, NON-DRIPPING COMPOSITIONS OF POLYPHENYLENE ETHER AND ACRYLONITRILE-BUTADIENE-STYRENE

ATENT NO.: 4,107,232

ISSUED: August 15, 1978 (19780815)

INVENTOR(s): Haaf, William Robert, Voorhesville, NY (New York), US (United States of America)  
Reinhard, Donald Lewis, Lenox, MA (Massachusetts), US (United States of America)

ASSIGNEE(s): General Electric, (A U.S. Company or Corporation), Pittsfield, MA (Massachusetts), US (United States of America)

APPL. NO.: 5-642,981

FILED: December 22, 1975 (19751222)

This is a division, of application Ser. No. 530,373 filed Dec. 6, 1974.

FULL TEXT: 541 lines

#### ABSTRACT

The present invention provides novel flame retardant, non-dripping thermoplastic molding compositions of polyphenylene ether resins or acrylonitrile-butadiene-styrene copolymers which include a flame retardant agent, and polytetrafluoroethylene.

...sup 1

lene HBB sup 1

TPP sup 2

AO sup 3

PTFE sup 4

Aroclor 1268 sup 5

Times...

3/4/4

(Item 3 from file: 652)

00910282 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility

FLAME-RETARDANT RESIN COMPOSITIONS

PATENT NO.: 4,034,136  
 ISSUED: July 05, 1977 (19770705)  
 INVENTOR(s): Wright, Carl Leonard, Pasadena, MD (Maryland), US (United States of America)  
 Beacham, Harry Hoyt, Severna Park, MD (Maryland), US (United States of America)  
 ASSIGNEE(s): FMC Corporation, (A U.S. Company or Corporation), Philadelphia, PA (Pennsylvania), US (United States of America)  
 APPL. NO.: 5-575,515  
 FILED: May 08, 1975 (19750508)

This is a division, of application Ser. No. 95,418, now U.S. Pat. No. 3,936,414, filed Dec. 4, 1970, which is a continuation-in-part of Ser. No. 837,911, filed June 30, 1969, now abandoned.

FULL TEXT: 1691 lines

ABSTRACT

This specification discloses that certain polychlorinated or polybrominated aromatic hydrocarbon compounds can be incorporated in thermosetting polymer compositions to make the thermosetting compositions flame retardant without injuring the desirable physical and electrical properties of the thermosetting compositions when in the thermoset state. These polyhalogenated aromatic hydrocarbons are apparently caused to react into the thermosetting composition, provided there is a minimum of 1 mol of a polyunsaturated monomer containing carbon to carbon unsaturation for each 3 mols of polychlorinated aromatic hydrocarbons. The thermosetting compositions generally contain a polyunsaturated monomer, a polyunsaturated polymer with carbon to carbon double bond unsaturation or a polyphenylene ether polymer which may be used in place of some or all of the polyunsaturated polymer, and a polyhalogenated aromatic hydrocarbon, containing at least 50% by weight combined chlorine or bromine and whose molecular weight is greater than 200. These thermosetting compositions can be heat cured or thermoset by a free radical initiator at a temperature of at least 130 degree(s) C. and can contain pigments, reinforcing fibers, release agents, coupling agents, and other incidental amounts of additives commonly used in thermosetting resin compositions.

...5460)

50 45 40 60 65 60 -- -- 40 74 87 78

Chlorinated biphenyl,  
 68% chlorine (Aroclor 1268)

-- -- -- -- -- 62 62 -- -- -- --

Diallyl phthalate

-- -- -- -- -- 40 -- 38 -- -- -- --

Diallyl isophthalate

50 55 60 40 35 -- 38 -- -- -- --

Diallyl

3/4/5

(Item 4 from file: 652)

00861778 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility

POLYAMIDE-IMIDES

PATENT NO.: 3,988,374  
 ISSUED: October 26, 1976 (19761026)  
 INVENTOR(s): Brode, George L., Piscataway, NJ (New Jersey), US (United States of America)  
 Kawakami, James H., Piscataway, NJ (New Jersey), US (United States of America)  
 ASSIGNEE(s): Union Carbide Corporation, (A U.S. Company or Corporation), New York, NY (New York), US (United States of America)  
 PPL. NO.: 5-182,526  
 FILED: September 21, 1971 (19710921)  
 FULL TEXT: 993 lines

# ABSTRACT

A series of polyamide-imides which are both tractable and soluble in polar solvents as well as stable at high temperatures has been prepared by the condensation polymerization of aromatic diamines or oligomers containing oxygen, sulfone, and optionally alkylidene linkages with trimellitic acid, trimellitic acid anhydride or trimellitic anhydride acid chloride.

...was removed until the pot temperature reached 135 degree(s) C.

A concentrated solution of Aroclor 1268 (453 grams, 1.0 mole) in 500 ml. of hot toluene was added via an...

3/4/6 (Item 5 from file: 652)  
00819147 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
SEALING ADHESIVE STRANDS AND COMPOSITIONS THEREFOR

PATENT NO.: 3,945,975  
ISSUED: March 23, 1976 (19760323)  
INVENTOR(s): Strack, Donald Robert, Dayton, OH (Ohio), US (United States of America)  
ASSIGNEE(s): Protective Treatments, Inc, (A U.S. Company or Corporation), Dayton, OH (Ohio), US (United States of America)  
APPL. NO.: 5-101,084  
FILED: December 23, 1970 (19701223)

This application is a continuation-in-part of my applications Ser. No. 551,878, now abandoned, filed May 23, 1966 for GLAZING COMPOSITIONS; Ser. No. 611,129 filed Jan. 23 1967, for MOUNTING UNIT EMPLOYING DUAL GASKET CONSTRUCTION now U.S. Pat. No. 3,478,475; Ser. No. 607,022 filed Jan. 3, 1967 for SELF SUPPORTING, NON-LOAD BEARING RESILIENT TAPE SEALANT now U.S. Pat. No. 3,500,603, and Ser. No. 738,548, now abandoned, filed June 20, 1968 for ADHESIVE SEALING GLAZING COMPOSITION, and the disclosures of such applications are incorporated herein by reference.  
FULL TEXT: 741 lines

## ABSTRACT

New sealing adhesive bed compositions for glazing are form retaining and comprise elastomeric material initially mixed with plasticizers of low volatility and finely divided solids which either are of fibrous form or produce thixotropic mixtures with the plasticizers. A ratio range of from 3/4 to 21/2 parts non-volatile plasticizers to one part elastomeric material, and from 0.3 to 21/2 parts such finely divided solids to one part elastomeric material is present. Other kinds of finely divided solids and other ingredients may be present. The glazing strand is formed by extrusion, and the formed strand is not cured after extrusion. The tapes permanently deform under pressure when the glass is installed, have low spring back and exhibit little or no creep or cold flow in service. ... the elastomer art, such as polychlorinated polyphenyls known under the commercial names "Aroclor 1254" and "Aroclor 1268" and the ester plasticizers for example, dioctyl phthalate, dioctyl sebacate, butyl oleate, and liquid wood...

3/4/7 (Item 6 from file: 652)  
00812483 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
VEHICLE LAMP CONSTRUCTION

PATENT NO.: 3,939,337  
ISSUED: February 17, 1976 (19760217)

INVENTOR(s): Oda, Roy, Westland, MI (Michigan), US (United States of America)  
De Frayne, Garry O., Pontiac, MI (Michigan), US (United States of America)  
Walker, Paul L., Royal Oak, MI (Michigan), US (United States of America)  
ASSIGNEE(s): Chrysler Corporation, (A U.S. Company or Corporation),  
Highland Park, MI (Michigan), US (United States of America)  
APPL. NO.: 5-422,808  
FILED: December 07, 1973 (19731207)  
FULL TEXT: 256 lines

#### ABSTRACT

A lamp assembly wherein the lens portion is secured to the lamp housing by means of an electromeric tape. The bonding tape replaces the gasket, gasket cement and retaining screws conventionally employed in current lamp assemblies.

...polymer.

Examples of other suitable plasticizers are certain polychlorinated polyphenyls known commercially as "Aroclor" 1254, Aroclor 1268, low molecular weight polyisobutylene ("Vistanex" LM-MS), medium viscosity propylene polymer (Polypropene C-175 of...

... tackifiers in the compositions. Examples of suitable tackifiers are chlorinated biphenyls illustrated by Aroclor 1254, Aroclor 1268 (which Aroclors also function as plasticizers) and Aroclor 1260, non-reactive polymethylol phenol resin (commercially...

3/4/8 (Item 7 from file: 652)  
00809550 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility

LAME-RETARDANT RESIN COMPOSITIONS

PATENT NO.: 3,936,414  
ISSUED: February 03, 1976 (19760203)  
INVENTOR(s): Wright, Carl Leonard, Pasadena, MD (Maryland), US (United States of America)  
Beacham, Harry Hoyt, Severna Park, MD (Maryland), US (United States of America)  
ASSIGNEE(s): FMC Corporation, (A U.S. Company or Corporation), Philadelphia, PA (Pennsylvania), US (United States of America)  
APPL. NO.: 5-95,418  
FILED: December 04, 1970 (19701204)

#### CROSS REFERENCE TO RELATED APPLICATION

This application is a continuation-in-part of U.S. patent application Ser. No. 837,911, filed June 30, 1969, entitled "Flame-Retardant Resin Compositions", now abandoned.

FULL TEXT: 1564 lines

#### ABSTRACT

This specification discloses that certain polychlorinated or polybrominated aromatic hydrocarbon compounds can be incorporated in thermosetting polymer compositions to make the thermosetting compositions flame retardant without injuring the desirable physical and electrical properties of the thermosetting compositions when in the thermoset state. These polyhalogenated aromatic hydrocarbons are apparently caused to react into the thermosetting composition, provided there is a minimum of 1 mol of a polyunsaturated monomer containing carbon to carbon unsaturation for each 3 mols of polychlorinated aromatic hydrocarbons. The thermosetting compositions generally contain a polyunsaturated monomer, a polyunsaturated polymer with carbon to carbon double bond unsaturation or a polyphenylene

ether polymer which may be used in place of some or all of the polyunsaturated polymer, and a polyhalogenated aromatic hydrocarbon, containing at least 50% by weight combined chlorine or bromine and whose molecular weight is greater than 200. These thermosetting compositions can be heat cured or thermoset by a free radical initiator at a temperature of at least 130 degree(s) C, and can contain pigments, reinforcing fibers, release agents, coupling agents, and other incidental amounts of additives commonly used in thermosetting resin compositions.

...5460)

	50	45	40	60	65	60	--	--	40	74	87	78
Chlorinated biphenyl, 68% chlorine ( Aroclor 1268 )	--	--	--	--	--	--	62	62	--	--	--	--
Diallyl phthalate	--	--	--	--	--	40	--	38	--	--	--	--
Diallyl isophthalate	50	55	60	40	35	--	38	--	--	--	--	--
Diallyl												

3/4/9 (Item 8 from file: 652)  
00772113 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
POLYAMIDE-IMIDES

PATENT NO.: 3,897,497  
ISSUED: July 29, 1975 (19750729)  
INVENTOR(s): Brode, George Lewis, Somerville, NJ (New Jersey), US (United States of America)  
Kawakami, James Hajime, Piscataway, NJ (New Jersey), US  
(United States of America)  
ASSIGNEE(s): Union Carbide Corporation, (A U.S. Company or Corporation),  
New York, NY (New York), US (United States of America)  
APPL. NO.: 5-456,805  
FILED: April 01, 1974 (19740401)

This is a division of Ser. No. 182,526 filed Sept. 21, 1971, now U.S. Pat. No. 3,817,921, which is in turn a continuation-in-part of Ser. No. 50,968 filed June 29, 1970, now abandoned.

FULL TEXT: 998 lines

#### ABSTRACT

A series of polyamide-imides which are both tractable and soluble in polar solvents as well as stable at high temperatures has been prepared by the condensation polymerization of aromatic diamines or oligomers containing oxygen, sulfone, and optionally alkylidene linkages with trimellitic acid, trimellitic acid anhydride or trimellitic anhydride acid chloride.

...was removed until the pot temperature reached 135 degree(s) C.

A concentrated solution of Aroclor 1268 (453 grams, 1.0 mole) in 500 ml. of hot toluene was added via an...

3/4/10 (Item 9 from file: 652)  
00770539 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
METHOD OF INHIBITING ALGAE EMPLOYING NITROGEN DERIVATIVES OF HALOGENATED BIPHENYLS

PATENT NO.: 3,895,932  
ISSUED: July 22, 1975 (19750722)

INVENTOR(s): Merianos, John J., Jersey City, NJ (New Jersey), US (United States of America)  
Shay, Edward Griffin, Suffern, NY (New York), US (United States of America)  
Adams, Phillip, Murray Hill, NJ (New Jersey), US (United States of America)  
Petrocci, Alfonso N., Glen Rock, NJ (New Jersey), US (United States of America)  
ASSIGNEE(s): Millmaster Onyx Corporation, (A U.S. Company or Corporation), New York, NY (New York), US (United States of America)  
APPL. NO.: 5-349,630  
FILED: April 09, 1973 (19730409)

This is a continuation-in-part of application Ser. No. 38,513 filed May 18, 1970, now issued as U.S. Pat. No. 3,733,421 dated May 15, 1973 said application being itself a continuation-in-part of application Ser. No. 883,636, filed Dec. 9, 1969 and issued as U.S. Pat. No. 3,663,620 on May 16, 1972.

FULL TEXT: 193 lines

#### ABSTRACT

Microbiocidal bis-amino substituted halogenated biphenyls, especially adapted for use as algacides.

...range of polychlorinated biphenyls, e.g. "Aroclor 1260" (said to contain 60% of chlorine) and "Aroclor 1268" (said to contain 68% of chlorine). Others with other chlorine content, especially "Aroclors" 1232, 1242...

3/4/11 (Item 10 from file: 652)  
00769666 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
POLYAMIDE-IMIDES

PATENT NO.: 3,895,064  
ISSUED: July 15, 1975 (19750715)  
INVENTOR(s): Brode, George Lewis, Somerville, NJ (New Jersey), US (United States of America)  
Kawakami, James Hajime, Piscataway, NJ (New Jersey), US (United States of America)  
ASSIGNEE(s): Union Carbide Corporation, (A U.S. Company or Corporation), New York, NY (New York), US (United States of America)  
APPL. NO.: 5-456,836  
FILED: April 01, 1974 (19740401)

This is a division on Ser. No. 182,526 filed Sept. 21, 1971, now U.S. Patent 3,817,921, which is in turn a continuation-in-part of Ser. No. 50,968 filed June 29, 1970, now abandoned.

FULL TEXT: 1013 lines

#### ABSTRACT

A series of polyamide-imides which are both tractable and soluble in polar solvents as well as stable at high temperatures has been prepared by the condensation polymerization of aromatic diamines or oligomers containing oxygen, sulfone, and optionally alkylidene linkages with trimellitic acid, trimellitic acid anhydride or trimellitic anhydride acid chloride.

...was removed until the pot temperature reached 135 degree(s) C.

A concentrated solution of Aroclor 1268 (453 grams, 1.0 mole) in 500 ml. of hot toluene was added via an...

3/4/12 (Item 11 from file: 652)  
00716262 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
POLYARYLIMIDES

PATENT NO.: 3,839,287  
ISSUED: October 01, 1974 (19741001)  
INVENTOR(s): Kwiatkowski, George T., Greenbrook, NJ (New Jersey), US  
(United States of America)  
Brode, George L., Somerville, NJ (New Jersey), US (United  
States of America)  
ASSIGNEE(s): Union Carbide Corporation, (A U.S. Company or Corporation),  
New York, NY (New York), US (United States of America)  
APPL. NO.: 5-362,287  
FILED: May 21, 1973 (19730521)  
This is a continuation-in-part of Ser. No. 182,527 filed Sept. 21, 1971 now  
abandoned.

FULL TEXT: 655 lines

ABSTRACT

Arylimides have been prepared from oligomer diamines and polyamines containing halogenated phenylene, oxygen, alkylidene and/or sulfone moieties and maleic anhydride. The resultant arylimides, in the form of B-stage resins can be used for the preparation of glass cloth prepreps, molding materials and adhesives which can be thermally converted to the thermoset state.

...was removed until the pot temperature reached 135 degree(s) C.

A concentrated solution of Aroclor 1268 (453 grams, 1.0 mole) in 500 ml. of hot toluene was added via an...

3/4/13 (Item 12 from file: 652)  
00689466 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
FLAME-RESISTANT RESISTOR COATINGS

PATENT NO.: 3,804,669  
ISSUED: April 16, 1974 (19740416)  
INVENTOR(s): Bockstie, Jr. Lawrence G., Bradford, PA (Pennsylvania), US  
(United States of America)  
ASSIGNEE(s): Corning Glass Works, (A U.S. Company or Corporation), Corning,  
NY (New York), US (United States of America)  
APPL. NO.: 5-186,787  
FILED: October 05, 1971 (19711005)

This is a division of application Ser. No. 652,412, filed July 11, 1967.

FULL TEXT: 233 lines

ABSTRACT

Flame resistant polyimide resistory coatings containing chlorinated polyphenyl and antimony trioxide.

... from 18 to 66 percent chlorine. Useful chlorinated polyphenyl resins include Aroclor 1260, Aroclor 1262, Aroclor 1268, Aroclor 1270, Aroclor 4465, Aroclor 5442, Aroclor 2565, and Aroclor 5460, the most preferred being...

3/4/14 (Item 13 from file: 652)  
00652180 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
N-(NONACHLOROBIPHENYL)AMINOPROPYL-N'-DIMETHYLAMINE



PATENT NO.: 3,759,995  
ISSUED: September 18, 1973 (19730918)  
INVENTOR(s): Merianos, John J., Jersey City, NJ (New Jersey), US (United States of America)  
Shay, Edward Griffin, Suffern, NY (New York), US (United States of America)  
Adams, Phillip, Murray Hill, NJ (New Jersey), US (United States of America)  
Petrocci, Alfonso N., Glen Rock, NJ (New Jersey), US (United States of America)  
ASSIGNEE(s): Millmaster Onyx Corporation, (A U.S. Company or Corporation), New York, NY (New York), US (United States of America)  
APPL. NO.: 5-235,064  
FILED: March 15, 1972 (19720315)

This is a division of co-pending application Ser. No. 883,636, filed Dec. 9, 1969 and now U.S. Pat. No. 3,663,620.  
FULL TEXT: 448 lines

#### ABSTRACT

Microbiocidal ammonia and amino substituted halogenated biphenyls, as well as the nitrogen derivatives thereof such as quaternary ammonium compounds, amine oxides, imidazolines, amides, enamines, ampholytes, and the like.

The ammonia and amino halobiphenyls are prepared by reacting the ammonia or the amine and the halobiphenyl reactants in such a manner that the ratio of amine to halobiphenyl is preferably limited to between two and five mols of amine to each mol of halobiphenyl.

...range of polychlorinated biphenyls, e.g. "Aroclor 1260" (said to contain 60% of chlorine) and "Aroclor 1268" (said to contain 68% of chlorine). Others with other chlorine content, especially "Aroclors" 1232, 1242... triamine.

#### EXAMPLE 2

In the apparatus of Example 1, 500 grams (or one mol) of "Aroclor 1268" (assaying 70% Cl) and 310 grams (or three mols) of diethylene triamine were reacted in...

3/4/15 (Item 14 from file: 652)  
00555594 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
COATED ABRASIVE BELT OVERLAP JOINT

PATENT NO.: 3,637,359  
ISSUED: January 25, 1972 (19720125)  
INVENTOR(s): Malloy, John F., Waterford, NY (New York), US (United States of America)  
Seiler, Charles J., Elnora, NY (New York), US (United States of America)  
ASSIGNEE(s): Norton Company, Troy, NY (New York), US (United States of America)  
APPL. NO.: 5-  
FILED: January 06, 1970 (19700106)

#### CROSS-REFERENCE TO RELATED APPLICATIONS

This application is a continuation-in-part of our application, Ser. No. 514,360 filed Dec. 16, 1965, for Coated Abrasive Belt Joining, now abandoned.

FULL TEXT: 663 lines

#### ABSTRACT

[54] FLAME-RETARDANT RESIN  
COMPOSITIONS

[75] Inventors: Carl Leonard Wright, Pasadena;  
Harry Hoyt Beacham, Severna Park,  
both of Md.

[73] Assignee: FMC Corporation, Philadelphia, Pa.

[22] Filed: Dec. 4, 1970

[21] Appl. No.: 95,418

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 837,911, June 30,  
1969, abandoned.

[52] U.S. CL..... 260/40 R; 106/15 FP; 260/42.18;  
260/869; 260/874; 260/DIG. 24

[51] Int. CL<sup>1</sup>..... C08L 71/04

[58] Field of Search..... 260/869, 874, 37 R, 42.18,  
260/861; 106/15 FP

[56] References Cited

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Schmidt, *Trans. J. Plastics Inst.*, Dec., 1965, pp.  
247-249.

Primary Examiner—Donald E. Czaja  
Assistant Examiner—S. M. Person

[57] ABSTRACT

This specification discloses that certain polychlorinated or polybrominated aromatic hydrocarbon compounds can be incorporated in thermosetting polymer compositions to make the thermosetting compositions flame retardant without injuring the desirable physical and electrical properties of the thermosetting compositions when in the thermoset state. These polyhalogenated aromatic hydrocarbons are apparently caused to react into the thermosetting composition, provided there is a minimum of 1 mol of a polyunsaturated monomer containing carbon to carbon unsaturation for each 3 mols of polychlorinated aromatic hydrocarbons. The thermosetting compositions generally contain a polyunsaturated monomer, a polyunsaturated polymer with carbon to carbon double bond unsaturation or a polyphenylene ether polymer which may be used in place of some or all of the polyunsaturated polymer, and a polyhalogenated aromatic hydrocarbon, containing at least 50% by weight combined chlorine or bromine and whose molecular weight is greater than 200. These thermosetting compositions can be heat cured or thermoset by a free radical initiator at a temperature of at least 130°C, and can contain pigments, reinforcing fibers, release agents, coupling agents, and other incidental amounts of additives commonly used in thermosetting resin compositions.

7 Claims, No Drawings

## FLAME-RETARDANT RESIN COMPOSITIONS

### CROSS REFERENCE TO RELATED APPLICATION

This application is a continuation-in-part of U.S. patent application Ser. No. 837,911, filed June 30, 1969, entitled "Flame-Retardant Resin Compositions", now abandoned.

### BACKGROUND OF THE INVENTION

#### A. Field of the Invention

This invention relates to thermosetting polymerizable compositions and particularly to flame-retardant thermosetting molding compositions.

#### B. Description of the of the Prior Art

Thermosetting polymerizable compositions, commonly termed thermosetting resins, are widely used in making castings, laminates, and molded articles. The high strength to weight ratios of thermoset resins when properly reinforced and cured and the resistance of these compositions to environmental conditions have suggested the application of thermoset compositions in numerous applications, including many applications where combustibility is a serious consideration.

A number of methods have been employed to reduce the rate of combustion of thermoset resin compositions. These methods have included the use of numerous flame-retardant additives, including inorganic additives, organic additives, a combination of inorganic and organic additives, the addition of a flame-retardant element that reacts and becomes part of the thermoset resin, and combinations of all these methods.

The use of inorganic fillers to impart flame retardance is attractive on a cost basis. Typical inert flame-retardant fillers retard the burning rate to the extent that they act as diluents of the combustible components. However, large amounts of fillers adversely affect the electrical properties of thermoset resin compositions.

Organic compounds containing halogen and phosphorus atoms such as chlorinated paraffins, chlorinated biphenyls, chlorinated aryl substituted alkyls, phosphorus containing hydrocarbons, and the like have been used for many years to impart flame resistance to plastic compositions including thermoset resins. The chlorinated materials are quite effective as flame retardants. The flame-retarding mechanism is believed to involve the release of hydrogen chloride at combustion temperatures which snuff out the flame by exclusion of oxygen. Only highly chlorinated organic materials have been considered, since the objective is to provide maximum flame resistance with a minimum of additive material. These highly chlorinated organics, including those which are solid, have a plasticizing action or thermoset compositions. Generally the plasticizing action of the chlorinated organic compounds lowers the heat deflection temperature and otherwise impairs the desirable properties of cured thermosetting resins.

Combinations of chlorinated organic additives with antimony trioxide have been known for a number of years. The effect of certain other metal oxides, such as magnesia or hydrated alumina, combined with antimony oxides in flameproofing chlorinated polymers, was noted by D. C. Thompson et al. in an article entitled "Flame Resistance of Neoprene" appearing in the August 1968 issue of *Rubber Age*. Combinations of hydrated alumina with antimony oxide and chlorinated organics are widely recognized as effective flame retar-

dants for resinous compositions including thermoset resin compositions.

Satisfactory flame resistance in thermoset polymers has been obtained by using reactive chlorinated intermediates which become a part of the cured thermoset system, thus avoiding the plasticizing effect experienced when using chlorinated paraffins, chlorinated polyphenyls and the like. An important advance in the chemistry of self-extinguishing or flame-retardant thermoset resins occurred as a result of the discovery of a commercial method of making chlorendic acid or anhydride in 1952. Chlorendic acid and anhydride have been esterified to form polyesters similar to those obtained when using phthalic anhydride. Additionally, chlorendic acid and anhydride have been used to prepare reactive monomers, such as diallyl chlorendate, which are useful in flameproofing thermosetting resins such as polyesters and diallylic phthalates without substantially reducing the desirable physical properties of the thermoset resin compositions.

The addition of a reactive flame-retardant additive to thermoset resins such as polyesters and diallylic phthalate resins is highly desirable, as this method of flameproofing offers flexibility in resin production, since it is possible to flameproof general-purpose resins rather than to make special flame-retardant grade resins. The chlorendic acid-based reactive flame-retardant materials are expensive and since 1952 no efficient and inexpensive reactive flame retardants for thermoset resins have been widely accepted. The principal object of this invention is the preparation of highly flame-retardant, inexpensive, thermosetting resin compositions.

### SUMMARY OF THE INVENTION

We have now discovered that certain polychlorinated and polybrominated aromatic hydrocarbon compounds can be incorporated in thermosetting polymer compositions to make the compositions flame retardant without injuring the desirable physical and electrical properties of the thermosetting compositions when in the thermoset state. We have found that this result is achieved by adding at least 1 mol of a polyunsaturated monomer with carbon to carbon unsaturation to the thermosetting composition for each 3 mols of polychlorinated or polybrominated aromatic hydrocarbon added to the thermosetting composition.

These thermosetting compositions generally comprise: (a) 100 parts by weight of the blend of about 10 to about 70 parts of a polyunsaturated monomer with carbon to carbon unsaturation, with about 90 to about 30 parts of a polymer of the class consisting of a polyunsaturated polymer with carbon to carbon unsaturation and polyphenylene ether polymers, and (b) about 5 to 50 parts by weight per 100 parts of a polychlorinated or polybrominated aromatic hydrocarbon containing at least 50% by weight of combined chlorine or bromine and whose molecular weight is greater than 200, in an amount not in excess of 3 mols of polyhalogenated aromatic hydrocarbon for each mol of the polyunsaturated monomer in a. These novel thermosetting compositions can also include pigments, fillers, reinforcing fibers, release agents, coupling agents and other incidental additives in amounts commonly used in thermosetting resin compositions.

We have also discovered that where high monomer levels cannot be tolerated, as in high-pressure molding compounds, it is possible to prereact the polychlorinated or polybrominated compound with mono-

mer prior to adding the polyhalogenated aromatic hydrocarbon in the thermosetting composition. The polyhalogenated aromatic compound is prereacted with the monomer by heating 3 to 2 mols of the polyhalogenated aromatic hydrocarbon with 2 to 3 mols of the polyunsaturated monomer containing carbon to carbon unsaturation at a temperature of at least 130°C in the presence of a free radical initiator for a period of time sufficient to initiate an exothermic reaction between the polyhalogenated aromatic compound and the monomer, generally about 2 to about 10 minutes. These reaction products can be added to thermosetting compositions and thermoplastic compounds as flame-retardant additives.

Another phase of our invention is the discovery that certain of our novel compositions adhere strongly to a wide variety of substrates including metals. The compositions of this invention which are useful as thermosetting coatings generally comprise: (a) 10 to 50 parts by weight of a polyunsaturated monomer containing carbon to carbon unsaturation, (b) 5 to 30 parts by weight of a polychlorinated or polybrominated aromatic hydrocarbon containing at least 50% by weight of chlorine or bromine and having a molecular weight of at least 200, the ratio of monomer to polyhalogenated aromatic hydrocarbon being at least 1 to 3 respectively, (c) 20 to 60 parts by weight of a polyphenylene ether polymer, and (d) 0 to 30 parts by weight of a polyunsaturated polymer containing carbon to carbon unsaturation. Because it is not necessary to dissolve the polyphenylene ether polymer portion of these coating compositions, the compositions may be dispersed in a liquid for ease of application, thus making it possible to prepare very high solids coating compositions containing as much as 70% solids. The compositions are useful for coating articles to seal them against moisture, as coating compositions for impregnating glass fabric and the like for making laminates, and because they adhere well to metal, they are useful in adhering metal to metal and metal to plastics such as in preparing a copper clad glass laminate.

All of our novel thermosetting compositions can be thermoset by free radical initiators at temperatures of at least 130°C for a period of time sufficient to initiate an exothermic reaction during which the polychlorinated or polybrominated aromatic compound reacts into the other components of the compositions and loses its thermoplastic or plasticizing properties.

#### DESCRIPTION OF THE INVENTION AND THE PREFERRED EMBODIMENTS

The polychlorinated or polybrominated aromatic hydrocarbons containing at least 50% by weight combined chlorine and whose molecular weight is greater than 200, useful in practicing this invention, include, but are not limited to, compounds such as chlorinated and brominated biphenyls and terphenyls, trichlorobenzene and higher chlorinated benzenes and alkyl substituted benzenes, chlorinated naphthalenes, and the like.

Polyunsaturated polymers useful in practicing this invention include, but are not limited to, diallylic phthalate prepolymers and polyester resins. Diallylic phthalate prepolymers useful in practicing this invention include prepolymers made from the diallylic esters of ortho-, iso-, and terephthalic acids. These diallylic phthalates may be manufactured by polymerizing a monomeric material to produce a solution of soluble

prepolymer in monomer. Polymerization is carried to a point short of gelation. The prepolymer must then be separated from the unpolymerized monomer. This may be done by treatment with a solvent which dissolves the monomer and precipitates the prepolymer. Such a general process is described by Heilberger in U.S. Pat. No. 3,096,310. A conventional method of separating allylic prepolymer from monomer by precipitating the prepolymer in an unreactive liquid precipitant that is a solvent for the monomer and a nonsolvent for the prepolymer in a shearing zone is described by Willard in U.S. Pat. No. 3,030,341. Prepolymers may also be separated from unpolymerized monomer by distillation as disclosed by Mednick et al. in U.S. Pat. No. 3,285,836 issued May 28, 1968. The diallyl phthalate prepolymers are solids containing little or no monomer; they can be stored indefinitely in this form, since they require catalysts and either heat, actinic light or nuclear particle radiation to convert them to the insoluble or thermoset stage.

The polyester resins useful in practicing this invention, whether liquid or solid, should be of the reactive type, that is, at least about 50 mol percent of the dibasic acid portion of the polyester resin should be an unsaturated dibasic acid such as maleic or fumaric acid. If these polyester resins are diluted or cut with a monomer, the polyester resin should preferably be cut with a polyfunctional monomer. The alcohol moiety of the polyester is generally a saturated, difunctional glycol containing 2 to 8 carbon atoms which may be cyclic or acyclic.

A reactive polyester may be prepared by reacting equimolar amounts of maleic anhydride and diethylene glycol at a temperature about 200°C. Water is azeotropically removed. The distillate is analyzed from time to time for starting ingredients, and a sufficient amount of material may be added to the reactor to maintain the initial proportions of the reacting ingredients. After 8 hours' reaction at such a temperature, a polyester is obtained in the form of a viscous liquid with an acid number of about 18.

A solid reactive polyester can be prepared by reacting equimolar amounts of a mixture of maleic anhydride and isophthalic acid with a 10% excess of propylene glycol. Water is removed azeotropically. The reaction is continued by heating until the acid number of the reaction mixture falls below 5. Excess unreacted propylene glycol is removed under vacuum and a solid polyester is obtained. Polyesters made from chlorendic anhydride and equimolar amounts of maleic anhydride or fumaric acid are well known reactive flame-retardant polyesters that can be used in practicing this invention.

Polyfunctional monomers useful in practicing this invention include, but are not limited to, diallyl phthalate, diallyl isophthalate, diallyl chlorendate, diallyl maleate, triallyl cyanurate, triallyl isocyanurate, glycol dimethacrylate, divinyl benzene and the like. So far as it is known to the inventors, any polyfunctional unsaturated monomer may be used in practicing this invention.

The polyunsaturated materials and polyhalogenated aromatic hydrocarbons can be either compounded into a thermosetting composition, such as a molding compound, in place of part of the conventional thermosetting polymer, or can be prereacted to form a kind of gel polymer by mixing about 3 to 2 mols of polyhalogenated compounds with 2 to 3 mols of a polyunsaturated

monomer and subjecting the mixture to a free radical initiator, by incorporating 1 to 5% by weight, preferably about 1.5 to 3% by weight, of a peroxide catalyst in such a composition.

The preferred ratio of polyhalogenated aromatic compounds to polyunsaturated monomer, when using the prereacted materials, is 1 mol of polyhalogenated aromatic compound to each mol of polyunsaturated monomer. The free radical initiation should take place at a temperature of at least 130°C and below the boiling point of the monomer. The reaction is exothermic and the temperature attained during the exothermic reaction period of a 100 g mass should be between about 200 and about 250°C under adiabatic conditions. Generally the reaction mixture must reach a temperature of at least 130°C for 2 to 5 minutes to initiate the exothermic reaction. Preferably the polyhalogenated aromatic hydrocarbon and the polyunsaturated monomer are selected so that a hard, grindable reaction product is produced. Fillers may be incorporated in the prereaction mixture, provided that the filler does not cause initiation of the reaction at less than 130°C or inhibit reaction about 130°C. Inert fillers can comprise up to 60% by weight of the total composition.

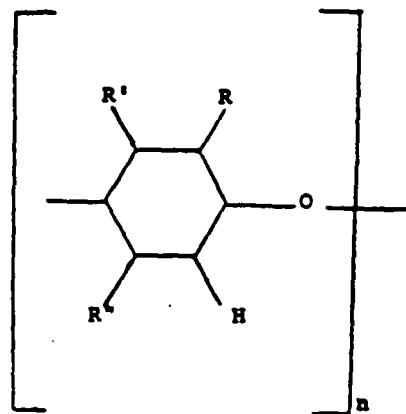
Typical polychlorinated aromatic hydrocarbons used in the practice of this invention are chlorinated biphenyls and terphenyls which are known to be so stable and chemically inactive that they are used as plasticizers, heat exchange fluids and the like. Diallyl phthalate resins and polyester resins, which are typical of the polyunsaturated materials used in practicing this invention, when cured, are known to be plasticized by chlorinated biphenyls and terphenyls. For example, ordinary molded diallyl phthalate articles containing chlorinated biphenyls do not have the characteristic "hot hardness" of diallyl phthalate molded articles; when removed from a hot mold, the molded articles containing a chlorinated biphenyl are soft, like hot rubber, and harden only on cooling. Physical mixtures of highly chlorinated biphenyls (some with melting points above 150°C) and an allylic prepolymer ball-milled together "ball up" or stick together in the ball mill (though either material alone will ball-mill in a conventional matter), thus showing solvation of resin by the polychlorinated aromatic compound. It is therefore quite surprising that, when there is at least 1 mol of a polyunsaturated monomer for every 3 mols of the polychlorinated aromatic compound, these polychlorinated aromatic hydrocarbons can be incorporated into a diallylic phthalate prepolymer or reactive polyester resin molding composition, which can be cured into articles that are essentially not plasticized by the polychlorinated aromatic compound.

The polyhalogenated aromatic hydrocarbon and polyunsaturated materials do not have to be prereacted. These materials may be incorporated into thermosetting resin compositions which are used for making moldings, castings, and laminates without substantially degrading the cured physical properties of the thermosetting resins, provided there is at least 1 mol of difunctional monomer for every 3 mols of polyhalogenated aromatic hydrocarbon in the thermosetting resin composition and the curing temperature is at least 130°C. Surprisingly, when cured to the thermoset stage, these novel thermosetting resin compositions exhibit excellent electrical and mechanical properties and are flame resistant. The polychlorinated or polybrominated aromatics have little or no plasticizing ef-

fect upon the cured thermoset compositions and there is substantially no loss of thermoset properties.

Polychlorinated and polybrominated aromatic hydrocarbons are commercially available and represent a low-cost halogen source for making resistant thermoset resin compositions. Surprisingly, compared to the more expensive flame-retardant compositions made with polyester or diallylic phthalate resins and diallyl chlorinate, the compositions of this invention have somewhat improved electrical properties. These novel compositions can be used with ordinary flame-retardant additives and synergists used in chlorinated flame-retardant resin compositions such as antimony trioxide and hydrated alumina.

Polyphenylene ether polymers can be used to replace some or all of the polyunsaturated polymers in these novel compositions, provided at least an additional 0.05 part by weight of polyunsaturated monomer is added to the composition for each part of polyphenylene ether polymer. Typical polyphenylene ether polymers have a repeating structural unit of the formula



wherein the oxygen atom of one unit is connected to the benzene nucleus of the adjoining unit,  $n$  is a positive integer and is at least 10,  $R$  is a monovalent substituent selected from the group consisting of hydrogen, hydrocarbon radicals free of tertiary alpha-carbon atom, halohydrocarbon radicals having at least two carbon atoms between the halogen atom and the phenol nucleus and being free of a tertiary alpha-carbon atom, hydrocarbonoxy radicals being free of a tertiary alpha-carbon atom, and halohydrocarbonoxy radicals having at least two carbon atoms between the halogen atom and phenol nucleus and being free of tertiary alpha-carbon atoms,  $R'$  and  $R''$  are both monovalent substituents which are the same as  $R$  and, in addition, halogen. All of the polyphenylene ether polymers currently available have been found to be useful in practicing this invention. A method of producing polyphenylene ether polymers by forming self-condensation products of phenylene by reacting oxygen with a phenol is described in U.S. Pat. No. 3,306,875 issued Feb. 28, 1967. We have used polyphenylene ether polymers available from the General Electric Company, such as Noryl and Grades 631-101, 691-111 and 631-111, and found them all to be useful in practicing this invention. As far as we know, all of the polyphenylene ether polymers known in the art are useful in practicing this invention.

The novel compositions of this invention employ a free radical initiator in sufficient amounts to convert the (polyunsaturated monomer, polyunsaturated polymer and polychlorinated aromatic hydrocarbon) compositions to the thermoset state upon the application of heat. Free radical initiation may be accomplished by electron beams or other sources of radiation including actinic light. Conveniently, peroxide catalysts which promote reaction between unsaturated compounds with carbon to carbon double bond unsaturation may be used. The peroxide catalyst does not have to exclude homopolymerization, but must be a catalyst that does not produce only homopolymers. We have found that when peroxide catalysts and heat are used to cure the compositions, peroxides which have a minimum 10 hour half life in benzene at a temperature in excess of 100°C should be used to some extent to catalyze the reactions of this invention i.e. 2-10 parts by weight per 100 parts of polymer-monomer-halogenated aromatic hydrocarbon mixture. Mixed catalysts may be used, but at least part of the catalyst must be a catalyst having a 10 hour half life at a temperature in excess of 100°C in benzene. We have successfully used dicumyl peroxide, tertiary butyl perbenzoate, 2,5-dimethyl-2,5-di(tert-butylperoxy)hexyne-3 and 2,5-dimethyl-2,5-di(tert-butylperoxy)hexane alone and in combination with benzoyl peroxide. We have found that dicumyl peroxide gives the best results in the novel compositions of this invention. Generally, catalysts well known in the art to cure allylic compositions are useful in practicing this invention.

A wide variety of water-insoluble, inert fillers may be used in preparing the molding compounds of this invention. Useful fillers include calcium carbonate, both precipitated and wet ground types, calcium silicate (wollastonite), silica, hydrated clays, calcined clays, chalk, calcium sulfate (anhydrous), barium sulfate, asbestos, glass (powdered), quartz, aluminum trihydrate, aluminum oxide, antimony oxide, magnesium oxide, inert iron oxides and groundstone such as granite, basalt, marble, limestone, sandstone, phosphate rock, travertine, onyx and bauxite. Additionally, inert fibrous materials may be used such as synthetic fibers, glass fibers, asbestos and cellulosic fibers. Up to 200 parts by weight of filler and/or fiber per 100 parts by weight of polyunsaturated polymerizable materials-polychlorinated aromatic compound may be used in these molding compositions.

Incidental additives widely incorporated in thermosetting molding compounds and laminates such as pigments, release agents, coupling agents, lubricants and other incidental additives may be incorporated in the compositions of this invention.

The novel molding compositions of this invention are prepared in conventional equipment and using techniques well known in the plastics industry to be useful in compounding allylic, epoxy, and polyester molding compounds. The molding compounds may be filled or unfilled and of the premixed, powdered, granular or dough type. The polyfunctional polymerizable materials, including polymer and monomer, a polyhalogenated aromatic hydrocarbon, free radical catalyst and an internal mold release, pigments, inhibitor, and so forth are simply mixed together in a heavy-duty mixer. The mixing may be done with or without the use of solvents. However, if solvents are used, they should be removed from the premixed compound before molding. The molding compositions can be molded under conditions

normally used for allylic molding compositions, that is, they may be molded at from about 130° to 180°C for about 1 to 60 minutes. Because of the varied viscosities of these novel molding compositions, the molding pressure may vary from near (0) zero to 10,000 pounds per square inch, depending on the composition.

There are two convenient ways to add the polychlorinated or polybrominated aromatic hydrocarbons to the polyunsaturated polymerizable materials. The preferred method of addition is to add the polyhalogenated aromatic hydrocarbon with the polyunsaturated materials as any other additive might be added in a molding compound or laminating solution. Although not the preferred method of addition, the polyhalogenated aromatic hydrocarbon may be added after it has been prereacted with a polyfunctional monomer. This is particularly useful where large amounts of monomer cannot be tolerated, as in high-pressure molding compounds. Direct addition of the polyhalogenated aromatic compound in preparation of the molding compound can be made by simply adding a polyunsaturated monomer and a polyhalogenated aromatic hydrocarbon to a molding compound prior to or during compounding in place of part of the polymer which would normally be used in the molding compound, providing there is at least 1 mol by weight of difunctional monomer present for each 3 mols of polyhalogenated aromatic compound.

The polyunsaturated polymerizable materials and polyhalogenated hydrocarbons have been prereacted to form solid reaction products by heating together a mixture of about 3 to 2 mols of polyhalogenated aromatic hydrocarbon with 2 to 3 mols of a polyunsaturated monomer in the presence of about 2% by weight of dicumyl peroxide. A number of filled and unfilled reaction products were prepared in a sigma blade mixer equipped with a steam jacket. Steam at a temperature of 160°C (100 pounds of pressure) was circulated through the jacket. The reactants, and in some cases filler and catalyst, were simply added to the sigma blade mixer with 160°C steam being supplied to the steam jacket and reacted together with agitation. Although the exact temperatures of the mixer blade and side walls were not known and accurate time measurements could not be made, reasonable initiation times, generally less than 10 minutes, were obtained. During the processing, the mixtures were highly fluid up to reaction and proceeded through reaction rapidly to a brittle rubber stage, finally breaking into small lumps. No particular advantage was observed for filled compositions. All of the reactants evaluated appeared to have about the same rate of reaction and the reaction appeared to be complete in a matter of 2 to 5 minutes after initiation. The hard reaction products were ball-milled for a period of 2 hours to produce a finely ground, uniform powder. The ratio of polyunsaturated monomer to polychlorinated aromatic compound was varied over ratios from 3 to 2 mols of monomer per 2 to 3 mols of polychlorinated aromatic compound. These products, due to their high chlorine content, were found to impart flame resistance to thermosetting compounds. These novel solid reaction products may be used in thermoplastics such as polyethylene, polyvinyl chloride acrylics and polystyrene, as well as rubber and other organic systems including thermosetting materials such as epoxy resins, polyurethane resins, polyimide resins and the like, where flame resistance is desirable.

These solid reaction products can also be added to unsaturated polyester resin compositions containing a monounsaturated monomer, for example a "styrene cut polyester". The physical properties of styrene-type polyester molding compounds containing the solid reaction products of this invention exceed the military specification requirements for polyester molding compounds as set forth in mil M-14F specification for MAI 60 Styrene-Polyester Molding compounds.

The materials of this invention permit compound versatility that has not previously been available. The materials can be used in processing compositions which are to be subjected to either low-pressure or high-pressure thermosetting conditions. Where it is desirable to incorporate these flame-retardant materials into high-pressure molding compounds such as in diallyl phthalate systems, the solid reaction products described above are preferred. These materials in the form of a fine powder are easily dispersed in an allylic resin composition in place of part of the allylic prepolymer. A diallylic phthalate molding composition containing the flame-retardant reaction products described above is processed in the same manner as conventional diallyl phthalate molding compounds. Up to 50% of the diallylic phthalate prepolymer can be replaced by these solid reaction products. Where it is desirable to achieve maximum flame retardancy in a molding compound or laminate with a minimum amount of halogen, it is advisable to incorporate antimony oxide along with the halogen-containing reaction product. Metal oxides such as hydrated alumina may be used with the antimony oxide. It is generally advisable to use a sufficient amount of the reaction product in a molding compound or laminate so that there is about 10 to 20% halogen based on the weight of the total organic content of the molding compound or laminate for high levels of flame resistance. However, less halogen is required where low levels of flame resistance can be tolerated.

Where low pressure, long flow laminating, impregnating and molding compositions are desired, the polyhalogenated aromatic hydrocarbon can be incorporated directly in allylic compositions or polyester compositions, provided there is at least 1 mol by weight of a polyunsaturated monomer for each 3 mols of polyhalogenated aromatic hydrocarbon and a peroxide catalyst which will not initiate the free radical curing at less than 100°C.

Polyphenylene ether polymers can be used to replace up to all of the polyunsaturated polymer in these novel compositions. When some or all of the polyunsaturated polymer is replaced with polyphenylene ether polymer, the compositions can be used as coating compositions, as glue lines for adhering materials to reinforced polymeric substrates or adhering two materials together, for use in reinforced laminates and as potting and encapsulating compositions. Although all of the polyunsaturated polymer can be replaced with polyphenylene ether polymer, it is preferred to retain at least some of the polyunsaturated polymer in the compositions, as the presence of the polyunsaturated polymer improves the handling characteristics of the compositions.

The novel compositions of this invention are readily used in preparing fiber reinforced laminates, generally fiber glass reinforced, by either the wet lay-up for prepreg techniques. Wet fiber glass lay-ups are prepared by making a liquid blend of the polymerizable materials, i.e., a monomer such as diallyl orthophthalate, a polymer containing carbon to carbon unsaturation or a

polyphenylene ether polymer, a polyhalogenated aromatic compound, a catalyst, and other modifying ingredients such as dyes, pigments, fillers, inhibitors, glass coupling agents and so forth, which is poured onto one or more layers of a fibrous non-woven glass mat or woven glass fabric, which has preferably been treated with a glass coupling agent, to impregnate the reinforcing glass; after impregnation, the product is laminated under heat and mild pressures according to procedures well known in the art to be useful for curing allylic resin laminates.

A typical slow cure is effected by placing the wet lay-up in a vacuum bag and applying a vacuum of 28 to 30 inches of mercury for about 1 to about 5 hours to remove bubbles; the evacuated lay-up is then preferably augmented by autoclave pressure at 30 to 50 psi for 30 minutes at 82°C, 60 minutes at 120°C, 30 minutes at 150°C, 15 minutes at 160°C, and then cured an additional 60 minutes at 160°C. Thin sections can be cured more rapidly; for example, 30 to 50 psi for 60 minutes at 160°C. The amount of glass in the lay-up can be as high as 80%, although the preferred amount of reinforcing glass is 50 to 75% by weight of the laminate.

The novel resin compositions of this invention can be employed in the usual process for manufacture of fibrous reinforced thermoset resin laminates using the prepreg technique. A polymerizable liquid, i.e., monomer such as diallyl orthophthalate and the like, a polyhalogenated aromatic hydrocarbon, a polyphenylene ether polymer, and, where desired, a polymer containing carbon to carbon unsaturation such as an allylic prepolymer or a reactive type polyester resin, a catalyst and modifying ingredients such as dyes, pigments, fillers, glass coupling agents, inhibitors and so forth are mixed together and used to impregnate a fibrous non-woven mat or a woven fabric; where glass mats or fabrics are used, it may be desirable to have the glass treated with a glass coupling agent. The use of some solvent is usually required in order to reduce the viscosity level of the resin composition to make it suitable for application to the mat or fabric with conventional, commercial saturating or impregnating equipment.

It is not necessary to dissolve the polyphenylene ether polymer in the impregnating compositions of this invention. Simple uniform dispersion of the polyphenylene ether polymer powder in the solvent-monomer-polymerizable resin mixtures suffices. Prepregs are generally most economically processed with 30 to 60 parts of the resin composition dispersed in 70 to 40 parts of a suitable solvent such as acetone, methylethyl ketone, methyl isobutyl ketone, toluene, xylene, chloroform, methylene chloride, trichloroethylene, perchloroethylene and mixtures thereof and other solvents known in the trade to be useful in preparing allylic prepregs.

The mat or fabric is impregnated with the solvent solution and then dried to remove the solvent. After impregnation and drying of the impregnated fabric, the laminate is laid up and cured with heat and mild pressure using cure cycles and conditions similar to those used in curing the wet lay-up type laminates. Roving, including glass roving, is similarly pre-impregnated for processing by filament winding techniques into pipe, other cylindrical shapes and hollow tapered and conical shapes. Products made by filament winding are generally cured at about 150°C in 60 minutes. The fiber content of the prepreg laminates varies from about 15 to about 40% by weight for low density fibers and up to

about 55 to 75% of the total weight of the cured laminate for glass mat or glass fabric laminates. The fiber content of filament wound constructions such as pipe, when made from impregnated glass roving, is generally about 70 to 80% of the total weight of the cured product.

Reinforced laminates of fibrous materials such as glass cloth, glass mats, synthetic fiber, cloth mats, paper and the like can be copper-clad to produce copper-clad laminates with excellent electrical properties to be used in preparing printed circuits and the like. Copper-clad laminates can be prepared by coating copper foil with a composition of this invention containing a polyphenylene ether polymer and then baking the coated copper foil at 160°C for about 15 minutes. The baked resin coated foil is then placed on resin impregnated fibrous materials such as glass cloth which has been impregnated with the novel resin compositions of this invention, unsaturated polyester resins, diallylic phthalate prepolymers and the like, and then the "laminate" is pressed at 50 to 2,000 psi at 100° to 170°C for at least 5 minutes to convert the resinous materials to the thermoset state. If desired, the coated copper foil can be laminated to almost any previously cured base. As indicated above, dicumyl peroxide is the preferred catalyst for producing the copper-clad laminates of this invention. The resulting copper-clad laminate has excellent adhesion of the copper to the base material and has excellent electrical properties. Quite surprisingly, when tested according to NEMA Standards Publication LI-1-1966, but at up to 200°C rather than 25°C standard, these copper-clad laminates retained essentially all of their electrical properties as measured at room temperature. Reinforced laminates made from those compositions of this invention which contain polyphenylene ether polymers adhere very well to the type of copper foil used in making printed circuit boards without precoating the foil. The compositions containing a polychlorinated aromatic hydrocarbon apparently "wet" the metal surface better than the similar compositions without the polychlorinated aromatic hydrocarbon disclosed in our copending application Ser. No. 682,326, filed Nov. 13, 1967.

Those compositions of this invention which contain polyphenylene ether resins as a portion of the system are useful as thermosetting coatings. They adhere strongly to a wide variety of substrates involving metals, ceramic bases and other resinous substances while providing excellent electrical insulating characteristics. They may be used as conformal coatings for electronic devices or as primer coats to bond additional electrical insulation to the devices. Many molding compounds used in the encapsulation of electronic components do not form good bonds to the devices and to the wire leads, thereby permitting access of moisture. When such devices or leads are first coated with the compositions of this invention, and then subjected to mold encapsulation, hermetically sealed systems are obtained which resist moisture even on prolonged exposure to boiling in water or high pressure steam.

The coatings may be formulated with all polymer components in solution. Useful solvents comprise chlorinated hydrocarbons and aromatic hydrocarbons. Alternatively, the coatings may be formulated as finely divided dispersions of the polymer system in liquid organic media. Useful dispersion media include organic

ketones and esters. Dispersed coatings are preferred for most applications, since they may be formulated at high polymer solids levels yet are capable of application by brushing, spraying or roller coating. Coatings containing up to 80% solids can be applied by knife coating techniques, yet they flow out to smooth adherent insulating varnishes when subjected to oven baking at temperatures of 120°C or higher.

The test methods appearing in the following list were followed in testing the molded and laminated test specimens made from the various compositions disclosed in the examples.

15	A. Flexural Strength*	ASTM	D-790
	B. Modulus of Elasticity	"	D-790
	C. Tensile Strength	"	D-638
	D. Izod Impact	"	D-256
	E. Compressive Strength	"	D-693
	F. Deflection Temperature	"	D-648
	G. Water Absorption	"	D-570 (a)
20	H. Specific Gravity	"	D-792
	I. Dielectric Constant	"	D-150
	J. Dissipation Factor**	"	D-150
	K. Volume and Surface Resistivity	"	D-257
	L. Flame Resistance (Burning and Ignition Tests Method 1 and 2)	"	D-229
25	M. Hardness	"	D-785
	N. Hardness (Rockwell)	"	D-785
	O. Shear Strength (Modified for Flat Beams)	"	D-2344

\*The flexural strengths at 150°C for the unfilled resin systems were obtained after 4 hour conditioning at 150°C. Each individual specimen was held to 4 hour conditioning to avoid any ambiguity that may be caused by post-curing in the test oven. Only maximum flexural yield was measured.

\*\*The wet test is conducted on samples which were conditioned by immersing the samples for 24 hours at 25°C in distilled water, removing the samples, blotting them dry and then testing the samples as soon as practical according to the test method

The following examples, illustrating the novel products disclosed herein, are given without any intention that the invention be limited thereto. All parts and percentages are by weight unless otherwise noted.

#### EXAMPLE I

A series of samples were prepared which were simple mixtures of chlorinated terphenyl containing 60% chlorine or a chlorinated biphenyl containing 68% chlorine with different monomers and 2% by weight of dicumyl peroxide based on the total weight of the chlorinated aromatic compound and monomer. The samples were placed in a paper cup and a thermocouple connected to a recording thermocouple bridge was inserted in each of the samples. The samples were placed in an oven maintained at a temperature of 160°C. Various concentrations of the chlorinated aromatics with various monomer concentrations were prepared. Some of the samples tested were subjected to a 16 hour acetone Soxhlet extraction at the reflux temperature of acetone. The composition details, the reaction initiating temperature, the reaction peak exotherm temperature, the reaction time to peak exotherm, and the results of the reaction are contained in Table I.

Soxhlet extraction studies were made on some of the compositions. Mixtures of the unreacted chlorinated aromatics and the monomers yield clear solutions in chloroform and hot acetone. The chlorinated aromatics can only be partially extracted from the reaction product with monomers, and that fraction which is extracted is different from the original crystalline chlorinated aromatic in that it tends to be a resinous gum.



TABLE I

	BATCH REACTION AT 160°C. MONOMER STUDY											
	1	2	3	4	5	6	7	8	9	10	11	12
Chlorinated terphenyl, 60% chlorine (Aroclor 5460)	50	45	40	60	65	60	—	—	40	74	87	78
Chlorinated biphenyl, 68% chlorine (Aroclor 1268)	—	—	—	—	—	—	62	62	—	—	—	—
Diallyl phthalate	—	—	—	—	—	40	—	38	—	—	—	—
Diallyl isophthalate	50	55	60	40	35	—	38	—	—	—	—	—
Diallyl chlrendate	—	—	—	—	—	—	—	—	60	—	—	—
Triallyl cyanurate	—	—	—	—	—	—	—	—	—	26	—	—
Triallyl phosphate	—	—	—	—	—	—	—	—	—	—	13	—
Diallyl maleate	—	—	—	—	—	—	—	—	—	—	—	22
Divinyl benzene (55%)	—	—	—	—	—	—	—	—	—	—	—	—
Ethylene Glycol Dimethacrylate	—	—	—	—	—	—	—	—	—	—	—	—
Dicumyl peroxide	2	2	2	2	2.5	2	2	2	2	2	2	2
Initiation Temp. °C.	130	130	130	135	155	125	145	140	135	125	135	122
Peak Exotherm Temp. °C.	223	211	227	218	273	222	225	216	205	245	248	230
Time to Exotherm, min.	3	5	7	4	5	3	3	3	3	4	4	3
16 hr. Acetone Soxhlet Extraction												
Nature of Solution												
Clear												
Cloudy				x			x	x				
Dried Extract												
Powder												
Gum				x			x	x				

	Comparison					
	13	14	C-1	C-2	C-3	C-4
Chlorinated terphenyl, 60% chlorine (Aroclor 5460)	70	70	100	—	—	50
Chlorinated biphenyl, 68% chlorine (Aroclor 1268)	—	—	—	—	100	—
Diallyl phthalate	—	—	—	80	—	50
Diallyl isophthalate	—	—	—	—	—	—
Diallyl chlrendate	—	—	—	—	—	—
Triallyl cyanurate	—	—	—	—	—	—
Triallyl phosphate	—	—	—	—	—	—
Diallyl maleate	—	—	—	—	—	—
Divinyl benzene (55%)	30	—	—	—	—	—
Ethylene glycol dimethacrylate	—	30	—	—	—	—
Dicumyl peroxide	2	2	2	2	2	—
Initiation temp. °C.	125	115	none	110	none	none
Peak exotherm temp., °C.	235	200	none	250	none	none
Time to exotherm, min.	3	4	x	5	x	x
16 hr. Acetone Soxhlet Extraction						
Nature of Solution						
Clear			x	x		
Cloudy					x	
Dried Extract						
Powder			x		x	
Gum				x		

## EXAMPLE 2

Diallyl phthalate prepolymer, prereacted poly-chlorinated aromatic compound with monomer, molding compounds were prepared. The prereacted poly-chlorinated aromatic hydrocarbon and monomer reaction products were prepared in 3½ lb. batches. Chlorinated biphenyls, containing 68% combined chlorine (Aroclor 1268 from Monsanto Chemical Company), and a diallylic phthalate monomer were heated together in a ratio of 62 parts of chlorinated biphenyls to 38 parts of diallylic phthalate monomer with 2 parts by weight of dicumyl peroxide per 100 parts of Aroclor plus monomer. The reaction products were made in a 1-gallon sigma blade mixer equipped with a steam jacket. The steam to the jacket was at 160°C and the internal temperature of the blade and side walls was about 120°C. Although accurate reaction time measurements could not be made, reasonably short reaction times of about 10 minutes were observed in preparing these reaction products. The reactants were

highly fluid up to the point of gelation, from which point the reaction proceeded rapidly to a brittle stage, breaking up thereafter into small lumps. The prereacted materials were ball-milled for a 2-hour period in a 1½ gallon jar mill to produce 1,000 g of ground product. Reaction products were prepared using diallyl phthalate and diallyl isophthalate monomer and are designated Reaction Product No. 1 and Reaction Product No. 2 respectively in Table II. The reaction products were mixed with diallyl phthalate prepolymer and diallyl isophthalate prepolymer along with some catalyst, monomer and fillers to make molding compounds. The composition details and physical properties of samples molded from the molding compounds are found in Table II. For comparison, the table includes qualifying specifications for diallyl phthalate molding compounds for military use under MIL M-14F and MIL-19833. (The largest market for the diallyl phthalate molding compounds in the United States is for military applications.)

TABLE II

MOLDING COMPOUNDS			MIL M-14F Specification Requirements for SDGF Molding Compounds
Formulation			
Diallyl phthalate prepolymer (Dapon 35, FMC Corp.)	74		
Diallyl isophthalate prepolymer (Dapon M, FMC Corp.)		70	
Diallyl phthalate monomer	3		
Diallyl isophthalate monomer		3	
Reaction Product No. 1 <sup>111</sup>	26		
Reaction Product No. 2 <sup>111</sup>		30	
tert Butyl perbenzoate	3	3	
Calcium Stearate	2	2	
Antimony oxide	15	18	
Wollastonite	30	20	
Glass (1/8" strands)	90	100	
Properties			
Ignition time (sec.)	93	98	90 (min.)
Burning time (sec.)	78	76	90 (max.)
Heat Deflection Temp. °C.	175	270	160 (min.)
Impact (ft. lbs.)	0.66	0.60	0.30 (min.)
Compressive (psi) Strength	24,620	26,500	16,000 (min.)
Flexural (psi) Strength	11,462	11,200	9,000 (min.)
Modulus (psi) of E			
Elasticity	1.70 x 10 <sup>6</sup>	1.72 x 10 <sup>6</sup>	—
Water absorption (%)	0.12	0.11	0.40 (max.)
Specific Gravity	1.902	1.897	—
D.C. 10 <sup>3</sup> /10 <sup>6</sup> cm	4.50/4.39	4.36/4.24	4.6/4.4 (max.)
D.C. 10 <sup>3</sup> /10 <sup>6</sup> (wet)	4.52/4.38	4.41/4.29	4.7/4.5 (max.)
D.F. 10 <sup>3</sup> /10 <sup>6</sup> cm	.005/.005	.005/.005	.009/.015 (max.)
D.F. 10 <sup>3</sup> /10 <sup>6</sup> (wet)	.006/.005	.006/.005	.013/.017 (max.)
Volume Resist. (ohm)	1.0 x 10 <sup>11</sup>	0.8 x 10 <sup>11</sup>	5 x 10 <sup>10</sup> (min.)
720 hr. at 70°C., 100% R.H.			

<sup>111</sup> 62/38 reaction product of chlorinated biphenyl with diallyl phthalate monomer.

<sup>112</sup> 62/38 reaction product of chlorinated biphenyl with diallyl isophthalate monomer.

<sup>113</sup> Dielectric constant.

<sup>114</sup> Dispersion Factor.

## EXAMPLE 3

A portion of the diallyl phthalate monomer-chlorinated biphenyl Reaction Product, Reaction Product No. 1 of Example 2, was incorporated into a diallyl phthalate polyester molding compound. Test specimens were molded from the compound for 3 minutes at 150°C at 2,000 psi (pounds per square inch). The composition of the molding compound, the physical and electrical properties of the molded test specimens and the MIL-M-14F specification requirements for MAI 30 diallyl phthalate polyester molding compounds are shown in Table III.

TABLE III

MAI-30 (DAP-POLYESTER) COMPOUNDS			MIL M-14 F Specification Requirements for MAI-30 (DAP-Polyester) Molding Compounds
Formulation			
Diallyl phthalate monomer	35		
Uncut isophthalate polyester resin, reactive type,	35 <sup>111</sup>		
Reaction product No. 1	30 <sup>112</sup>		
Glass (1/8" strands)	57		
Hydrated alumina	48		
Wollastonite	48		
Antimony oxide	18		
Calcium stearate	2		
tert Butyl perbenzoate	2		
Physical Properties			
Compressive	21,200	20,000 (min.)	
D.C. 10 <sup>3</sup> /10 <sup>6</sup>	4.95/4.54	6.3/6.2 (max.)	
D.C. 10 <sup>3</sup> /10 <sup>6</sup> (wet)	5.15/4.63	6.4/6.4 (max.)	

TABLE III-continued

MAI-30 (DAP-POLYESTER) COMPOUNDS			MIL M-14 F Specification Requirements for MAI-30 (DAP-Polyester) Molding Compounds
Formulation			
D.F. 10 <sup>3</sup> /10 <sup>6</sup>	.010/.009	.015/.012 (max.)	
D.F. 10 <sup>3</sup> /10 <sup>6</sup> (wet)	.012/.011	.017/.015 (max.)	
Flexural (psi)	16,700	14,000 (min.)	
Flexural Modulus (psi)	2.0 x 10 <sup>6</sup>	—	
Heat Deflection Temp. °C	272	200 (min.)	
Impact (ft. lbs.)	4.37	3.0 (min.)	
Water Absorption (%)	0.17	0.5 (max.)	
Ignition time (sec.)	105	100 (min.)	
Burning time (sec.)	17	75 (max.)	
Rockwell Hardness (M)	108	—	
Specific Gravity	1.93	—	
Vol. Resistivity (ohm.cm.)	1.2 x 10 <sup>11</sup>	—	
Surface Resistivity (ohm.)	2.8 x 10 <sup>11</sup>	—	

<sup>111</sup> Equimolar amounts of maleic anhydride and isophthalic were heated together with a 10% excess of diethylene glycol. Water was removed azeotropically and the diethylene glycol was removed.

<sup>112</sup> 62/38 Reaction Product of chlorinated biphenyl with diallyl phthalate monomer from Example 2.

<sup>113</sup> Heating at about 200°C. was continued until the acid number of the polyester under vacuum and the polyester resin recovered.

## EXAMPLE 4

Example 3 was repeated except that a styrene polyester was used rather than a DAP polyester with a portion of the diallyl phthalate-chlorinated biphenyl gel of Example 2. The molding compositions were molded for 3 minutes at 150°C at 2,000 psi. The composition of the molding compound, physical and electrical properties of the molded samples, and, for comparison, the MIL

M-14 F specification requirements for MAI 60 flame-resistant styrene polyesters are shown in Table IV.

TABLE IV

STYRENE-POLYESTER MOLDING COMPOUNDS		
MIL M-14 F Specification Requirements for MAI 60 (Styrene-Polyester) Molding Compounds		
Formulation		
Monomer, styrene	24.5	
Polyester Resin <sup>***</sup>	45.5	
Reaction Product No. 1 <sup>m</sup>	30	
Glass (1/8" strands)	67	
Hydrated alumina (Hydral 710)	48	
Wollastonite	48	
Antimony oxide	18	
Calcium stearate	2	
tert Butyl perbenzoate	2	
Properties		
Compressive (psi)	23,150	18,000 (min.)
D.C. 10 <sup>3</sup> /10 <sup>4</sup>	4.77/4.53	6.0/5.7 (max.)
D.C. 10 <sup>3</sup> /10 <sup>4</sup> (wet)	5.04/4.51	7.0/6.0 (max.)
D.F. 10 <sup>3</sup> /10 <sup>4</sup>	.012/.008	.03/.03 (max.)
D.F. 10 <sup>3</sup> /10 <sup>4</sup> (wet)	.016/.013	.08/.05 (max.)
Flexural (psi)	17,700	12,000 (min.)
Flexural modulus (psi)	2.0 x 10 <sup>6</sup>	—
Heat Deflection Temp. °C	272	200 (min.)
Impact (ft. lbs.)	7.45	6.0 (min.)
Water Absorption (%)	0.17	1.5 (max.)
Ignition time (sec.)	95	90 (min.)
Burning time (sec.)	58	90 (max.)
Rockwell Hardness (M)	107	—
Specific Gravity	1.898	—

<sup>\*\*\*</sup> Reactive polyester resin from Example 1 made by reacting equimolar portions of maleic and isophthalic acid with a 10% excess of diethylene glycol.

<sup>m</sup> a2/38 Reaction Product No. 1 of chlorinated terphenyl with diallyl phthalate monomer from Example 2.

## EXAMPLE 5

High pressure laminates containing diallylic phthalatepolychlorinated aromatic reaction products were prepared using polychlorinated aromatic diallylic phthalate reaction products made according to Example 2. Sixty parts of polychlorinated aromatic compound (Aroclor 5460), a chlorinated terphenyl containing 60% by weight of chlorine, was reacted with 40 parts of diallylic phthalate monomer and 2 parts of dicumyl peroxide as described in Example 2, and is labeled Reaction Product No. 3 in Table V. A similarly prepared reaction product, using 40 parts of diallyl isophthalate monomer in place of diallyl phthalate monomer, is labeled Reaction Product No. 4 in Table V. A glass cloth laminate was prepared as follows: A "resin blend" of the polychlorinated aromatic diallylic phthalate reaction product with the corresponding diallylic phthalate prepolymer and a small amount of diallylic phthalate monomer and 2 parts of dicumyl peroxide catalyst per 100 parts of reactants as shown in Table V was dispersed, in the amount of 100 parts by weight of the resin blend, in 100 parts by weight of acetone. Woven glass cloth was impregnated with this dispersion and allowed to dry at least 48 hours in air at room temperature. The impregnated glass cloth, often termed prepreg, was cut into 12 by 12 inch squares and stacked 18 plies deep with the warp yards parallel. The pre-impregnated lay-up was laminated in a flat bed press for 30 minutes at 80°C at contact pressure, 30 minutes at 120°C at 300 psi and 1 hour at 160°C at 300 psi. Two samples were prepared, one using diallyl phthalate monomer and another sample using diallyl isophthalate prepolymer and diallyl isophthalate monomer. The composition details of the resin mixture used

to impregnate the glass fabric, the resin content of the prepregs prepared from the resin systems and the physical properties of the cured laminates are found in Table V.

TABLE V

## HIGH PRESSURE DIALLYL PHTHALATE CHLORINATED TERPHENYL GFL LAMINATES

Formulation		
10	Reaction Product No. 3	50
	Reaction Product No. 4	50
	Diallyl phthalate prepolymer (Dapon 35)	45
	Diallyl isophthalate prepolymer (Dapon M)	45
	Diallyl phthalate monomer	5
15	Diallyl isophthalate monomer	5
	Dicumyl peroxide	2
Physical Properties		
	Burning <sup>***</sup> time (sec.)	12.7
	Compressive (psi)	49,500
	Rockwell Hardness (M)	118
	Flexural (psi) R.T.	67,800
20	150°C	13,700
	Modulus (psi) R.T.	2.43 x 10 <sup>6</sup>
	150°C	0.97 x 10 <sup>6</sup>
	Tensile (psi)	49,100
	Specific Gravity	1.822
	Shear (psi)	4,010
	Flexural (psi) 2 hr. boil	67,900
25	Modulus (psi) 2 hr. boil	2.48 x 10 <sup>6</sup>
	Flexural (psi) after aging:	at 155°C
	1 day	68,200
	4 days	70,900
	10 days	65,600
	D.C. 10 <sup>3</sup> /10 <sup>4</sup>	4.46/4.35
30	D.C. 10 <sup>3</sup> /10 <sup>4</sup> (wet)	4.52/4.38
	D.F. 10 <sup>3</sup> /10 <sup>4</sup>	.0055/.0054
	D.F. 10 <sup>3</sup> /10 <sup>4</sup> (wet)	.0063/.0061
	Vol. Resist. (ohm. cm.)	5.2 x 10 <sup>10</sup>
	Surface Resist. (ohm.)	1.3 x 10 <sup>10</sup>
	Insulation Resist. (ohm.)	—
	10 day cycle	72/70/100
35		72/25/100
		100/70/100
		8.3 x 10 <sup>9</sup>
		1.25 x 10 <sup>10</sup>
		5.60 x 10 <sup>11</sup>
		6.3 x 10 <sup>11</sup>
		3.6 x 10 <sup>10</sup>
		1.9 x 10 <sup>11</sup>

<sup>\*\*\*</sup>ASTM D229, Part I

## EXAMPLE 6

A molding compound was prepared directly from a mixture of a chlorinated terphenyl (Aroclor 5460 containing 60% combined chlorine by weight) and diallyl phthalate prepolymer (Dapon 35) and diallyl phthalate monomer. The following composition was compounded as an "SDG-F Type" flame-resistant molding compound designed to meet Military Specification MIL M-14 F.

Parts by weight	
Diallyl phthalate prepolymer (Dapon 35)	73
Diallyl phthalate monomer	9
Chlorinated terphenyl (60% combined chlorine)	18
Chopped glass (1/8" strands)	90
Clay, hydrated	30
Antimony oxide	15
Calcium stearate	1
Tertiary butyl perbenzoate	3
Acetone	160

All the ingredients in the above were mixed together in a heavy-duty mixer until the glass and the fillers were thoroughly wetted. The composition was spread on trays and the acetone was allowed to evaporate at room temperature. The composition was then compounded on a two-roll rubber mill at a temperature of 80°-90°C. The mill sheet was cooled and ground into a molding powder. Test pieces molded at 150°C at 2,000 psi for a

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minimum of 1.5 minutes showed full cure as measured by development of hot hardness and resistance to boiling chloroform. The test data in the following table show the properties of the above molded compositions compared to the MIL M-14 F specification requirements for diallyl phthalate molding compounds.

TABLE VI

CHLORINATED TERPHENYL AROCLOR/MONOMER/DAPON SDG-F COMPOUND		
	Example 6	Comparison <sup>10</sup>
Ignition time (sec.)	97	90 (min.)
Burning time (sec.)	70	90 (max.)
Heat Deflection Temp. °C	165	160 (max.)
Rockwell Hardness (M)	115	—
Impact (ft. lb.)	0.58	0.30 (min.)
Flexural strength, psi	12,120	9000 (min.)

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the laminates were placed in a vacuum bag and evacuated to remove air from the polymerizable liquid resin mixture. The lay-up was laminated while maintaining the vacuum at pressures at 15 psi or less, as in the very early stages of cure it was very difficult to maintain pressure on the polymerizable liquid. Full cure was obtained after about 15 minutes at 150°C as measured by hot hardness, hot strength, and solvent resistance in boiling acetone. This laminate construction was also cured in a vacuum bag by exposure to a 3 MEV (million electron volts) electron beam in multiple passes of 5 megarads each until a room temperature Barcol hardness of 60 was developed. The polymerizable liquid details and the physical properties of the cured laminates are set forth in Table VII.

TABLE VII

VACUUM BAG LAMINATES			
Formulation			
Diallyl phthalate prepolymer (Dapon 35)	30		
Diallyl isophthalate prepolymer (Dapon M)		50	
Diallyl phthalate monomer	20		
Diallyl isophthalate monomer		20	
Chlorinated terphenyl (Aroclor 5460)	30		30
Dicumyl peroxide	2		2
Burning time (ASTM D229, Pt. 1)	16 sec.		27 sec.
Specific Gravity	2.104		2.040
Percent Resins	28.0		32.7
Rockwell Hardness (M)	117		121
Flex. strength, psi, 25°C	94,000		101,700
Flex. strength, psi, 150°C	25,410	(260°C)	6,050 <sup>10</sup>
Modulus, psi, 25°C	$4.08 \times 10^6$		$3.86 \times 10^6$
Modulus, psi, 150°C	$2.49 \times 10^6$	(260°C)	$1.47 \times 10^6$ <sup>10</sup>
Compressive Str., psi	49,110		48,740
Tensile Str., psi	63,460		62,110
Shear, psi	3710		2040
Flex., 2 hr. H <sub>2</sub> O/100°C	93,390		88,810
Modulus, 2 hr. H <sub>2</sub> O/100°C	$4.18 \times 10^6$		$2.26 \times 10^6$
Flexural (psi) after aging:			
1 day	101,900		95,400
4 days	97,660		92,870
10 days	93,900		88,580
D.C. $10^3/10^6$	4.94/4.85		4.72/4.65
D.C. $10^3/10^6$ (wet)	5.00/4.84		4.81/4.65
D.F. $10^3/10^6$	.0034/.0049		.0068/.0040
D.F. $10^3/10^6$ (wet)	.0088/.0057		.0091/.0048
Vol. Resistivity, (ohm cm.)	$4.17 \times 10^{11}$		$3.56 \times 10^{11}$
Surf. Resistivity, (ohm)	$5.08 \times 10^{12}$		$2.36 \times 10^{12}$

<sup>10</sup> Test run at 260°C

Modulus, psi	$1.72 \times 10^6$	—
Compressive strength, psi	23,060	16,000 (min.)
Water absorption (%)	0.08	0.30
Specific gravity	1.880	—
D.C. $10^3/10^6$	4.44/4.35	4.6/4.4 (max.)
D.C. $10^3/10^6$ (wet)	4.47/4.37	4.7/4.5 (max.)
D.F. $10^3/10^6$	.0044/.0043	.0091/.015 (max.)
D.F. $10^3/10^6$ (wet)	.0054/.0047	.013/.017 (max.)
Vol. Resist., (ohm cm)	$6.76 \times 10^{11}$	—
Surf. Resist., (ohm)	$7.95 \times 10^{12}$	—
Vol. Resist., (ohm) (720 hrs. 70°C at 100% relative humidity)	$1.97 \times 10^{12}$	$5 \times 10^6$

<sup>10</sup> MIL M-14F Specification requirement properties for glass filled flame-retardant diallyl phthalate molding compound.

## EXAMPLE 7

Wet lay-up or vacuum bag laminates were prepared using a diallylic phthalate prepolymer, a diallylic phthalate monomer and chlorinated terphenyl (Aroclor 5460). The wet lay-up was prepared by making a liquid blend of the monomer, chlorinated terphenyl and diallylic phthalate resin and catalyst. The liquid polymerizable resin composition was poured into several layers of woven glass fabric. After impregnation,

## EXAMPLE 8

A premixed molding compound containing a portion of the polyester resin described in Example 3 and a polychlorinated aromatic compound was prepared. The formulations listed in Table VIII were mixed in a heavy duty mixer until the glass and fillers were thoroughly wetted with the polyester resin-monomer-chlorinated terphenyl mixture. The compositions of this invention were compared with similarly compounded conventional diallyl phthalate polyesters. The composition details and physical properties are set forth in the following Table. The test specimens were molded at 1000 psi at 150°C for 3 minutes.

TABLE VIII

PREMIX MOLDING COMPOUNDS			
Formulation			
Polyester resin, Reactive isophthalate type <sup>10</sup>	50		41
Diallyl phthalate monomer	50		41
Chlorinated terphenyl (60% chlorine)	0		18
4" Chopped glass fibers	57		57

TABLE VIII-continued

PREMIX MOLDING COMPOUNDS		
Formulation		
Wollastonite	55	55
Hydrated alumina (Hydral 710)	55	55
tert Butyl perbenzoate	2	2
Zinc stearate	1	1
Antimony oxide	18	18
Physical properties		
Ignition time, sec.	103	102
Burning time, sec.	244	31
Deflect. temp. °C	283	270
Rockwell Hardness (M)	102	103
Izod Impact (ft.lbs.)	4.90	6.90
Specific Gravity	1.935	1.933
Water absorption, (%)	0.37	0.25
Compressive str., psi	20,360	21,450
Flex. strength, psi	15,860	18,450
Modulus, psi	2.0 × 10 <sup>6</sup>	2.01 × 10 <sup>6</sup>
D.C. 10 <sup>3</sup> /10 <sup>6</sup>	5.56/4.85	5.11/4.66
D.C. 10 <sup>3</sup> /10 <sup>6</sup> (wet)	6.25/5.08	5.46/4.79
D.F. 10 <sup>3</sup> /10 <sup>6</sup>	.023/.016	.014/.010
D.F. 10 <sup>3</sup> /10 <sup>6</sup> (wet)	.041/.025	.018/.016
Vol. Resistivity, ohm-cm	6.82 × 10 <sup>14</sup>	8.74 × 10 <sup>14</sup>
Surf. Resistivity ohms	7.43 × 10 <sup>15</sup>	5.13 × 10 <sup>15</sup>

<sup>100</sup> Reactive polyester resin from Example 3 made by reacting equimolar portions of maleic and naphthalic acids with a 117% excess of diethylene glycol.

## EXAMPLE 9

A low-pressure laminate containing diallyl isophthalate monomer and prepolymer, a chlorinated terphenyl and polyphenylene ether polymer was prepared in which the following formulation was used to saturate glass cloth from which the laminate was prepared.

	Parts by Weight
Polyphenylene ether resin (General Electric Grade 691-111)	25
Chlorinated terphenyl containing 60% combined chlorine (Aroclor 5460)	15
Diallyl isophthalate prepolymer (Dapon M)	30
Diallyl isophthalate monomer	30
Dicumyl peroxide (recrystallized)	3
Acetone	130

The diallyl isophthalate prepolymer and polyphenylene ether polymer were ball-milled together until 70% of the product was -200 mesh (U.S. Standard Sieve Series 1940). The chlorinated terphenyl-diallyl isophthalate monomer and dicumyl peroxide were dissolved in the acetone solvent and the ball-milled powder was added to this solution to make up a saturating mixture. Woven glass cloth, type 181, was impregnated with this mixture and allowed to dry at least 48 hours at room temperature. The dry impregnated glass cloth was cut into 12 by 12 inch squares, stacked 18 plies deep with the warp yarns parallel. The pre-impregnated lay-up was made in a flat bed press for 30 minutes at 80°C at contact pressure, 30 minutes at 120°C at 300 psi and 1 hour at 160°C at 300 psi. The laminated sample had a resin content of 44% and was found to be non-burning in ASTM Flammability Test D-229, Part 1. The following physical properties were obtained on the laminated sample:

Laminate Physical and Electrical Properties

Resin content	44%
Specific Gravity	1.752
Rockwell Hardness (M)	120
Compressive strength, psi	50,400
Tensile strength, psi	43,700
Flexural strength, psi at 23°C	67,300

-continued

Laminate Physical and Electrical Properties

Flexural strength, psi at 200°C	13,700
Flexural modulus at 23°C, psi	3.0 × 10 <sup>6</sup>
Flexural modulus at 200°C, psi	1.3 × 10 <sup>6</sup>
Tensile shear strength, psi	3,530

## EXAMPLE 10

A vacuum bag laminate was prepared from a composition containing no polyunsaturated polymer. Thirty parts of polyphenylene ether polymer was ball-milled 1 hour together with 10 parts of trihydrate of aluminum oxide (Hydral 710 from Alcoa). The ball-milled materials were mixed with a solution containing 90 parts of diallyl isophthalate monomer, 17.5 parts of chlorinated terphenyl containing 60% by weight combined chlorine (Aroclor 5460) and 4.5 parts of dicumyl peroxide catalyst (DiCup R) with stirring at 60° to 80°C. This mixture was used to saturate 12 plies of 181 glass cloth 12 inches square. The impregnated cloth was stacked 12 plies deep with the warp yarns parallel. The impregnated lay-up was placed in a vacuum bag which was then evacuated until essentially all of the air was removed. The lay-up was then laminated in a flat bed press, maintaining the vacuum, throughout the lamination, for 1 hour at 93°C, 1 hour at 121°C and 1 hour at 160°C. The cured laminate had good "hot" hardness when removed from the vacuum bag at about 150°C and when cooled was a tough flexible laminate.

## EXAMPLE 11

A copper-clad laminate was prepared by roller coating onto 1 oz. copper foil (1 oz. per sq. ft.), a standard copper used in the preparation of printed circuit boards, the following coating composition:

Ingredients	Parts by weight
Polyphenylene ether polymer (General Electric Company Grade 631-101)	60
Diallyl isophthalate prepolymer (FMC Corp., Dapon M)	10
Diallyl isophthalate monomer	10
Chlorinated terphenyl, 60% chlorine (Aroclor 5460), Monsanto Chemical Corp.	20
Dicumyl peroxide	5
Chloroform	130
Trichloroethylene	150

After solvent removal and baking for 5 minutes at 165°C, the average film thickness of the coating on the copper was 1.5 mil. An 18 ply, low pressure, glass cloth laminate was prepared for laminating as described in Example 9. The treated copper foil was placed on one side of the laminate, with the treated side of the foil facing the laminate, and the laminate was then cured as described in Example 9.

The bond and peel strength of the copper foil to the laminate base after laminating exceeded 10 lbs. per inch. This bond strength was essentially unchanged after immersion in a solder bath at 260°C for 2 minutes.

## EXAMPLE 12

A copper-clad glass cloth laminate was prepared using the following coating composition to saturate glass cloth as described in Example 9:

Three hundred twenty parts of polyphenylene ether polymer (General Electric Grade 691-111), 80 parts of

diallyl isophthalate prepolymer (Dapon M) and 50 parts of hydrated alumina (Hydral 710) were ground together in a ceramic ball mill until 70% of the mixture passed through a 200 mesh sieve (U.S. Standard Sieve Series, 1940). Forty-five parts of this powdered mixture was added with agitation to a solution of 30 parts of diallyl phthalate monomer, 30 parts of chlorinated terphenyl (Aroclor 5460), 10 parts of diallyl isophthalate prepolymer (Dapon M), and 5 parts of recrystallized dicumyl peroxide (DiCup R — Hercules Powder Company) in 100 parts of methyl ethyl ketone.

The saturated glass cloth was cut into 12 inch squares prepared for laminating as described in Example 9. A 12 inch square of 1 oz. copper foil was placed on one side of the saturated glass cloth lay-up which was then laminated (cured) as described in Example 9 to produce a copper-clad laminate. When laminated, the copper foil had a bond strength to the glass cloth base in excess of 8 lbs. per inch peel strength at 90°. This bond strength was maintained, and the laminate maintained its integrity, when immersed for 2 minutes in solder at 260°C.

#### EXAMPLE 13

A silicon diode was coated by dipping the diode into the polymerizable coating composition of Example 11. Only about one-eighth inch of the metal leads (Covar) to the diode, nearest to the body of the diode, was coated, thereby leaving part of the leads uncoated to facilitate soldering. The coated diode was baked in an air circulating oven at 170°C for 2 minutes to remove solvent and polymerize the coating. The resulting coating was between 1 and 2 millimeters thick.

The coated diode was then mold encapsulated using the following encapsulating composition:

	Parts by weight
Diallyl phthalate prepolymer (Dapon D - FMC Corp.)	45
Glass, 4" chopped fibers	25
Clay, calcined	30
Calcium stearate	0.5
Tertiary butyl perbenzoate	1.5

The encapsulation was done by transfer molding at a ram pressure of 1,000 psi at 160°C for a cure time of 3 minutes. The encapsulated silicon diode showed no loss of electrical performance after 100 hours' immersion in boiling water.

A second silicon diode was coated as described above. This second coated diode was mold encapsulated as described above using a bisphenol A diglycidyl ether type epoxy polymer encapsulating composition (Polyset 300 available from Morton Chemical Company). The wet electrical performance of the coated-encapsulated diode was improved as compared to a similar diode that was not coated prior to encapsulation with the epoxy encapsulating composition.

#### EXAMPLE 14

Vacuum bag laminates were prepared using diallyl isophthalate prepolymer and monomer, brominated biphenyl and in one example a polyphenylene ether polymer. Woven glass cloth, type 181, was impregnated with the mixtures set forth below and allowed to dry for 48 hours at 25°C. The dry impregnated glass cloth was cut into 12 inch squares, stacked 18 plies

deep with the warp yarns parallel. The impregnated lay-up was molded by the vacuum-bag technique. Full cure was obtained after 15 minutes at 150°C.

TABLE IX

Formulation		
Diallyl isophthalate monomer	250	350
Diallyl isophthalate prepolymer	650	310
Hexabromobiphenyl	200	150
Dicumyl peroxide	30	30
Gamma methacryloxypropyl-trimethylsilane	5	5
Hydroquinone	0.3	0.3
Aluminum trihydrate	70	40
Methyl ethyl ketone	400	400
Polyphenylene ether blend ***	—	285
Physical Properties		
Resin content, weight %	29.8	35.1
Rockwell hardness (M)	120	122
Compressive strength, psi	26,400	44,500
Flexural strength, psi at 25°C	65,800	83,900
psi at 205°C	31,100	22,000
after 2 hours in boiling water	47,800	76,900
Flexural modulus, psi at 25°C	2.7 x 10 <sup>6</sup>	2.8 x 10 <sup>6</sup>
psi at 205°C	2.6 x 10 <sup>6</sup>	1.8 x 10 <sup>6</sup>
after 2 hours in boiling water	2.6 x 10 <sup>6</sup>	2.9 x 10 <sup>6</sup>
Tensile strength, psi	52,600	55,300
Shear strength, psi at 25°C	3,010	5,400
psi at 205°C	1,740	1,470
Moisture absorption, 48 hours under water at 50°C, weight %	.44	.08
Specific gravity	1.974	1.911
Dielectric constant 10 <sup>3</sup> /HZ dry	4.33	4.30
10 <sup>6</sup> /HZ dry	4.46	4.33
Burning test (ASTM D-635)	non-burning	non-burning

\*\*\* 200 parts of polyphenylene ether polymer, 57 parts of diallyl isophthalate prepolymer, 28 parts of aluminum trihydrate, ball-milled together for 12 hours.  
\*\*\* Sample contained some small voids.

Obviously, these examples can be multiplied indefinitely in view of the possible permutations and combinations of resins, polychlorinated aromatic hydrocarbons, fillers and additives known in the art to be useful in preparing molding compounds and laminates. The principle of this invention has been explained and exemplified so that it can readily be practiced by those skilled in the art. The best mode contemplated by the inventors has been set forth. Clearly, within the scope of the appended claims, the invention may be practiced by those skilled in the art having benefit of this disclosure, otherwise than as specifically described and exemplified herein.

1. A thermosetting composition comprising:

- 10 to 50 parts by weight of a polyunsaturated monomer;
- 5 to 30 parts by weight of a polychlorinated or polybrominated aromatic hydrocarbon containing at least 50% by weight chlorine or bromine and having a molecular weight of at least 200;
- 20 to 60 parts by weight of a polyphenylene ether polymer;
- 0-30 parts by weight of a polyunsaturated polymer having carbon to carbon unsaturation and
- 2-10 parts by weight per 100 parts of all of (1) plus (2) plus (3) plus (4) of a peroxide catalyst which has a 10 hour half-life in benzene at a temperature in excess of 100°C.

2. The composition of claim 1 further comprising a fluid in which the thermosetting composition is dispersed.

3. The composition of claim 1 in which the fluid is a solvent selected from the group consisting of chlorinated hydrocarbon solvents and aromatic hydrocarbon solvents.

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4. The composition of claim 1 in which the polychlorinated or polybrominated aromatic hydrocarbon is selected from the group consisting of chlorinated or brominated biphenyls, chlorinated or brominated terphenyls, trichlorobenzene, and chlorinated naphthalenes.

5. The composition of claim 1 in which the polyunsaturated monomer is selected from the group consisting of diallyl phthalate, diallyl isophthalate, diallyl chloridate, diallyl maleate, triallyl cyanurate, triallyl iso-

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cyanurate, glycol dimethylacrylate, and a divinyl benzene.

6. The composition of claim 1 in which the peroxide catalyst is selected from the group consisting of dicumyl peroxide, tertiary butyl perbenzoate, 2,5-dimethyl-2,5-di(tertiary-butylperoxy) hexyne-3 and 2,5-dimethyl-2,5-di(tertiary-butylperoxy) hexane.

7. The composition of claim 1 further comprising an inert filler selected from the group consisting of inorganic fillers, fibrous fillers, fibrous mats, woven fabric and non-woven fabric.

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August 31, 1993

Mr. Richard Desrosiers  
Environmental Laboratories, Inc.  
142 Temple Street  
New Haven, CT 06510

Subject: Review of PCDD/PCDF Analytical Data from Raymark Industries, Inc.,  
Site, MRI Project No. 3387-A

Dear Mr. Desrosiers:

Midwest Research Institute has reviewed the analytical data and background information pertaining to the Raymark Industries, Inc. We have reviewed the data with respect to assisting Environmental Laboratories, Inc. (ELI), in assessing the origination of PCDD/PCDF residues on site. ELI has provided to MRI a number of items which include historical data and perspective of production efforts at the Raymark Industries site, data tables (hardcopy and Lotus 1-2-3 format) for the targeted analytes, and hardcopy results for each of the samples for the PCDDs and PCDFs to allow review of the chromatographic patterns.

In approaching this effort, MRI has reviewed all information supplied by ELI pertaining to the site history, confirmed the calculations for the determination of toxicity equivalence factors, reviewed the chromatographic patterns, reviewed available literature, and contacted a number of other scientists with experience in the area of PCDDs, PCDFs, and PCBs.

Based on the data received to date, MRI has not found conclusive evidence for the source of origination of the PCDDs and PCDFs. The chromatograms for the PCDDs and PCDFs detected in the samples do not reflect a common fingerprint to any single known source of these compounds. The range and pattern of PCDDs and PCDFs indicates that multiple sources may contribute to the overall background at the site. Background contribution may arise from general atmospheric transport, and residues from the Aroclor 1268 and the phenoxy herbicides (2,4-D, 2,4,5-T and 2,4,5-TP) detected in the field samples. MRI has included information regarding these potential sources of PCDD and PCDF sources in this report.

Our review of the literature and personal contacts with experts in the field have not yielded any additional information on the residue levels of PCDDs and PCDFs in Aroclor 1268. Without the information on the residual levels in the Aroclor 1268, it



must be considered that the PCDDs and PCDFs may have originated from other sources outside the manufacturing processes and procedures.

## **PCDD/PCDF BACKGROUND LEVELS IN THE STATE OF CONNECTICUT**

MRI has previously worked with the State of Connecticut to determine background levels of PCDDs and PCDFs in soil and sediment from several locations throughout the state to develop a baseline and evaluation of the impact of municipal waste incineration. These data have demonstrated a background of PCDDs and PCDFs in the state. The table in Attachment 1 presents a summary of data from sediments from sites located in Bridgeport, Connecticut. The data base from which this information was extracted contains data on up to 971 samples (77 soils, 344 sediments, and 550 fish) collected from 1987 to 1990 from areas within or adjacent to nine different municipalities. MRI has contacted the Connecticut Department of Environmental Protection to obtain approval for the release of our final report, "Multivariate Statistical Analysis of Dioxin and Furan Levels in Fish, Sediment and Soil Collected near Resource Recovery Facilities" (Final Report for PSA Contract 91-1526). MRI has previously provided this report to your office under separate cover.

A comparison of the general Connecticut background dioxins and furans and the ELI results illustrates the difference between these data. The first figure in Attachment 1 f shows the average level dioxins and furans from the Raymark Industries Site (referred to as ELI Dioxins/Furans) expressed as a percent of the total dioxin and furan level. In this figure, the average level of OCDD is more than 40% of the total of all dioxins and furans. Also the average level of furans in these samples is nearly 20% for HxCDF. The second figure shows a similar treatment of the samples which MRI has analyzed from Bridgeport, Connecticut. In these samples, the average level of OCDD is over 60% of the total of all dioxins and furans. This figure also illustrates the fact that the overall level of furans as a percentage of the total response in the Bridgeport results is lower than in the ELI data. This higher level of furans may be result of the presence of Aroclor 1268.

## **PCDDs/PCDFs FROM AROCLORS**

As per our past telephone discussions, the Aroclor 1268 may be an important contributor to the levels of PCDFs observed in the sample. The chromatographic patterns observed for the TCDFs, PeCDFs, and HxCDFs reflect some similarities with other Aroclors that have been studied in round robin investigations by the Electric Power Research Institute (EPRI) and in investigations of thermal degradation products from dielectric fluids conducted by MRI. Unfortunately, the samples in both cases focused on Aroclors that were typically used in transformers. Aroclor 1268 was not used for the same purpose as it is a material of higher molecular weight and hence

was not suitable as a fluid. At this time we have not been able to identify any research efforts or investigations pertaining to the levels of PCDFs in Aroclor 1268.

Some examples of chromatographic patterns for Aroclor 1254 that has been intentionally thermally degraded are presented in the series of Figures presented as attachments (Attachment 2) to this letter. These data were generated by MRI as an investigation of the thermal degradation of dielectric fluids ("Thermal Degradation Products from Dielectric Fluids," M. D. Erickson, et al., EPA-560/5-84-009, December 1984). The chromatographic patterns for the tetra, penta, and hexachlorinated dibenzofurans demonstrate some similarities with the data in the soil samples showing positive results. The other striking factor is the relative difference in the PCDF to PCDD ratios. The PCDFs are greater than the PCDDs by factors of up to 20 or more in some of the field samples from the Raymark Industries site.

Additional data on PCDFs and PCDDs in Aroclor mixtures were obtained from the EPRI report on PCDFs and PCDDs in Utility Transformers and Capacitors (EPRI EL/EA-4858, December 1986). MRI has spoken with the EPRI project officer on that work, Mr. Gil Addis, and two of the principal investigators from IIT Research Institute and Battelle Columbus Laboratories. The effort on this work focused on Aroclors used within the utility industry. Some of the pertinent data from that effort follow.

**Aroclor 1016.** Aroclor 1016 did not exhibit any PCDDs or PCDFs in the samples analyzed in this study. Approximate detection limits appear to be on the order of 10 ng/g of sample.

**Aroclor 1242.** Aroclor 1242 demonstrated responses to the TCDF, PeCDF, HxCDF, and OCDF. The PCDDs were not detected in this particular matrix. The total PCDF values ranged from approximately 130 ng/g for the OCDF up to 870 ng/g for the TCDFs. The TCDFs were the predominant responses for this mixture.

**Aroclor 1260.** Aroclor 1260 also demonstrated responses to the PCDFs, but not the PCDDs. The PCDF homolog concentrations were as follows OCDF (4150 ng/g) > HpCDF (1985 ng/g) > HxCDF (1400 ng/g) > PeCDF (1040 ng/g) > TCDF (600 ng/g).

**Askarel Load Center Network Transformer.** This sample contained 70% Aroclor 1260, 29% trichlorobenzenes, and 1% tetrachlorobenzene from a transformer of over 30 years' service. PCDDs were not detected in the sample to any significant extent. The data demonstrated a different order of predominance of the PCDFs: HxCDF (2300 ng/g) > HpCDF (1590 ng/g) > PeCDF (1530 ng/g) > TCDF (760 ng/g) > OCDF (560 ng/g).

Some effort was made to correlate the Aroclor 1268 concentrations to the total PCDDs and PCDFs using the I-TEF values. However, a graphical representation of the data

did not result in any clear or coincident relationship of these values. The correlation of data may be affected by the homogeneity of the matrices analyzed.

Using the information obtained from the EPRI round robin study on various dielectric fluids an estimate of the contaminant levels of PCDFs in the soil samples resulting from the Aroclor 1268 was developed as follows.

If one assumes that the TCDF concentration of 8.1 ng/g in field sample 92-1949 is attributed to the Aroclor 1268 (determined at ~ 30 µg/g of soil), the contaminant level of the TCDF in the Aroclor would originally be 270 µg/g or

$$(8.1 \times 10^{-9} / 30 \times 10^{-6}) = 0.27 \times 10^{-3} \text{ g/g or } 270 \text{ µg/g}$$

This contaminant level is considerably higher than found in any other PCB mixture reported in the EPRI study.

Some data on the thermal degradation products of PCBs indicate that conversion efficiencies of up to 2% can be achieved (M.D.Erickson, et al, 1984). However, this data was developed from laboratory experiments that were intentionally optimized to produce PCDDs and PCDFs based on the presence of oxygen, temperature and residence time within a combustion or pyrolysis zone.

#### PCDDS/PCDFS FROM PHENOXY HERBICIDES

Our review of the data tables indicates some potential association of the PCDD/PCDF levels with the presence of phenoxy herbicides in a number of the samples. However, as with the PCB data, there does not appear to be a strict statistical correlation with any one of the herbicide residues pertaining to the overall TCDD TEF values. Attachment 2 to this letter summarizes available published literature through approximately 1984 ("Guidelines for Determination of Halogenated Dibenzo-*p*-dioxins and Dibenzofurans in Commercial Products," EPA-560/5-87/007, September 1987).

Other sources of information in commercial products, particularly the journal *Chemosphere*, have been reviewed. While additional information has been published regarding PCDD/PCDF levels, the information is consistent with citations from the late 1970s and early 1980s demonstrating the major contaminants are PCDDs. In many products, particularly 2,4,5-T, the primary contaminant is 2,3,7,8-TCDD which was not present in the field samples.

## TOXICITY EQUIVALENTS

Toxic equivalence is an attempt to estimate the total cumulative toxic effect of the variety of dioxin and furan isomers which are detected in a sample. Using this approach, the quantitated result for the various dioxins and furans can be reduced to a single number (TEF). At best, this cumulative dioxin/furan result is an estimate and it should be applied with caution. The purpose of this approach is to provide a basis for comparison of the dioxin/furan results from different samples. As such it has limited usefulness when comparing measured results variable chromatographic patterns or fingerprints of individual isomers from different samples. This technique has very little value when it is calculated from estimated results such as detection limits.

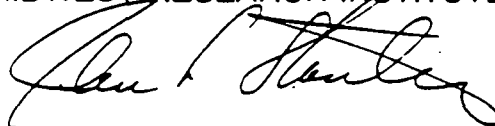
The majority of the samples from ELI reviewed for this report had no detectable dioxins other than OCDD (the least toxic congener). If these congeners are not detected, they would not be expected to contribute to the overall estimate of toxicity as calculated using the TEF formula. As per MRI's previous recommendations, if detection limit estimates based on method 8280 are used to estimate the total TEF, ELI should evaluate the benefit of the method 8290 HRMS, which provides significantly lower detection levels.

However, there are several approaches to the use of detection limits in the calculation of TEF. In the calculation of TEF, a congener which is not detected can have a value of zero, or half the analytical detection limit or the detection limit. When the detection limit is used in the calculation of TEF, a comparison of results or a regulatory decision can be influenced by the sensitivity of the analytical method and not by the actual amount of dioxin or furan in the sample. Therefore, the approach used for non-detects can have a significant impact on the estimated TEF value.

If there are any questions regarding this information, please call. A hardcopy of this letter and contents will be forwarded under separate cover.

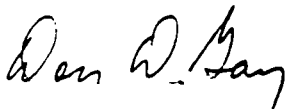
Sincerely,

MIDWEST RESEARCH INSTITUTE



John S. Stanley, Ph.D.  
Section Head  
Analytical Chemistry

Approved:



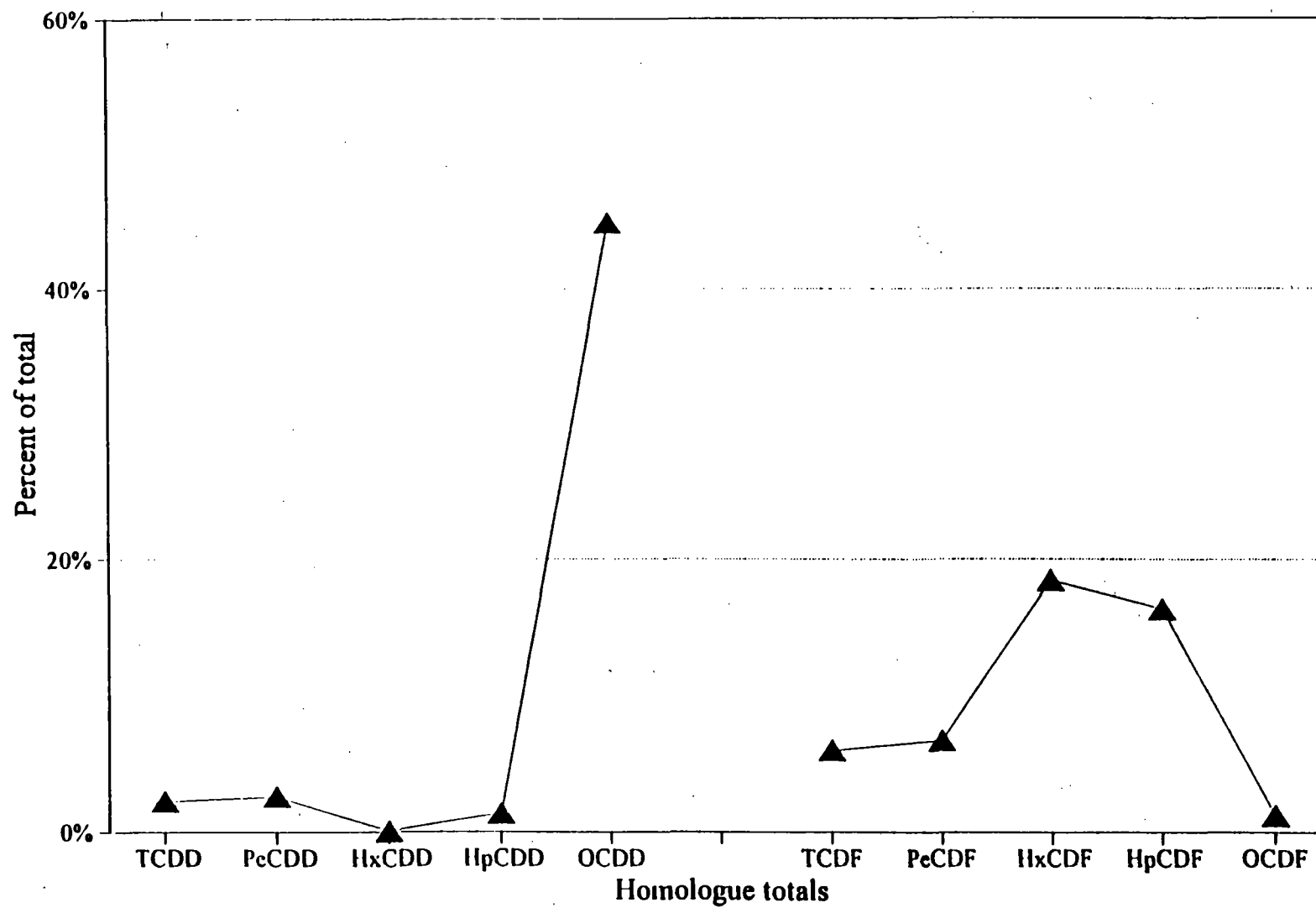
Don D. Gay, Ph.D.  
Director  
Chemical Sciences Department

## **ATTACHMENT 1**

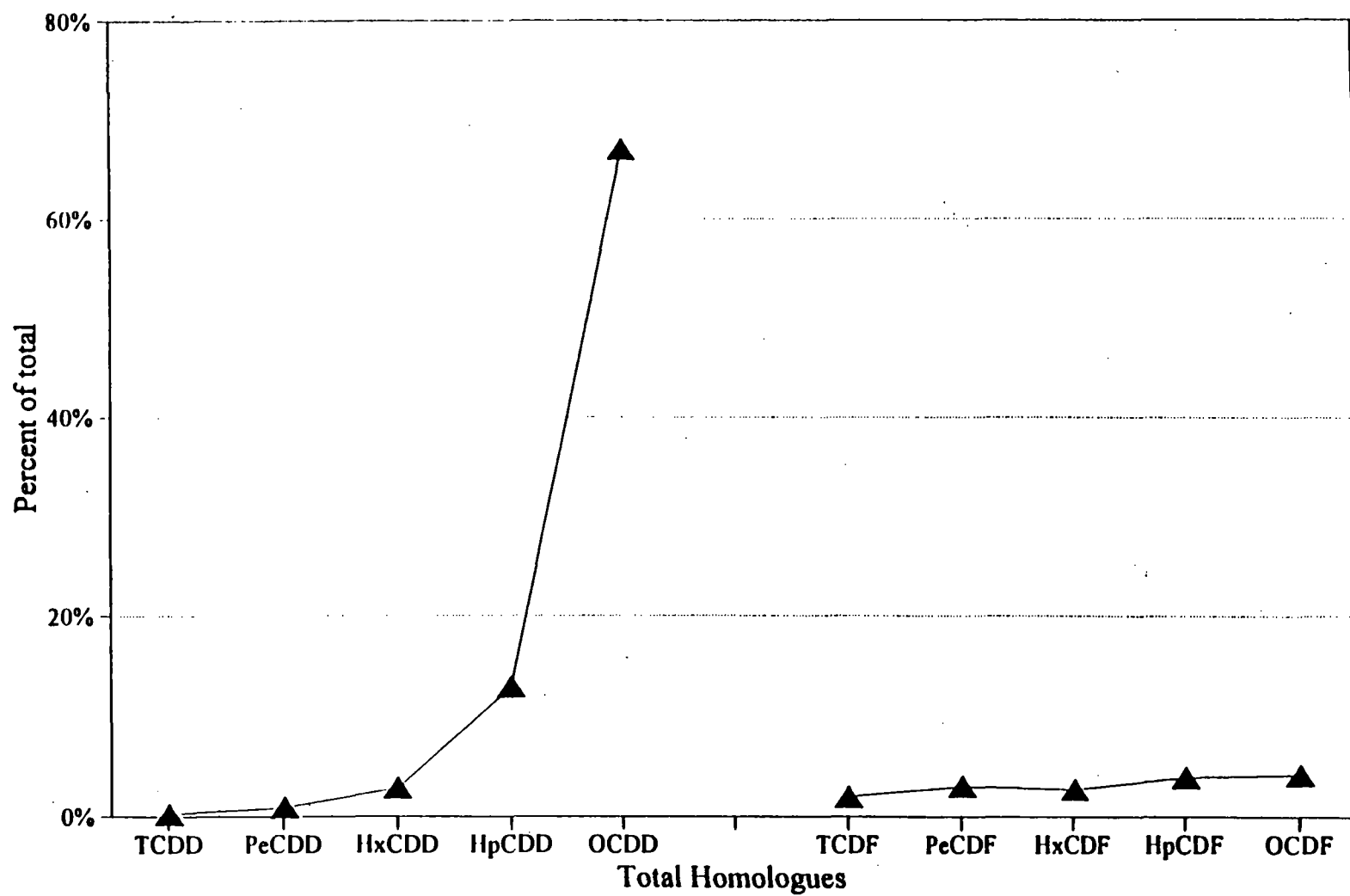
### **Background Levels of PCDDs/PCDFs in the Bridgeport, Connecticut, Area**

# ELI Dioxins/Furans

Totals



# Bridgeport Dioxins/Furans Totals



BRIDGEPORT SUMMARY (pg/g, dry weight basis)

Analyte	Lab ID	30264	30265	30266	30267	30268	30271	30272	30273
TCDF		238	929	404	29.5	218	59.1	39.5	68
TCDD		49.9	288	115	5.63	38.5	5.57	5.86	11.2
PeCDF		236	1330	470	30.9	405	90.5	56.6	105.4
PeCDD		168	718	151	5.96	79.4	22.4	10.8	20.2
HxCDF		373	1610	370	24.5	365	108	57.6	97.2
HxCDD		430	1670	434	25.9	380	118	84.4	146
HpCDF		552	1990	574	40.6	635	214	160	234
HpCDD		1843	5587	1340	95.6	2039	599	323	476
2378TCDF		47.7	206	65.8	5.08	39.7	15.6	10.8	18.3
2378TCDD		13.9	51.5	6.23	ND( .193 mpc)	ND( 1.24 mpc)	0.762	ND( .412)	ND( .383 mpc)
12378PeCDF	ND( 22.7 mpc)		82.9	22	1.24	13.4	5.16	3.95	6.95
23478PeCDF		20.1	96.9	27.2	1.77	18.3	6.51	4.88	8.42
12378PeCDD		19.6	87.8	13.3	0.78	7.38	2.77	1.4	2.25
123478HxCDF		59.2	277	46.4	3.04	25.2	11.6	5.95	9.77
123678HxCDF		18.4	94.4	18.1	1.26	14.5	5.15	2.56	4.33
234678HxCDF		28.6	134	28.9	2.24	26.7	8.49	4.34	7.71
123789HxCDF	ND( .488)		ND( 9.13 cdl)	ND( 1.72)	ND( .578 mpc)	ND( .647)	ND( .325)	ND( .203)	ND( .964 mpc)
123478HxCDD	ND( 10.3 mpc)		42	11.1	ND( .581 mpc)	10.4	2.25	2.08	2.98
123678HxCDD		41.4	150	35.6	1.99	36.4	8.62	5.99	8.82
123789HxCDD		36.3	146	32.8	2.15	30.6	8.15	5.33	8.5
1234678HpCDF		253	1100	273	18.7	252	93	67.6	101
1234789HpCDF		21	70.5	17	ND( 1.19 mpc)	15.1	3.92	2.36	4.04
1234678HpCDD		773	2330	565	42	831	187	122	192
12346789OCDF		478	1480	665	70.5	772	300	259	380
12346789OCDD		7160	16700	5210	364	6650	1720	1220	1860
I-TE		86.7	356	80.1	5.50	60.6	20.3	13.6	21.9
TOC (mg/kg)		31,900	87,500	43,700	5,120	50,100	34,000	23,000	38,000
Grain Size:									
Sand (%)		45	55	28	82	34	74	60	15
Silt (%)		45	36	58	8	52	21	35	75
Clay(%)		10	9	14	10	14	5	2	10



**BRIDGEPORT SUMMARY (pg/g, dry weight basis)**

Analyte	Lab ID	30274	30275	30276	30277	30278	30279	30280	30281
TCDF		70	64.1	60	56.8	14.6	26.6	60.6	90.6
TCDD		9.12	10.9	1.08	1.61	2.7	2.86	4.86	10.8
PeCDF		150	93.4	103	71.9	15.4	35.3	71.9	110
PeCDD		43.9	37.9	30.9	23.9	4.91	7.83	20.7	34.6
HxCDF		354	78	122	89.6	4.11	22.2	22	52
HxCDD		365	182	140	102	13.4	21.6	36.4	45.6
HpCDF		743	261	155	121	9.24	16.8	24.9	30
HpCDD		2540	757	723	557	39.7	85.5	89.3	126
2378TCDF		22.2	17.2	17.4	14.7	4.2	6.55	20.3	28.4
2378TCDD		2.18	1.66	ND( .483 mpc)	ND( .736)	ND( .631)	ND( .459)	ND( .754)	ND( .434 mpc)
12378PeCDF		12.3	5.87	4.92	4.2	0.98	1.78	4.38	5.41
23478PeCDF		9.02	5.69	10.1	6.07	1.57	2.71	5.05	7.89
12378PeCDD		11.1	5.94	ND( 3.92 mpc)	3.2	ND( .299 mpc)	0.718	1.28	2.01
123478HxCDF		30.7	14.6	11.5	9.77	1.78	3.22	5.58	7.3
123678HxCDF		17.5	7.01	5.62	4	0.78	0.211	1.91	3.55
234678HxCDF		25.6	11.2	9.08	7.35	1.68	3.07	4.55	5.74
123789HxCDF		ND( .592)	ND( .259)	ND( .546)	ND( .355)	ND( .0844 mpc)	0.537	ND( .303)	ND( .51 mpc)
123478HxCDD		12.8	ND( 1.96 mpc)	5.93	ND( 3.23 mpc)	ND( .146 mpc)	1.08	1.22	ND( .727)
123678HxCDD		58.7	21.2	13.6	14.6	ND( .465 mpc)	1.4	2.56	2.16
123789HxCDD		47.3	16.2	13.2	11.5	1.47	2.5	3.57	4.34
1234678HpCDF		318	109	69.6	57.3	4.74	9.73	15.4	17.7
1234789HpCDF		22.2	4.85	5.28	4.29	0.757	0.999	ND( 1.53 mpc)	1.72
1234678HpCDD		1310	319	338	260	17.7	35.1	37.2	53.4
12346789OCDF		603	236	120	98	9.44	10.7	36.7	18.5
12346789OCDD		10600	2580	4530	3560	519	1210	799	1970
I-TE		67.1	27.1	28.5	22.0	4.30	7.42	12.1	17.8
TOC (mg/kg)		130,000	93,000	100,000	88,000	35,000	150,000	220,000	240,000
Grain Size:									
Sand (%)		45	46	28	64	90	99	94	90
Silt (%)		53	53	72	36	8	1	6	10
Clay(%)		2	1	0	0	0	0	0	0

BRIDGEPORT SUMMARY (pg/g, dry weight basis)

Analyte	Lab ID	30282	30283	32586	32587	32588	32589	32590	32591
TCDF		2.04	1.58	40.9	272	8.67	37.7	57.1	159
TCDD		ND( .255)	ND( .233)	4.02	46.9	0.516	2.97	3.13	13.8
PeCDF		8.6	6.83	73.5	399	17.2	91.2	173	479
PeCDD		2.11	ND( .338)	27.6	121	6.03	23.5	29.3	55
HxCDF		10.9	7.57	73.7	213	15.6	64.9	140	370
HxCDD		12.9	4.93	59.3	233	23.6	48.8	62.4	181
HpCDF		17.5	13.1	80.2	255	16.9	40	149	198
HpCDD		65.9	44.1	303	909	129	265	286	846
2378TCDF		0.943	ND( .323 mpc)	8.87	57.4	2.21	8.01	11.1	18.2
2378TCDD		ND( .255)	ND( .233)	ND( .298 mpc)	1.74	ND( .43)	ND( .386 mpc)	ND( .343 mpc)	0.955
12378PeCDF		0.493	ND( .118 mpc)	3.55	23.4	0.798	3.22	4.51	6.64
23478PeCDF		0.535	0.324	4.74	25.8	ND( 1.35 mpc)	4.54	6.63	10.5
12378PeCDD		0.366	ND( .0952 mpc)	2	6.65	0.614	1.36	2.07	5.53
123478HxCDF		0.952	ND( .314 mpc)	7.9	ND( 61.3 mpc)	2.76	7.13	11.6	17.5
123678HxCDF		ND( .567 mpc)	0.274	3.9	ND( 19.2 mpc)	1.37	3.19	5.43	13.6
234678HxCDF		0.859	0.679	5.77	30.1	1.92	5.33	9.63	25.8
123789HxCDF		ND( .0677 mpc)	ND( .0597 mpc)	ND( .35)	ND( 1.02)	ND( .18 mpc)	ND( 2.36 cdl)	ND( .565)	ND( 1.68)
123478HxCDD		ND( .223 mpc)	ND( .0191 mpc)	2.04	ND( 1.33)	0.824	1.86	ND( 2.34 mpc)	ND( 2.05 mpc)
123678HxCDD		2.05	1.3	5.8	26.9	1.9	3.17	6.84	23.5
123789HxCDD		1.02	0.744	5.36	20.6	2.22	4.28	ND( 5.92 mpc)	19.1
1234678HpCDF		6.79	5.13	37.9	138	9.11	20.5	71.6	85.8
1234789HpCDF		0.562	ND( .437 mpc)	2.16	14.3	0.761	1.88	4.87	6.62
1234678HpCDD		33.4	23.6	142	377	53.6	100	137	394
12346789OCDF		15.5	12.1	48.9	86.2	13.1	45	110	142
12346789OCDD		448	436	3420	4790	3130	4450	2520	6150
I-TE		2.87	2.04	15.8	60.3	8.84	15.2	18.2	38.2
TOC (mg/kg)		3,600	18,000	21,000	75,000	84,000	84,000	53,000	74,000
Grain Size:									
Sand (%)		90	100	53	- (I)	- (I)	44	56	48
Silt (%)		1	0	41	-	-	50	42	48
Clay (%)		0	0	6	-	-	6	2	4

**BRIDGEPORT SUMMARY (pg/g, dry weight basis)**

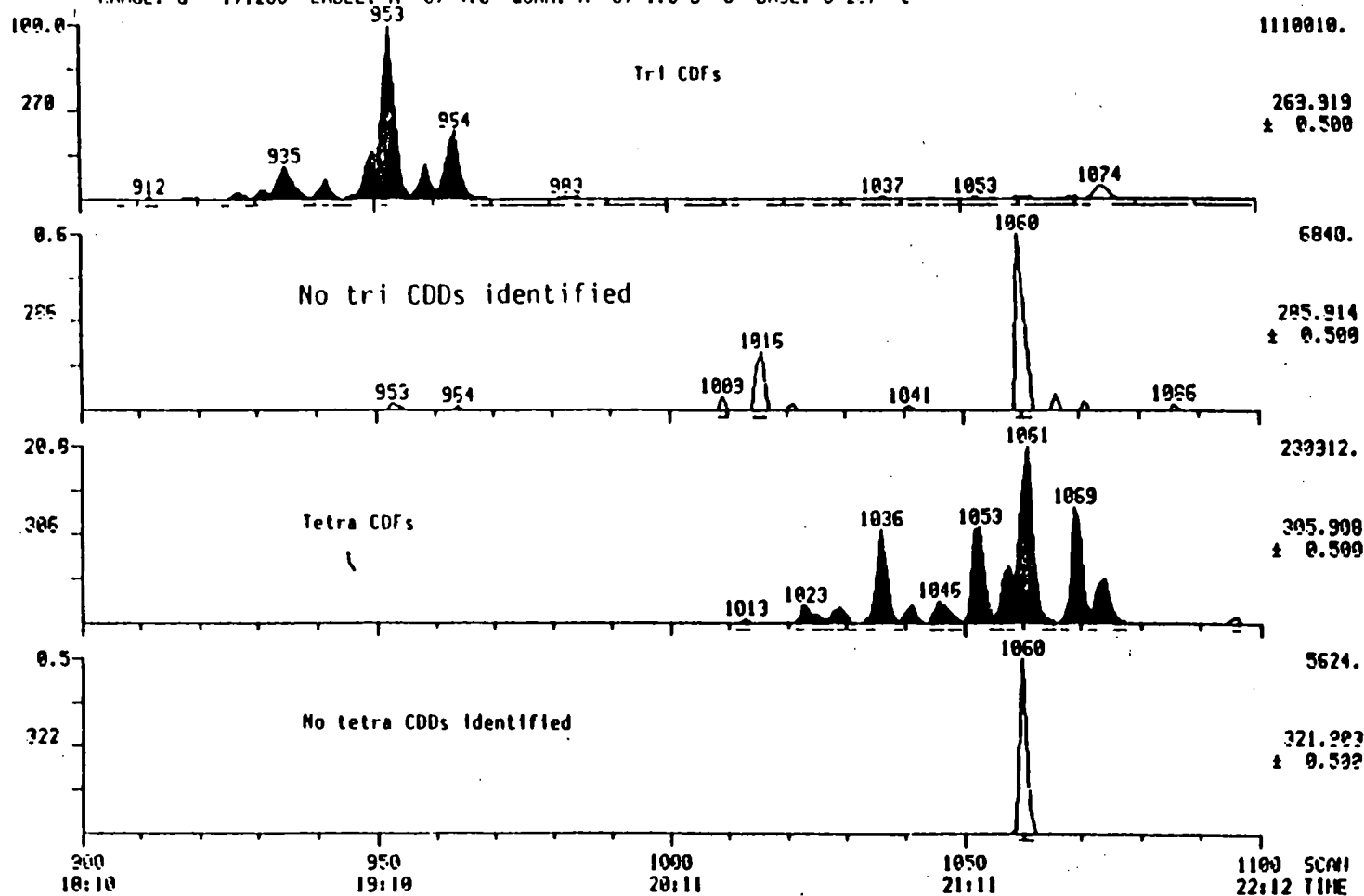
Analyte	Lab ID	32592	31460		
TCDF		101	151	ND(0.5);	1.58 - 929
TCDD		8.41	29.5	ND(0.5);	0.516 - 288
PeCDF		211	119	ND(0.5);	6.83 - 1330
PeCDD		41.4	166	ND(0.5);	2.11 - 718
HxCDF		143	524	ND(1.25);	4.11 - 1610
HxCDD		86.6	525	ND(1.25);	4.93 - 1670
HpCDF		97.4	745	ND(1.25);	9.24 - 1990
HpCDD		386	3610	ND(1.25);	39.7 - 5587
2378TCDF		25.3	44	ND(0.5);	0.943 - 208
2378TCDD	ND( .878 mpc)		12.8	ND(0.5);	0.762 - 51.5
12378PeCDF	11.1	ND( 25.8 mpc)		ND(0.5);	0.493 - 82.9
23478PeCDF	9.1	7.63		ND(0.5);	0.324 - 96.9
12378PeCDD	3	19.9		ND(0.5);	0.368 - 87.8
123478HxCDF	16.4	72.4		ND(1.25);	0.952 - 277
123678HxCDF	6.95	28.5		ND(1.25);	0.211 - 94.4
234678HxCDF	11.8	41.8		ND(1.25);	0.679 - 134
123789HxCDF	ND( .297)	1.84		ND(1.25);	0.537 - 1.84
123478HxCDD	ND( .969 mpc)	15.5		ND(1.25);	0.824 - 42
123678HxCDD	9.66	58.4		ND(1.25);	1.300 - 150
123789HxCDD	8.16	42.9		ND(1.25);	0.744 - 146
1234678HpCDF	49.7	317		ND(1.25);	4.74 - 1100
1234789HpCDF	3.6	23.5		ND(1.25);	0.562 - 70.5
1234678HpCDD	168	1560		ND(1.25);	17.7 - 2330
12346789OCDF	53.9	638		ND(2.5);	9.440 - 1480
12346789OCDD	3850	> 9700		ND(2.5);	364 - 16700
I-TE		26.1	87.1	0.83 (I);	2.040 - 356
TOC (mg/kg)		71,000	49,500	ND(1,040)	3600 - 240000
Grain Size:					
Sand (%)		71	47	-	15 - 100
Silt (%)		28	43	-	0 - 75
Clay (%)		0	10	-	0 - 14

## **ATTACHMENT 2**

### **Example Chromatograms from Thermally Degraded PCB Mixtures**

**(Taken from EPA 560/5-84-009, December 1984,  
Thermal Degradation Products from Dielectric Fluids)**

MID MASS CHROMATOGRAMS DATA: 9201127P6 #1 SCANS 900 TO 1100  
 06/27/84 11:27:00 CALI: MID250M21 #2  
 SAMPLE: 8201A23-RUN 8-22-52-S500 TUL INJ  
 COND: 1 -2000EUV 70EV IIIA DB5-30M-80-2M-320-10/  
 RANGE: G 1.1200 LABEL: N 0. 4.0 QUAN: A 0. 1.0 J 0 BASE: U 23. 3



Tri and tetra CDFs and CDDs in sample 8-22-52-S500,  
 silicone oil at 500 µg/g PCB.

MID MASS CHROMATOGRAMS

DATA: 820112X4 #1

SCANS 1650 TO 1390

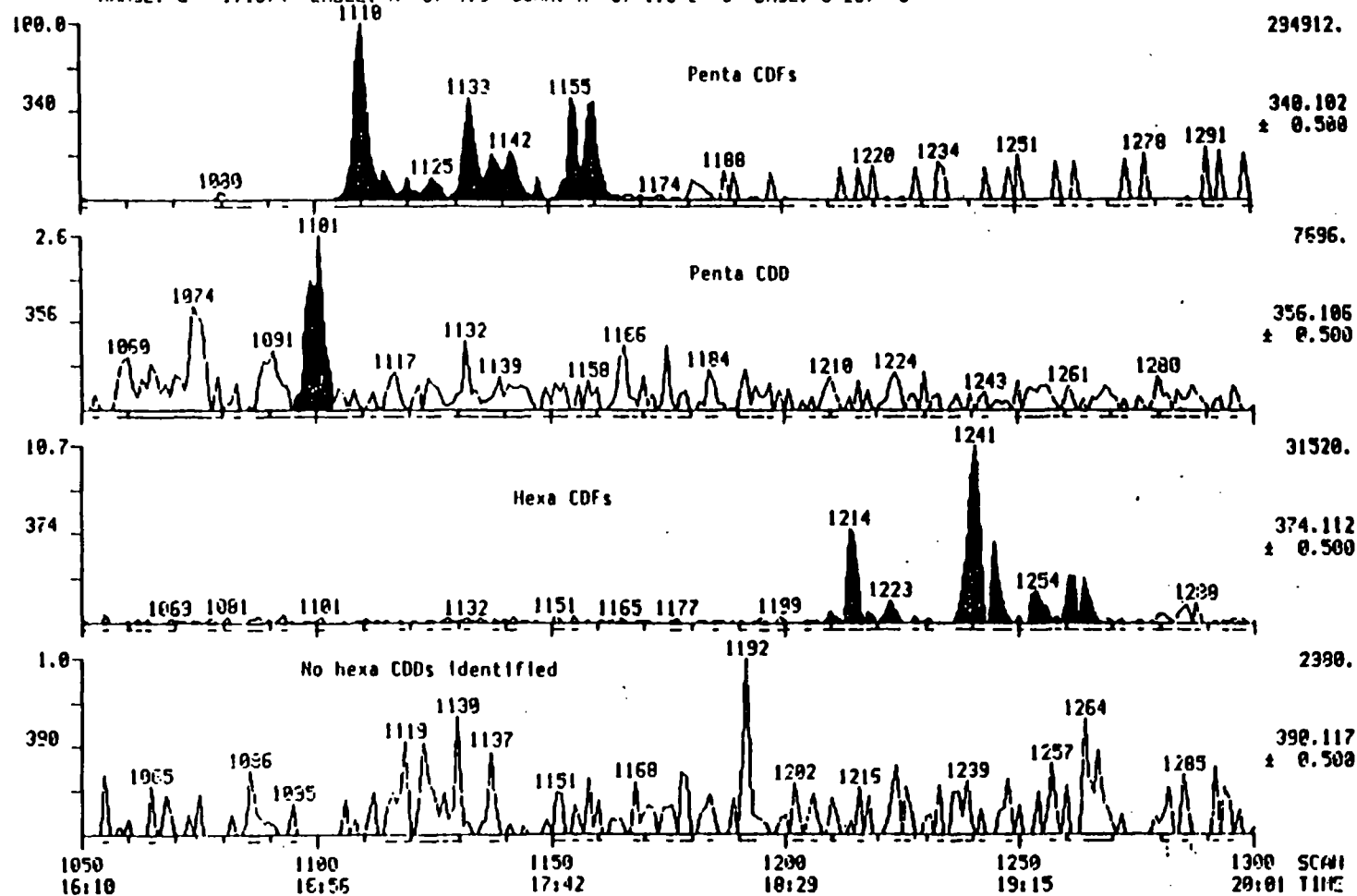
09/12/84 17:16:00

CAL: 110315112X1 #3

SAMPLE: 8-22-52-S500 1UL PCDD/F/BN CL5-6

CONV.S: 1 MG=3.5 70EV BC=3 DB5-15M 100-IN-325-10/ 45 SEC SPLTL

RANGE: G 1.1574 LABEL: N 0. 4.0 QUAN: A 0. 1.0 J 0 BASE: U 20. 3



Penta and hexa CDFs and CDDs in sample 8-22-52-S500, silicone oil at 500 µg/g PCB.

MID MASS CHROMATOGRAMS

DATA: 820115R5 N1

SCANS 850 TO 1040

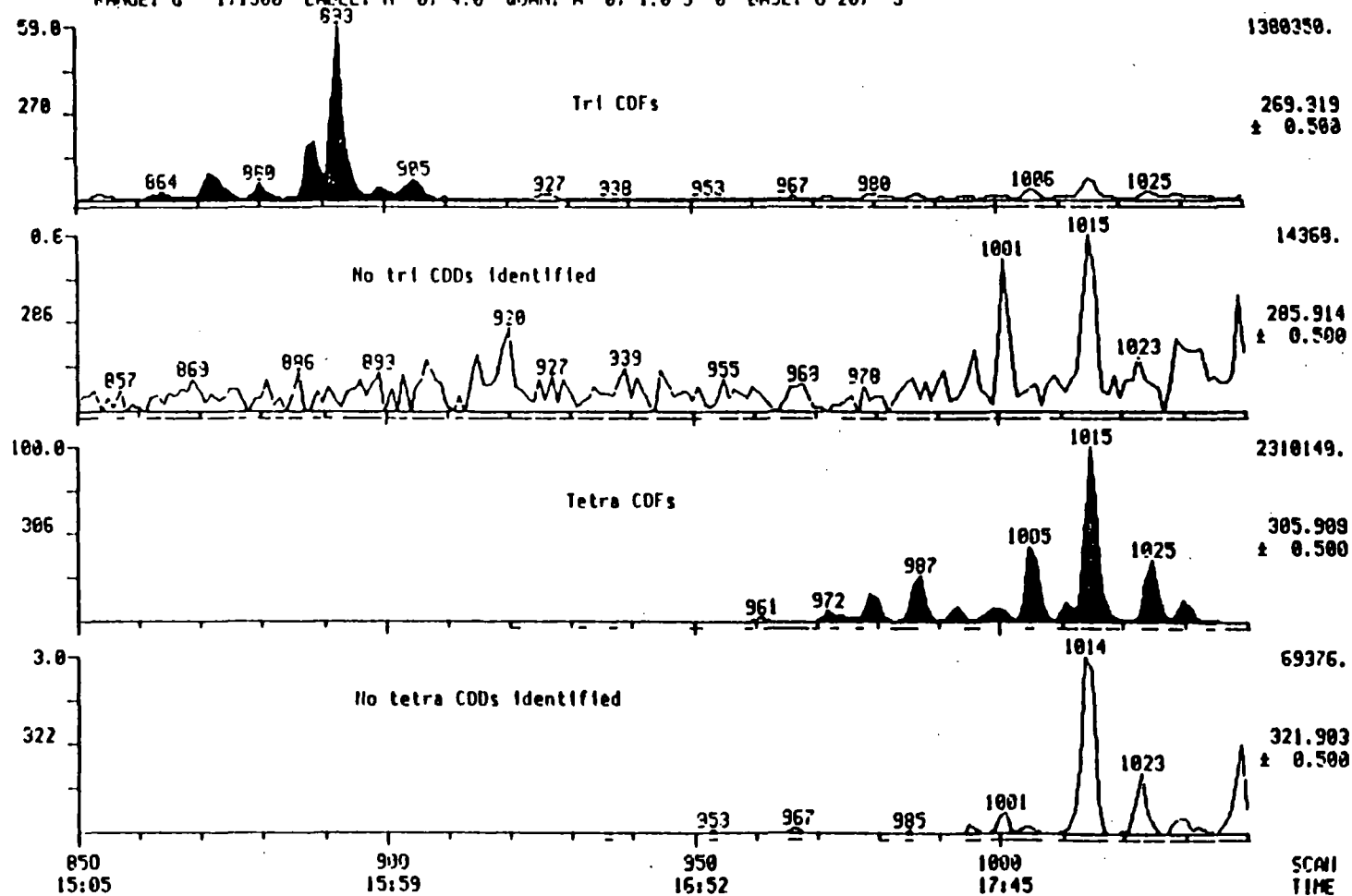
03/15/84 16:34:00

CAL: MID250121 N2

SAMPLE: 8201A23 RUN 8-30-62 ASKL 1/100 DIL 100

COND: -2200EMV 78EV 1MA DB5-3PM 100-111-329-10

RANGE: C 1.1300 LABEL: H 0. 4.0 QUAN: A 0. 1.0 J 0 BASE: U 20. 3



Tri and tetra CDFs and CDDs in sample 8-30-62-ASKL,  
Askarel sample.

MID MASS CHROMATOGRAMS

DATA: 820115R1 #1

SCANS 1200 TO 1500

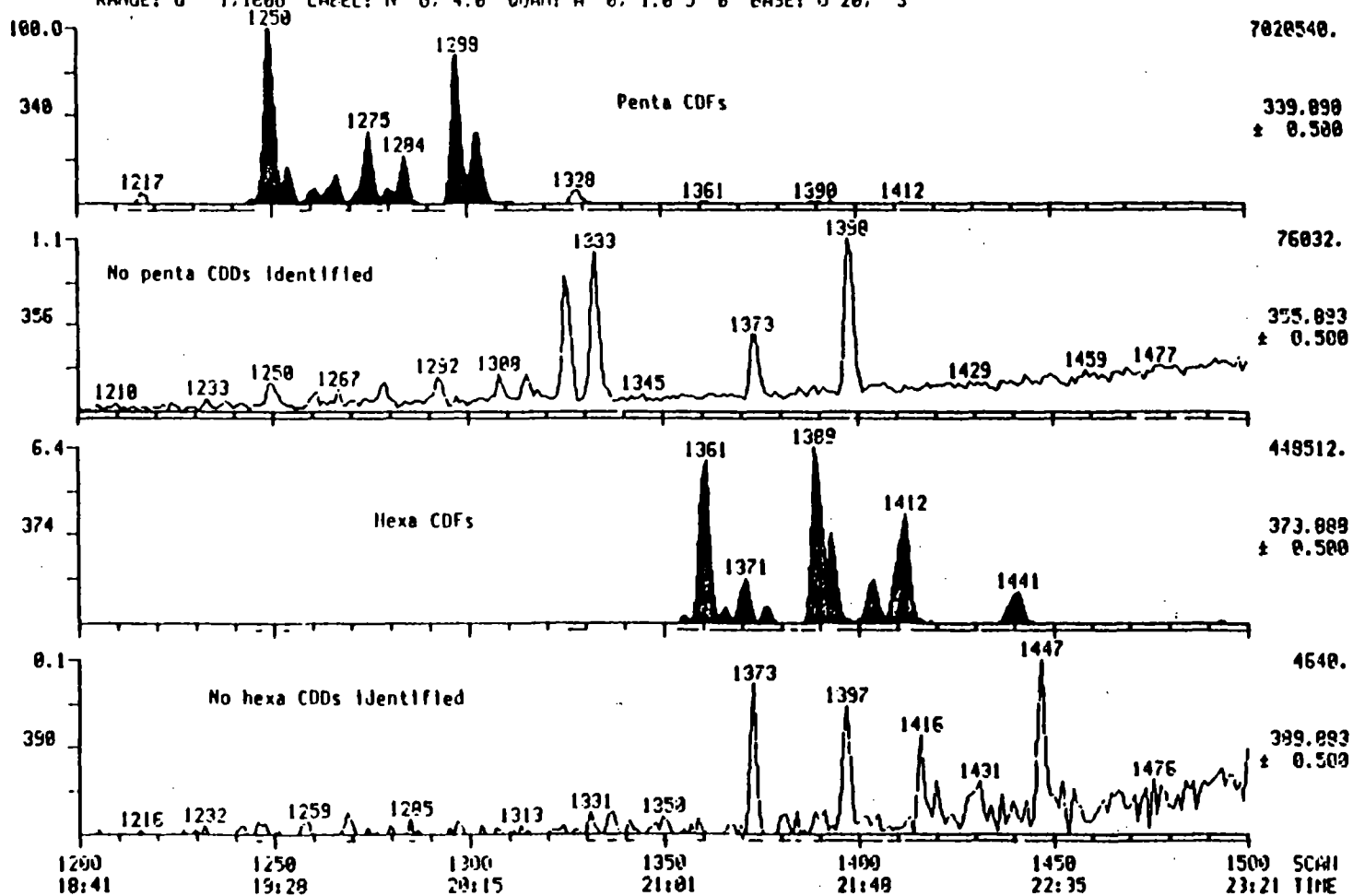
09/15/84 0:35:00

CAL: MID315114R1 #3

SAMPLE: 8-30-62-ASKL 1/1000IL FCOO/F/BFI IUL CL5-6

COMPS.: -2200ENV 70EV INA DB5-3M 100-1H-320-10/

RANGE: G 1.1E00 LABEL: H 0. 4.0 DIAM: A 0. 1.0 J 0 BASE: U 20, 3



Penta and hexa CDFs and CDDs in sample 8-30-62-ASKL,  
Askarel sample.



MID MASS CHROMATOGRAMS

DATA: 8201111R12 #1

SCANS 580 TO 850

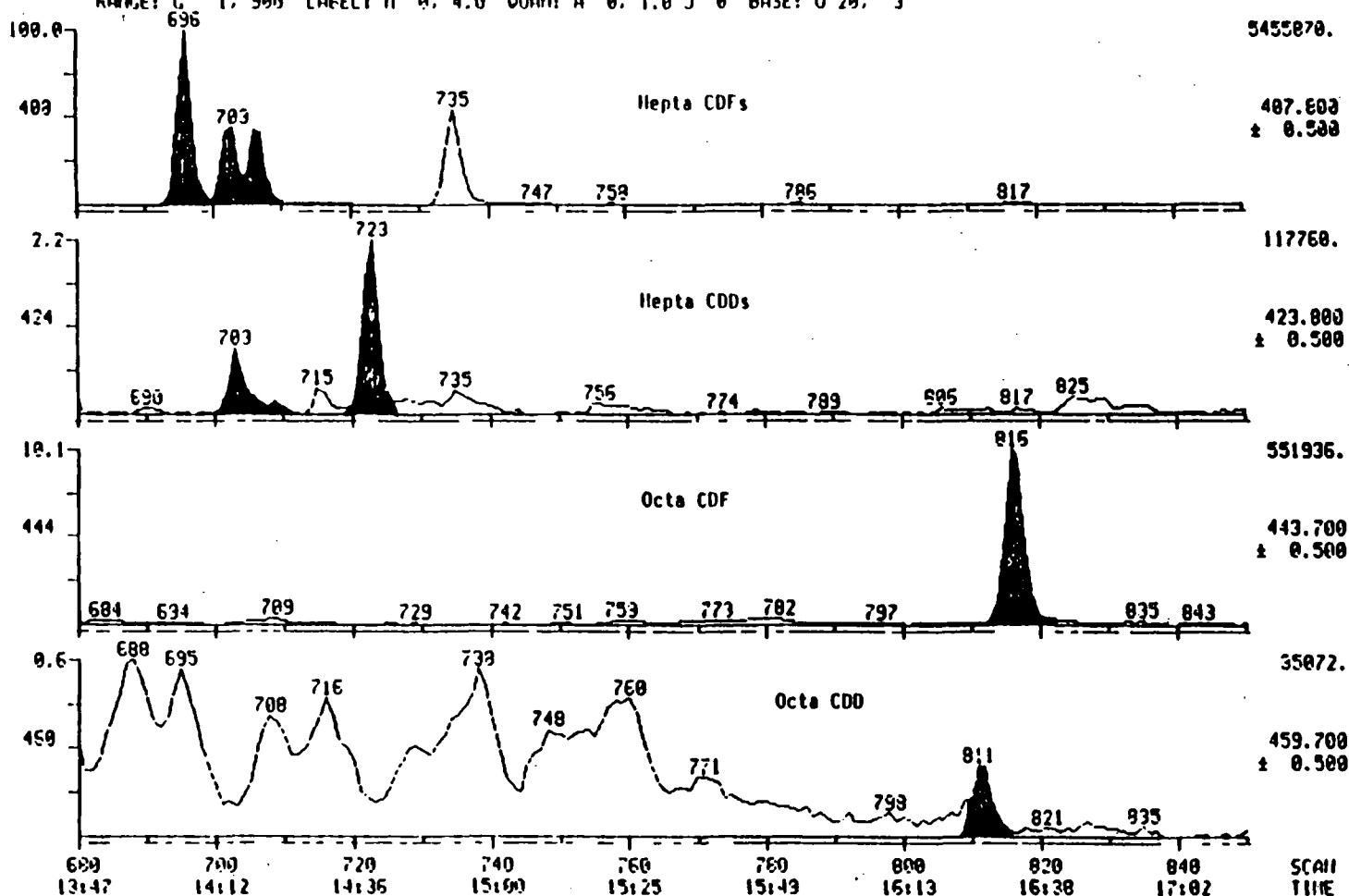
03/11/84 16:50:00

CAL1: 1110390111 #2

SAMPLE: 8-30-62-ASKL (D/F) 1UL FCOO/F/BPH CL7-8

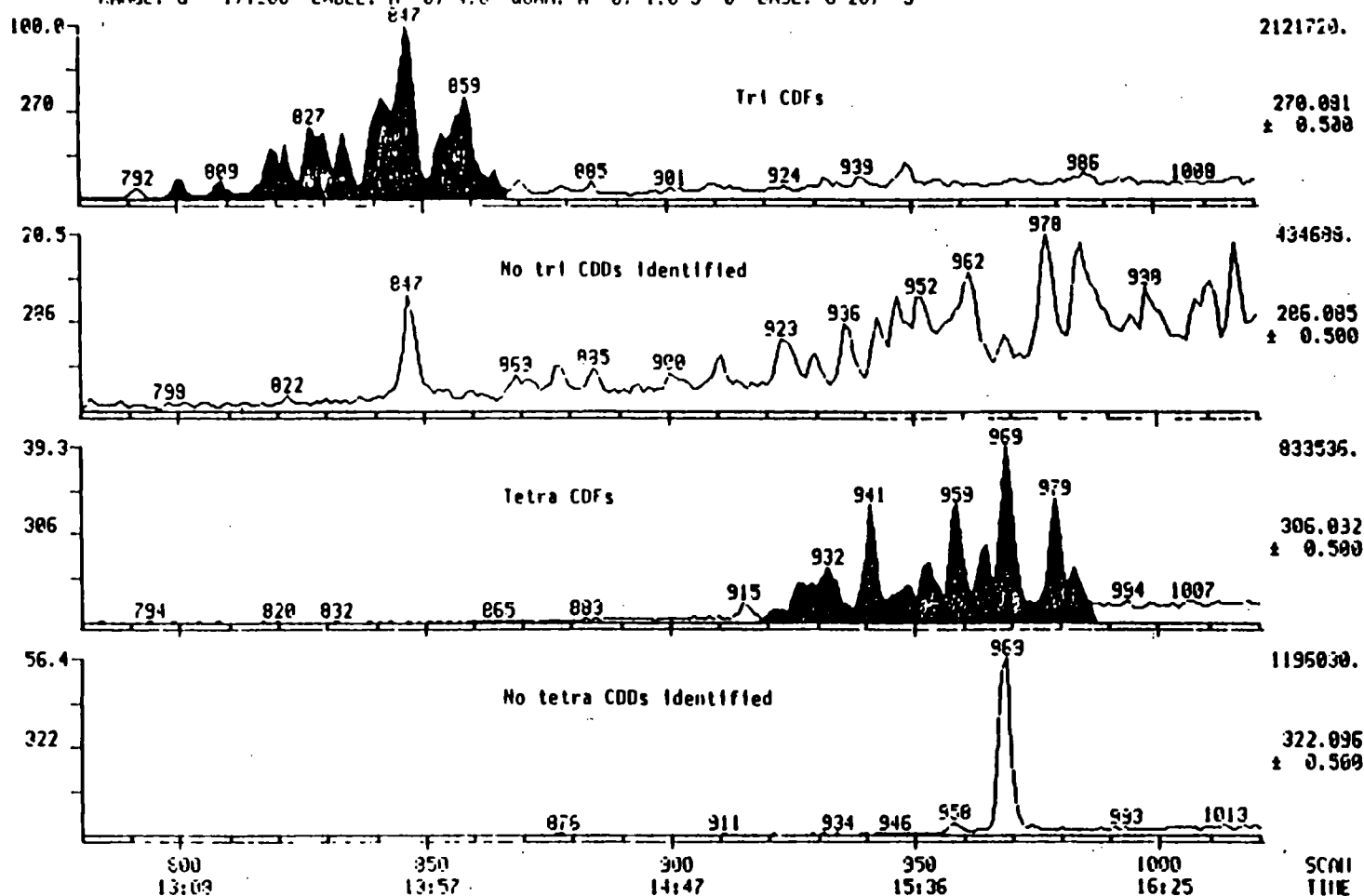
COND5: 1 -2000EMU 70EV 1MA DBS-30M-200-2H-320-10/

RANGE: G 1, 900 LABEL: H 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3



Hepta and octa CDFs and CDDs in sample 8-30-62-ASKL,  
Askarel sample.

MID MASS CHROMATOGRAMS DATA: 8201111X5 P1 SCANS 700 TO 1020  
 03/11/84 11:22:00 CALI: MID250110X1 03  
 SAMPLE: 8201A23 RWI 8-15-43-M5 1M 1M  
 CONDS.: MS=3.5 70EV BC=3 DB5-15M 100-1H-325-10/  
 RANGE: G 1.1260 LABEL: H 0. 4.0 QUAN: A 0. 1.0 J 0 BASE: U 20. 3



Tri and tetra CDFs and CDDs in sample 8-15-43-M5,  
 mineral oil at 5 µg/g PCB.

MID MASS CHROMATOGRAMS

DATA: 829111X3 #1  
(ALL: MID250118X1 #3)

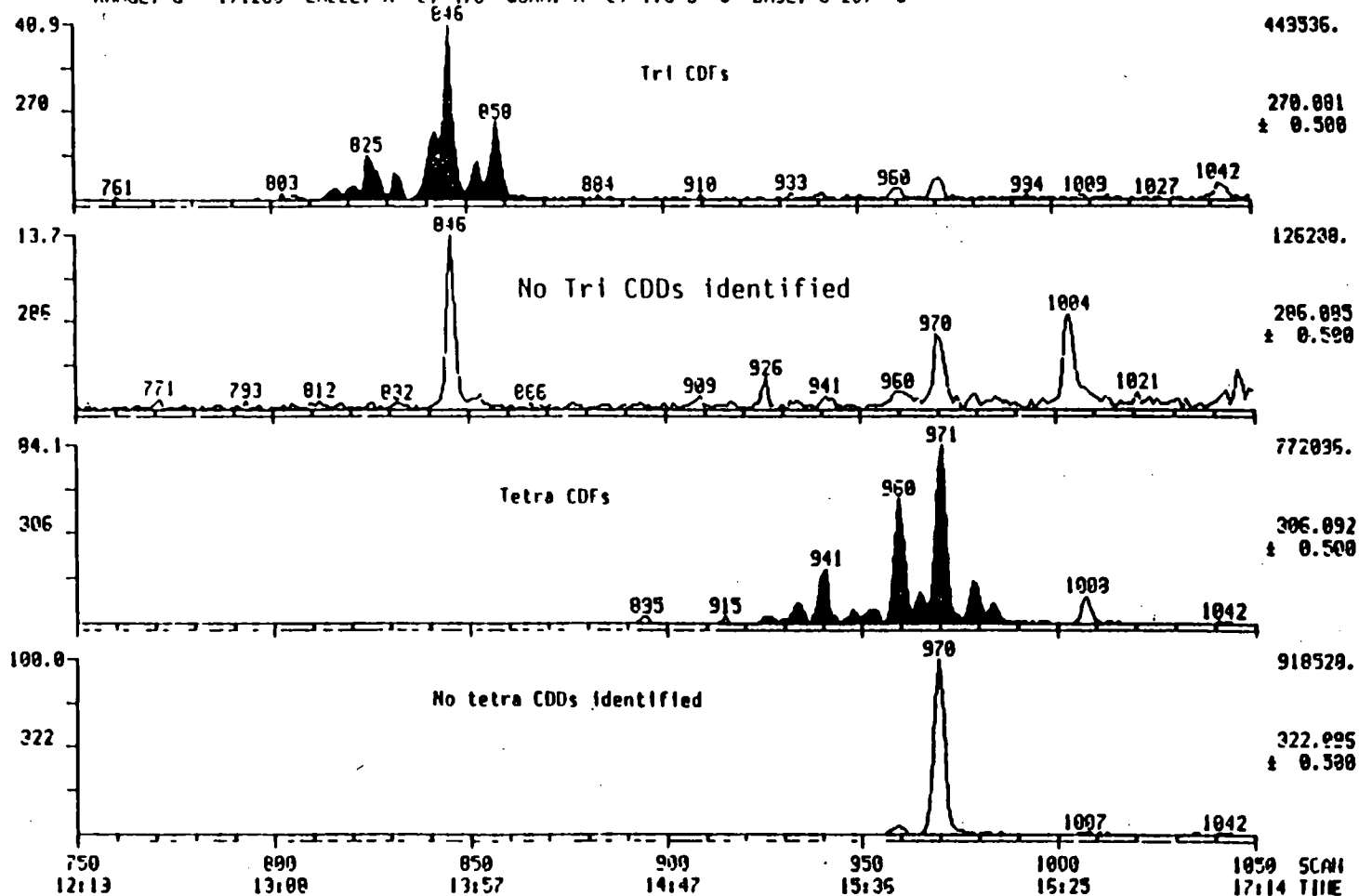
SCANS 750 TO 1050

03/11/84 10:10:00

SAMPLE: 8201A23 RUN 8-17-47 S5 XAD 10L INJ

CONDOS: 1 MG=3.5 70EV GC=3 DB5-15M 100-1H-325-10/

RANGE: G 1.1200 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3



Tri and tetra CDFs and CDDs in sample 8-17-47-S5,  
silicone oil at 5 µg/g PCB.

MID MASS CHROMATOGRAMS

DATA: 0201116X2 #1

SCANS 000 TO 1050

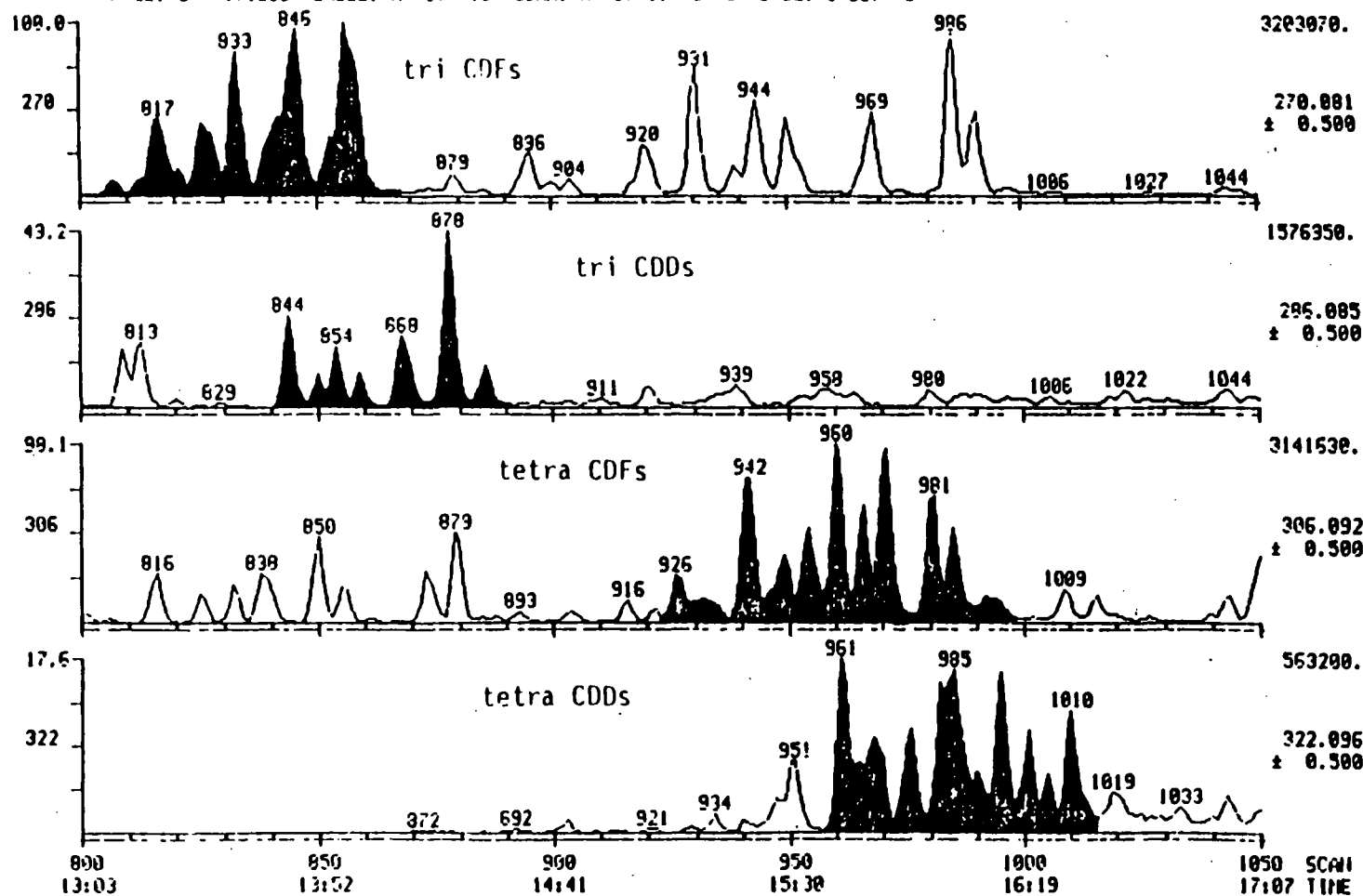
03/10/84 15:45:00

CAL: MID250110X1 #3

SAMPLE: 0201A23 RUN 0-20-57 IUL INJ

CONDS.: MS=3.5 70EV BC=3 DB5-15M 100-III-325-10/

RANGE: G 1.1250 LABEL: H 0. 4.0 QUANT: A 0. 1.0 J 0 BASE: U 20. 3



Tri and tetra CDFs and CDDs in sample 8-28-57-CLBZ,  
chlorobenzene sample.

## **ATTACHMENT 3**

### **PCDD/PCDF Residue Levels in Phenoxy Herbicides**

**A SUMMARY OF THE REPORTED ANALYTICAL LIMITS OF DETECTION FOR POLYCHLORODIBENZO-*p*-DIOXINS (PCDDs)  
AND POLYCHLORODIBENZOFURANS (PCDFs) IN PHENOXYALKANOIC ACID HERBICIDES\***

Reference	Applicability	Compounds detected	Limit of detection (mg kg <sup>-1</sup> of sample)
Storherr et al.	2,4,5-T	TCDD	0.05
Brenner et al. (1972)	2,4-D, dichlorprop, mecoprop, and 2,4,5-T	TCDD	0.01
Vogel (1976)	2,4,5-T	TCDD	0.03
Ramstad et al. (1977)	Butoxypropyl esters of 2,4,5-T and of fenoprop	TCDD	0.005
Elvidge (1971)	2,4,5-T, ethylhexyl ester of 2,4,5-T, and formulations containing the ethylhexyl ester of 2,4,5-T	TCDD	0.05 (on 2,4,5-T content)
Woolson et al. (1972)	2,4-D, 2,4-DB, dichlorprop, 2,4,5-T, fenoprop, and dicamba	Di-, tri-, tetra-, penta-, hexa-, hepta-, and octa-CDDs	0.5 (for any one of the compounds detected)
Edmunds et al. (1973)	2,4,5-T, the butyl and octyl esters of 2,4,5-T, and formulations of the butyl ester of 2,4,5-T (50% in mineral oil)	TCDD	0.05
Crummett and Stehl (1973)	Esters of 2,4,5-T and of fenoprop, Herbicide Orange	TCDD	0.05
Buser et al. (1974)	2,4,5-T, formulations of esters of 2,4,5-T and of amine salts of 2,4,5-T	TCDD	0.001 (on a standard solution)
Huckins et al. (1978)	Herbicide Orange	TCDD and penta-CDD	0.02
Rappe et al. (1978)	Formulations of esters of 2,4,5-T, Herbicide Orange	Di-, tri-, tetra-, penta-, and hexa-CDDs, di-, tri-, tetra-, penta-, and hexa-CDFs	0.01-0.05
Polzhofer (1979)	2,4,5-T, alkyl esters of 2,4,5-T and formulations	TCDD	0.005 (on 2,4,5-T content)
Carello et al.	Fenoprop	TCDD	0.01

\* Baker, P. G., R. H. Hoodless, and J. C. Tyler, *Pestic. Sci.*, 12, 297-304.

**ATTACHMENT 4**

**Literature and Patent Search  
for Applications of  
Aroclor 1268**

ONLINE LITERATURE SEARCH FOR  
BRIAN BIRD -- AROCLOR 1268

PROJ/OVERHEAD #: 3387

L1 ANSWER 1 OF 1 COPYRIGHT 1993 ACS  
RN 11100-14-4 REGISTRY  
CN \*\*\*Aroclor 1268 (9CI)\*\*\* (CA INDEX NAME)  
MF Unspecified  
CI MAN  
LC BIOBUSINESS, BIOSIS, CA, CAPREVIEWS, CHEMLIST, CJACS, CSCHEM,  
IFICDB, IFIPAT, IFIUDB, RTECS

\*\*\* STRUCTURE DIAGRAM IS NOT AVAILABLE \*\*\*  
1 REFERENCES IN FILE CAPREVIEWS  
57 REFERENCES IN FILE CA (1967 TO DATE)  
1 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA



FILE COVERS 1950 TO DATE.

FILE LAST UPDATED: 2 Aug 1993 (930802/ED) PATENT PUBLICATION: 27 Jul 1993

HIGHEST PATENT NUMBER: 5231699 - INDEXING IS AVAILABLE IN THE IFIUDB

FILE AND IS CURRENT THROUGH 30 MAR 1993 (930731/UP)

FILE 'IFIPAT' ENTERED AT 10:07:55 ON 09 AUG 93

L4 10 S L2

L4 ANSWER 1 OF 10

AN 1158491 IFIPAT;IFIUDB;IFICDB

TI FLAME RETARDANT, NON-DRIPPING COMPOSITIONS OF POLYPHENYLENE ETHER  
AND ACRYLONITRILE-BUTADIENE-STYRENE; HALOGENATED COMPOUND,  
POLYTETRAFLUOROETHYLENEINF Haaf, William Robert, Voorhesville, NY  
Reinhard, Donald Lewis, Lenox, MA

IN HAAF WILLIAM ROBERT; REINHARD DONALD LEWIS

PAF General Electric, Pittsfield, MA

PA GENERAL ELECTRIC CO (33808)

EXNAM Ziegler, J

AG Morgan, Finnegan, Pine, Foley &amp; Lee

PI US 4107232 15 Aug 1978 (CITED IN 008 LATER PATENTS)

AI US 75-642981 22 Dec 1975

RLI US 74-530373 6 Dec 1974 DIVISION

FI US 4107232 15 Aug 1978

DT UTILITY

FS CHEMICAL

OS CA 90:55799

AB The present invention provides novel flame retardant, nondripping  
thermoplastic molding compositions of polyphenylene ether resins or  
acrylonitrile-butadiene-styrene copolymers which include a flame  
retardant agent, and polytetrafluoroethylene.

L4 ANSWER 2 OF 10

AN 1084847 IFIPAT;IFIUDB;IFICDB

TI FLAME-RETARDANT RESIN COMPOSITIONS; COPPER CLAD LAMINATES,  
POLYHALOGENATED AROMATIC HYDROCARBONS, POLYPHENYLENE ETHERS, COATED  
ELECTRICAL DEVICES

INF Beacham, Harry Hoyt, Severna Park, MD

Wright, Carl Leonard, Pasadena, MD

IN BEACHAM HARRY HOYT; WRIGHT CARL LEONARD

PAF FMC Corporation, Philadelphia, PA

PA FMC CORP (31248)

EXNAM Lesmes, George F

EXNAM Thibodeau, P J

AG Fellows, Charles C

Ianno, Frank

PI US 4034136 5 Jul 1977 (CITED IN 010 LATER PATENTS)

AI US 75-575515 8 May 1975

RLI US 69-837911 30 Jun 1969 CONTINUATION-IN-PART ABANDONED

US 70-95418 4 Dec 1970 DIVISION 3936414

FI US 4034136 5 Jul 1977

US 3936414

DT UTILITY; REASSIGNED

FS CHEMICAL

OS CA 87:69143

AB This specification discloses that certain polychlorinated or  
polybrominated aromatic hydrocarbon compounds can be incorporated  
in thermosetting polymer compositions to make the thermosetting  
compositions flame retardant without injuring the desirable  
physical and electrical properties of the thermosetting  
compositions when in the thermoset state. These polyhalogenated  
aromatic hydrocarbons are apparently caused to react into the  
thermosetting composition, provided there is a minimum of 1 mol of  
a polyunsaturated monomer containing carbon to carbon unsaturation  
for each 3 mols of polychlorinated aromatic hydrocarbons. The  
thermosetting compositions generally contain a polyunsaturated  
monomer, a polyunsaturated polymer with carbon to carbon double  
bond unsaturation or a polyphenylene ether polymer which may be

polyhalogenated aromatic hydrocarbon, containing at least 50% by weight combined chlorine or bromine and whose molecular weight is greater than 200. These thermosetting compositions can be heat cured or thermoset by a free radical initiator at a temperature of at least 130° C. and can contain pigments, reinforcing fibers, release agents, coupling agents, and other incidental amounts of additives commonly used in thermosetting resin compositions.

L4 ANSWER 3 OF 10  
AN 0947133 IFIPAT;IFIUDB;IFICDB  
TI POLYAMIDE-IMIDES  
INF Brode, George Lewis, Somerville, NJ  
Kawakami, James Hajime, Piscataway, NJ  
IN BRODE GEORGE LEWIS; KAWAKAMI JAMES HAJIME  
PAF Union Carbide Corporation, New York, NY  
PA UNION CARBIDE CORP (87136)  
EXNAM Shaver, Paul F  
AG Crowe, B F  
PI US 3897497 29 Jul 1975 (CITED IN 003 LATER PATENTS)  
AI US 74-456805 1 Apr 1974  
RLI US 70-50968 29 Jun 1970 CONTINUATION-IN-PART ABANDONED  
US 71-182526 21 Sep 1971 DIVISION 3817921  
FI US 3897497 29 Jul 1975  
US 3817921  
DT UTILITY; REASSIGNED  
FS CHEMICAL  
OS CA 84:5655  
AB A series of polyamide-imides which are both tractable and soluble in polar solvents as well as stable at high temperatures has been prepared by the condensation polymerization of aromatic diamines or oligomers containing oxygen, sulfone, and optionally alkylidene linkages with trimellitic acid, trimellitic acid anhydride or trimellitic anhydride acid chloride.

L4 ANSWER 4 OF 10  
AN 0888552 IFIPAT;IFIUDB;IFICDB  
TI POLYARYLIMIDES; GLASS CLOTH PREPEGS, MOLDING, ADHESIVES  
INF Brode, George L, Somerville, NJ  
Kwiatkowski, George T, Greenbrook, NJ  
IN BRODE G; KWIATKOWSKI G  
PAF Union Carbide Corporation, New York, NY  
PA UNION CARBIDE CORP (87136)  
EXNAM Lee, Lester L  
AG Crowe, Bernard Francis  
PI US 3839287 1 Oct 1974 (CITED IN 014 LATER PATENTS)  
AI US 73-362287 21 May 1973  
RLI US 71-182527 21 Sep 1971 CONTINUATION-IN-PART ABANDONED  
FI US 3839287 1 Oct 1974  
DE 2246026  
FR 2153331  
GB 1408933  
DT UTILITY; REASSIGNED  
FS CHEMICAL  
OS CA 78:160446  
AB Arylimides have been prepared from oligomer diamines and polyamines containing halogenated phenylene, oxygen, alkylidene and/or sulfone moieties and maleic anhydride. The resultant aryylimides, in the form of B-stage resins can be used for the preparation of glass cloth prepreps, molding materials and adhesives which can be thermally converted to the thermoset state.

L4 ANSWER 5 OF 10  
AN 0839576 IFIPAT;IFIUDB;IFICDB  
TI MICROBIOCIDAL NITROGEN DERIVATIVES OF HALOGENATED BIPHENYLS  
IN ADAMS P; MERIANOS J; PETROCCI A; SHAY E  
PA MILLMASTER ONYX CORP (55656)  
PI US 3790678 5 Feb 1974  
AI US 71-111681 1 Feb 1971  
RLI US 69-883636 9 Dec 1969 DIVISION 3663620

US 3663620  
DT UTILITY; REASSIGNED  
FS CHEMICAL  
OS CA 80:129250  
AB A METHOD OF INHIBITING MICROORGANISMS WHICH COMPRISES APPLYING TO THE ENVIRONMENT IN WHICH SUCH MICROORGANISMS ARE FOUND AMMONIA AND AMINOSUBSTITUTED HALOGENATED BIPHENYLS, AS WELL AS THE NITROGEN DERIVATIVES THEREOF SUCH AS QUATERNARY AMMONIUM COMPOUNDS AMINE OXIDES, IMIDAZOLINES, AMIDES, ENAMINES, AMPHOLYTES, AND THE LIKE. THE AMMONIA AND AMINO HALOBIPHENYLS ARE PREPARED BY REACTING THE AMMONIA OR THE AMINE AND THE HALOBIPHENYL REACTANTS IN SUCH A MANNER THAT THE RATIO OF AMINE TO HALOBIPHENYL IS PREFERABLY LIMITED TO BETWEEN TWO AND FIVE MOLS OF AMINE TO EACH MOL OF HALOBIPHENYL

L4 ANSWER 6 OF 10  
AN 0811931 IFIPAT;IFIUDB;IFICDB  
TI N-(PENTACHLOROBIPHENYL) ETHYLENE DIAMINE; MICROBIOCIDES  
IN ADAMS P; MERIANOS J; PETROCCI A; SHAY E  
PA MILLMASTER ONYX CORP (55656)  
PI US 3763238 2 Oct 1973  
AI US 72-234987 15 Mar 1972  
RLI US 69-883636 9 Dec 1969 DIVISION 3663620  
FI US 3763238 2 Oct 1973  
US 3663620  
DT UTILITY; REASSIGNED  
FS CHEMICAL  
OS CA 79:146165  
AB MICROBIOCIDAL AMMONIA AND AMINO SUBSTITUTED HALOGENATED BIPHENYLS, AS WELL AS THE NITROGEN DERIVATIVES THEREOF SUCH AS QUATERNARY AMMONIUM COMPOUNDS, AMINE OXIDES, IMIDAZOLINES, AMIDES, ENAMINES, AMPHOLYTES, AND THE LIKE. THE AMMONIA AND AMINO HALOBIPHENYLS ARE PREPARED BY REACTING THE AMMONIA OR THE AMINE AND THE HALOBIPHENYL REACTANTS IN SUCH A MANNER THAT THE RATIO OF AMINE TO HALOBIPHENYL IS PREFERABLY LIMITED TO BETWEEN TWO AND FIVE MOLS OF AMINE TO EACH MOL OF HALOBIPHENYL.

L4 ANSWER 7 OF 10  
AN 0808677 IFIPAT;IFIUDB;IFICDB  
TI N-(NONACHLOROBIPHENYL) AMINOPROPYL-N'-DIMETHYLAMINE; ANTISEPTICS, PRESERVATIVES, MICROBIOCIDES  
INF Adams, Phillip, Murray Hill, NJ  
Merianos, John J, Jersey City, NJ  
Petrocci, Alfonso N, Glen Rock, NJ  
Shay, Edward Griffin, Suffern, NY  
PAF Millmaster Onyx Corporation, New York, NY  
PA MILLMASTER ONYX CORP (55656)  
EXNAM Hines, Robert V  
AG Jacobs, Arthur A  
PI US 3759995 18 Sep 1973  
AI US 72-235064 15 Mar 1972  
RLI US 69-883636 9 Dec 1969 DIVISION 3663620  
FI US 3759995 18 Sep 1973  
US 3663620  
DT UTILITY; REASSIGNED  
FS CHEMICAL  
OS CA 79:126057  
AB Microbiocidal ammonia and amino substituted halogenated biphenyls, as well as the nitrogen derivatives thereof such as quaternary ammonium compounds, amine oxides, imidazoles, amides, enamines, ampholytes, and the like. The ammonia and amino halobiphenyls are prepared by reacting the ammonia or the amine and the halobiphenyl reactants in such a manner that the ratio of amine to halobiphenyl is preferably limited to between two and five mols of amine to each mol of halobiphenyl.

L4 ANSWER 8 OF 10  
AN 0804103 IFIPAT;IFIUDB;IFICDB  
I N-(PENTACHLOROBIPHENYL)-DIETHYLENE TRIAMINE; MICROBIOCIDES  
IN ADAMS P; MERIANOS J; PETROCCI A; SHAY E

PI US 3755448 28 Aug 1973 (CITED IN 001 LATER PATENTS)  
AI US 72-235092 15 Mar 1972  
RLI US 69-883636 9 Dec 1969 DIVISION 3663620  
FI US 3755448 28 Aug 1973  
US 3663620  
DT UTILITY; REASSIGNED  
FS CHEMICAL  
JS CA 79:104918  
AB MICROBIOCIDAL AMMONIA AND AMINO SUBSTITUTED HALOGENATED BIPHENYLS,  
AS WELL AS THE NITROGEN DERIVATIVES THEREOF SUCH AS QUATERNARY  
AMMONIUM COMPOUNDS, AMINE OXIDES, IMIDAZOLINES, AMIDES, ENAMINES,  
AMPHOLYTES, AND THE LIKE. THE AMMONIA AND AMINO HALOBIPHENYLS ARE  
PREPARED BY REACTING THE AMMONIA OR THE AMINE AND THE HALOBIPHENYL  
REACTANTS IN SUCH A MANNER THAT THE RATIO OF AMINE TO HALOBIPHENYL  
IS PREFERABLY LIMITED TO BETWEEN TWO AND FIVE MOLS OF AMINE TO EACH  
MOL OF HALOBIPHENYL.

L4 ANSWER 9 OF 10  
AN 0781923 IFIPAT;IFIUDB;IFICDB  
TI MICROBIOCIDAL NITROGEN DERIVATIVES OF HALOGENATED BIPHENYLS  
IN ADAMS P; MERIANOS J; PETROCCI A; SHAY E  
PA MILLMASTER ONYX CORP (55656)  
PI US 3733421 15 May 1973 (CITED IN 003 LATER PATENTS)  
AI US 70-38513 18 May 1970  
RLI US 69-883636 9 Dec 1969 CONTINUATION-IN-PART 3663620  
FI US 3733421 15 May 1973  
US 3663620  
DT UTILITY; REASSIGNED  
FS CHEMICAL  
OS CA 79:42132  
AB MICROBIOCIDAL BIS-AMINO SUBSTITUTED HALOGENATED BIPHENYLS, AS WELL  
AS THE NITROGEN DERIVATIVES THEREOF SUCH AS QUATERNARY AMMONIUM  
COMPOUNDS, AMINE OXIDES, IMIDAZOLINES, AMIDES, ENAMINES,  
AMPHOLYTES, AND THE LIKE.

L4 ANSWER 10 OF 10  
N 0711662 IFIPAT;IFIUDB;IFICDB  
I N-(HALOGENATED BIPHENYL)-DIETHYLENE TRIAMINES  
IN ADAMS PHILLIP; MERIANOS JOHN J; PETROCCI ALFONSO N; SHAY EDWARD  
GRIFFIN  
PA MILLMASTER ONYX CORP (55656)  
PI US 3663620 16 May 1972 (CITED IN 007 LATER PATENTS)  
AI US 69-883636 9 Dec 1969  
FI US 3663620 16 May 1972  
DT UTILITY; REASSIGNED  
FS CHEMICAL  
OS CA 77:48033  
AB MICROBIOCIDAL AMMONIA AND AMINO SUBSTITUTED HALOGENATED BIPHENYLS,  
AS WELL AS THE NITROGEN DERIVATIVES THEREOF SUCH AS QUATERNARY  
AMMONIUM COMPOUNDS, AMINE OXIDES, IMIDAZOLINES, AMIDES, ENAMINES,  
AMPHOLYTES, AND THE LIKE. THE AMMONIA AND AMINO HALOBIPHENYLS ARE  
PREPARED BY REACTING THE AMMONIA OR THE AMINE AND THE HALOBIPHENYL  
REACTANTS IN SUCH A MANNER THAT THE RATIO OF AMINE TO HALOBIPHENYL  
IS PREFERABLY LIMITED TO BETWEEN TWO AND FIVE MOLS OF AMINE TO EACH  
MOL OF HALOBIPHENYL.

L1 57 S 11100-14-4  
L2 5 S L1 AND (USE OR USES OR APPLICATION!)  
L3 6 S L1 AND (USE OR USES OR APPLICATION!)/AB  
L4 6 S L3 NOT L2

L2 ANSWER 1 OF 5 COPYRIGHT 1993 ACS

CA118(8):65794m On the \*\*\*use\*\*\* of tree leaves as bioindicators of the contamination of air by organochlorines in France. Granier, Laurent; Chevreuil, Marc (Inst. Hydrol. Climatol., Univ. Pierre et Maris Curie, Paris 75252, Fr.). Water, Air, Soil Pollut., 64(3-4), 575-84 (Eng) 1992. CODEN: WAPLAC. ISSN: 0049-6979.

AB The possibility of using leaves of plane trees (*Platanus vulgaris*) as bioindicators of air pollution by organochlorines was studied in France. Inter- and intraspecific variations by a factor of 3 in the concns. of polychlorinated biphenyls were obsd. This type of biomonitoring appears appear to be capable of discerning, on a continental scale, variations of a factor 10 in atm. concns. Air/leaf bioconc. factors are .apprx.105 for PCBs and 104 for lindane.

L2 ANSWER 2 OF 5 COPYRIGHT 1993 ACS

CA109(16):134468m Degradation and dehalogenation of polychlorobiphenyls and halogenated aromatic molecules by superoxide ion and by electrolytic reduction. Sugimoto, Hiroshi; Matsumoto, Shigenobu; Sawyer, Donald T. (Dep. Chem., Texas A and M Univ., College Station, TX 77843, USA). Environ. Sci. Technol., 22(10), 1182-6 (Eng) 1988. CODEN: ESTHAG. ISSN: 0013-936X. OTHER SOURCES: CJACS.

AB Polyhalogenated arom. hydrocarbons (e.g., PCB's and hexachlorobenzene) are rapidly degraded by O<sub>2</sub><sup>-</sup> in DMF to give CO<sub>3</sub><sup>2-</sup> and halide ions. The efficient destruction of such materials is accomplished via the in-situ electrolytic redn. of dissolved O to generate O<sub>2</sub><sup>-</sup>, which reacts with polyhalo aroms. by nucleophilic substitution. The reaction stoichiometries were detd. by cyclic voltammetric measurements, and the reactant/produced profiles were assayed by capillary gas chromatog. and potentiometric titrns. Complete destruction by O<sub>2</sub><sup>-</sup> occurs for PCB's that contain .gtoreq.3 Cl atoms per arom. ring. Another means to the dehalogenation of halo arom. hydrocarbons is their electrolytic redn. to the parent hydrocarbon in O-free DMF solns. Electrochem. studies confirm that all PCB's can be dehalogenated via anaerobic electrolysis.

L2 ANSWER 3 OF 5 COPYRIGHT 1993 ACS

CA107(12):108523q The \*\*\*use\*\*\* of simplex optimization in evaluating complex chromatograms of mixtures. Tomas, X.; Sabate, L. G. (Dep. Chemometrics, Inst. Quim. Sarria, Barcelona 08017, Spain). Anal. Chim. Acta, 191, 439-43 (Eng) 1986. CODEN: ACACAM. ISSN: 0003-2670.

AB The application of simplex optimization to the qual. and quant. evaluation of chromatograms of complex mixts. is outlined. The chromatogram is considered as a linear combination of pure component chromatograms (assuming no interactions are present); if suitable components are selected and the coeffs. of the linear combinations are properly evaluated, the exptl. chromatogram can be reproduced numerically. The simplex method is then used to evaluate the soln. with minimal error. The quantitation of polychlorobiphenyls from Aroclor mixts. in marine sediments and fish, and the identification of essential oils in a perfume base ext. are outlined. Errors of .ltoreq.10% (25% in environmental samples) between the exptl. and reconstructed chromatograms were achieved.

L2 ANSWER 4 OF 5 COPYRIGHT 1993 ACS

CA90(8):55799f Flame retardant, nondripping compositions of polyphenylene ether and acrylonitrile-butadiene-styrene. Haaf, William Robert; Reinhard, Donald Lewis (General Electric Co., USA). U.S. US 4107232 15 Aug 1978, 6 pp. (Eng). CODEN: USXXAM. CLASS: IC: C03L051-00. NCL: 260876000R. APPLICATION: US 74-530373 6 Dec 1974.

AB The addn. of PTFE [9002-84-0] to a poly(2,6-dimethyl-1,4-phenylene) ether (I) [24938-67-8] compn. or a ABS polymer [9003-56-9] compn., each contg. a fireproofing agent, increased to the non-dripping

80, polyethylene [9002-88-4] 1.5, tetrabromobisphenol A [79-94-7] 8, triphenyl phosphate [115-86-6] 1, Sb2O3 3, and PTFE 0.25 part was molded into self-extinguishing non-dripping sheets cf. to self-extinguishing but dripping sheets when PTFE was omitted.

L2 ANSWER 5 OF 5 COPYRIGHT 1993 ACS

CA89(16):134737s Separation of chlorinated dibenzo-p-dioxins from chlorinated congeners. Dolphin, R. J.; Willmott, F. W. (Philips Res. Lab., Redhill/Surrey, Engl.). J. Chromatogr., 149, 161-8 (Eng) 1978. CODEN: JOCRAM. ISSN: 0021-9673.

AB Chlorinated dibenzo-p-dioxins, e.g. 2,3,7,8-tetrachlorodibenzo-p-dioxin [1746-01-6] and octachlorodibenzo-p-dioxin [3268-87-9], were sepd. from polychlorinated biphenyls, polychlorinated naphthalenes, polychlorinated dibenzofurans, and organochlorine pesticides by high-performance liq.-chromatog. on microparticulate Al2O3 column.

L4 ANSWER 1 OF 6 COPYRIGHT 1993 ACS

CA117(6):61928q Barycentric coordinates and the orientation of the classical mixture surface. Rayens, William S. (Dep. Stat., Univ. Kentucky, Lexington, KY 40506, USA). J. Math. Chem., 9(2), 147-60 (Eng) 1992. CODEN: JMCHEG. ISSN: 0259-9791.-Q

AB The signs of the barycentric coordinates of a point exterior to a nondegenerate k-simplex in IR<sup>p</sup> contain useful information about how that point is positioned relative to the vertices of that simplex. This relationship is certainly not newly obsd., with some of the first ideas dating back to Moebius in 1827. However, this article presents some new geometrical results which further quantify the relationship and focuses on applying these new results to help solve the problem of finding the point on a simplex that is closest to a given exterior point. In particular, it is shown that the signs of the barycentrics can be used to immediately identify a potentially large set of facets that could not contain this closest point. Such results have immediate \*\*\*applications\*\*\* to the problem of identifying the components in a chem. linear mixt. Real polychlorobiphenyl mixts. are employed to illustrate the new ideas.

L4 ANSWER 2 OF 6 COPYRIGHT 1993 ACS

CA115(14):141483q Evidence of an unusual pattern of polychlorinated biphenyls in the serum of some residents and canines in Paoli, Pennsylvania. Burse, Virlyn W.; Groce, Donald F.; Korver, Margaret P.; Caudill, Samuel P.; McClure, Patricia C.; Lapeza, Chester R., Jr.; Head, Susan L.; Schilling, Rebecca J.; Farrar, Jeffrey A.; et al. (Cent. Environ. Health Injury Control, Cent. Dis. Control, Atlanta, GA 30333, USA). J. - Assoc. Off. Anal. Chem., 74(4), 577-86 (Eng) 1991. CODEN: JANCA2. ISSN: 0004-5756. OTHER SOURCES: CJAOC.

AB The present study \*\*\*uses\*\*\* gas chromatog. with electron capture detection with packed and capillary columns to detect polychlorinated biphenyls (PCBs) in serum samples from people living near the elec. car repair and maintenance facility of the Southeastern Pennsylvania Transit Authority in Paoli, Pennsylvania. Most of the cohort surveyed had serum patterns similar to patterns for Aroclor 1260 (AR 1260); a small portion (3 of 89) had patterns indicative of an AR with higher chlorination (e.g., AR 1268). In addn. to analyzing serum samples from humans, serum samples from canines (pet of some of the subjects) were analyzed. In general, the serum pattern for canines was less descriptive for AR 1260 than the pattern for humans; however, the pattern for several canines (9 of 16) was that of the higher chlorinated PCBs (e.g., AR 1268). By using mass spectrometry and capillary column gas chromatog., the presence of high-mol.-wt. polychlorinated congeners in both human and animal samples was confirmed. A statistically significant relationship was not shown between serum patterns of PCBs in canines and their owners or between canines and certain behavioral traits (e.g., runs free, retrieves, hours outside, hours inside). However, the correlation between PCBs quantified as AR 1266 and canines' residence time was statistically significant.

CA115(10):105183h Determination of congeners of polychlorinated biphenyls in reference materials. Anderson, Jerry W. (Robert S. Kerr Environ. Res. Lab., NSI Technol. Serv., Inc., Ada, OK 74820, USA). J. High Resolut. Chromatogr., 14(6), 369-72 (Eng) 1991. CODEN: JHRCE7. ISSN: 0935-6304.

AB Aroclor ref. materials from the United States Environmental Protection Agency repository are proposed for \*\*\*use\*\*\* as secondary stds. for the congener-specific anal. for polychlorinated biphenyls in environmental samples. Representative samples of the materials have been analyzed using a gas chromatog. equipped with photoionization and electron capture detectors. The elution pattern of the congeners was detd. for a DB-17 capillary column.

L4 ANSWER 4 OF 6 COPYRIGHT 1993 ACS

CA114(20):191848n Speciation and quantitation of Aroclors in hazardous wastes based on PCB congener data. Draper, William M.; Wijekoon, Donald; Stephens, Robert D. (Hazard. Mater. Lab., State of California Dep. Health Serv., Berkeley, CA 94704, USA). Chemosphere, 22(1-2), 147-63 (Eng) 1991. CODEN: CMSHAF. ISSN: 0045-6535.

AB Selected chlorobiphenyl congener data were used to accurately est. the Aroclor content of hazardous wastes. Only 12 chlorobiphenyl congeners are detd., nominally, IUPAC Nos. 15, 18, 31, 87, 105, 110, 118, 170, 180, 183, 203, and 206, and from these marker compds. Aroclors 1016, 1254, 1260 and 1268 are estd. The remaining regulated Aroclor mixts. also are measured by this technique making it suitable for enforcement of existing California and US regulations. An addnl. 34 chlorobiphenyl congeners are routinely analyzed allowing detn. of PCBs in highly weathered and/or treated wastes. Included are the major constituents of the com. Aroclors and the predominant congeners in the environment. The sum of PCB congener concns., s-PCB, is generally .apprx.70% of the total Aroclor content. The \*\*\*use\*\*\* of capillary column sepns. and data redn. procedures described has a no. of advantages over packed column GC procedures: (1) data interpretation does not require analyst judgement and can be automated; (2) the technique makes full \*\*\*use\*\*\* of the sepn. power of capillary columns to minimize interference by pesticides and other sample components; and (3) the technique accurately measures PCBs in samples with >1 Aroclor. Application of this technique to the detn. of Aroclors in auto shredder waste is described and results compared to a packed-column procedure, U.S. EPA method 8080.

L4 ANSWER 5 OF 6 COPYRIGHT 1993 ACS

CA103(8):58345c Optimization of perchlorination conditions for some representative polychlorinated biphenyls. Lin, Jia Ming; Hee, Shane S. Que (Med. Cent., Univ. Cincinnati, Cincinnati, OH 45267-0056, USA). Anal. Chem., 57(11), 2130-4 (Eng) 1985. CODEN: ANCHAM. ISSN: 0003-2700. OTHER SOURCES: CJACS.

AB The yield of decachlorobiphenyl (I) [2051-24-3] from representative Aroclors (50 to 6000 ng) using SbCl<sub>5</sub> as perchlorinating agent was extremely temp.-dependent at <236 for a reaction period of 2 h. At 288.degree., a 35-min reaction period was sufficient to obtain I yields of >80% for Aroclor 1016 [12674-11-2], 1242 [53469-21-9], 1254 [11097-69-1], and 1268 [11100-14-4]. The perchlorination process was shown to be 1st-order. The temp. dependence of the reaction times below 236.degree. was largely responsible for the inconsistent perchlorination yields reported previously in the literature. The extn. of I with hexane from a HCl-acidified perchlorinated soln., and the subsequent column chromatog. on silica gel were also essential after the perchlorination to quantitate the I by <sup>63</sup>Ni electron capture/gas chromatog. The structures of the 2 nonachlorobiphenyls which were the penultimate stable intermediates before I, were found. The methodol. to \*\*\*use\*\*\* the technique for air and blood samples was described.

CA83(19):158697f Occurrence of polychlorinated biphenyls in humans.  
Price, Harold A.; Welch, Robert L. (Michigan Dep. Public Health  
Pestic., Lansing, Mich., USA). Environ. Health Perspect., 1, 73-8  
(Eng) 1972. CODEN: EVHPAZ.

AB Anal. of human adipose tissue, collected through the human  
monitoring survey program (Division of Pesticide Community Studies,  
E. P. A.) revealed that 41-5% of the general population have levels  
of .gtoreq.1.0 ppm (wet wt.) polychlorinated biphenyls, with isomers  
from Aroclors 1254 [11097-69-1], 1260 [11096-82-5], 1262  
[37324-23-5], and 1268 [11100-14-4]. The presence of polychlorinated  
biphenyls ranging from pentachlorobiphenyls to decachlorobiphenyls  
was confirmed in selected samples by the \*\*\*use\*\*\* of combined  
gas chromatog.-mass spectrometry. The possible interference of these  
compds. with the anal. of organochlorine pesticide residues is  
discussed.



Utility  
 FLAME RETARDANT, / NON-DRIPPING COMPOSITIONS OF POLYPHENYLENE ETHER AND  
 ACRYLONITRILE-BUTADIENE-STYRENE

PATENT NO.: 4,355,126  
 ISSUED: October 19, 1982 (19821019)  
 INVENTOR(s): Haaf, William R., Voorhesville, NY (New York), US (United  
 States of America)  
 Reinhard, Donald L., Lenox, MA (Massachusetts), US (United  
 States of America)  
 ASSIGNEE(s): General Electric Company, (A U.S. Company or Corporation),  
 Pittsfield, MA (Massachusetts), US (United States of America)  
 APPL. NO.: 6-15,103  
 FILED: February 26, 1979 (19790226)

This is a continuation of application Ser. No. 530,373 filed Dec. 6,  
 1974, abandoned.

FULL TEXT: 585 lines

#### ABSTRACT

The present invention provides novel flame retardant, non-dripping  
 thermoplastic molding compositions of polyphenylene ether resins or  
 acrylonitrile-butadiene-styrene copolymers which include a flame retardant  
 agent, and polytetrafluoroethylene.

...sup 1

lene HBB sup 1  
 TPP sup 2  
 AO sup 3  
 PTFE sup 4  
 Aroclor 1268 sup 5  
 Times (sec...)

3/4/2 (Item 1 from file: 652)  
 01043890 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
 FLAME-RETARDANT RESIN COMPOSITIONS

PATENT NO.: 4,159,371  
 ISSUED: June 26, 1979 (19790626)  
 INVENTOR(s): Wright, Carl L., Pasadena, MD (Maryland), US (United States of  
 America)  
 Beacham, Harry H., Severna Park, MD (Maryland), US (United  
 States of America)  
 ASSIGNEE(s): FMC Corporation, (A U.S. Company or Corporation), Philadelphia  
 , PA (Pennsylvania), US (United States of America)  
 APPL. NO.: 5-783,533  
 FILED: April 01, 1977 (19770401)

#### CROSS REFERENCE TO RELATED APPLICATION

This is a division, of application Ser. No. 575,515, filed May 8, 1975,  
 and now U.S. Pat. No. 4,034,436 which is a division of application Ser. No.  
 95,418, now U.S. Pat. No. 3,936,414, filed Dec. 4, 1970, which is a  
 continuation-in-part of Ser. No. 837,911, filed June 30, 1969, now  
 abandoned.

FULL TEXT: 1575 lines

#### ABSTRACT

This specification discloses that certain polychlorinated or polybrominated  
 aromatic hydrocarbon compounds can be incorporated in thermosetting polymer

injuring the desirable physical and electrical properties of the thermosetting compositions when in the thermoset state. These polyhalogenated aromatic hydrocarbons are apparently caused to reset into the thermosetting composition, provided there is a minimum of 1 mol of a polyunsaturated monomer containing carbon to carbon unsaturation for each 3 mols of polychlorinated aromatic hydrocarbons. The thermosetting compositions generally contain a polyunsaturated monomer, a polyunsaturated polymer with carbon to carbon double bond unsaturation or a polyphenylene ether polymer which may be used in place of some or all of the polyunsaturated polymer, and a polyhalogenated aromatic hydrocarbon, containing at least 50% by weight combined chlorine or bromine and whose molecular weight is greater than 200. These thermosetting compositions can be heat cured or thermoset by a free radical initiator at a temperature of at least 130 degree(s) C., and can contain pigments, reinforcing fibers, release agents, coupling agents, and other incidental amounts of additives commonly used in thermosetting resin compositions.

...5460)  
 50 45 40 60 65 60 -- -- 40 74 87 78  
 Chlorinated biphenyl,  
 68% chlorine ( Aroclor 1268 )  
 -- -- -- -- -- 62 62 -- -- -- --  
 Diallyl phthalate  
 -- -- -- -- -- 40 -- 38 -- -- -- --  
 Diallyl isophthalate  
 50 55 60 40 35 -- 38 -- -- -- -- --  
 Diallyl

3/4/3 (Item 2 from file: 652)  
 00988079 DIALOG File 652: U.S. Patents-Full Text (1971-79)  
 Utility  
 FLAME RETARDANT, NON-DRIPPING COMPOSITIONS OF POLYPHENYLENE ETHER AND  
 ACRYLONITRILE-BUTADIENE-STYRENE

PATENT NO.: 4,107,232  
 ISSUED: August 15, 1978 (19780815)  
 INVENTOR(s): Haaf, William Robert, Voorhesville, NY (New York), US (United States of America)  
 Reinhard, Donald Lewis, Lenox, MA (Massachusetts), US (United States of America)  
 ASSIGNEE(s): General Electric, (A U.S. Company or Corporation), Pittsfield, MA (Massachusetts), US (United States of America)  
 APPL. NO.: 5-642,981  
 FILED: December 22, 1975 (19751222)

This is a division, of application Ser. No. 530,373 filed Dec. 6, 1974.

FULL TEXT: 541 lines

#### ABSTRACT

The present invention provides novel flame retardant, non-dripping thermoplastic molding compositions of polyphenylene ether resins or acrylonitrile-butadiene-styrene copolymers which include a flame retardant agent, and polytetrafluoroethylene.

...sup 1  
 lene HBB sup 1  
 TPP sup 2  
 AO sup 3  
 PTFE sup 4  
 Aroclor 1268 sup 5  
 Times...

3/4/4 (Item 3 from file: 652)  
 00910282 DIALOG File 652: U.S. Patents-Full Text (1971-79)  
 Utility

PATENT NO.: 4,034,136  
ISSUED: July 05, 1977 (19770705)  
INVENTOR(s): Wright, Carl Leonard, Pasadena, MD (Maryland), US (United States of America)  
Beacham, Harry Hoyt, Severna Park, MD (Maryland), US (United States of America)  
ASSIGNEE(s): FMC Corporation, (A U.S. Company or Corporation), Philadelphia, PA (Pennsylvania), US (United States of America)  
APPL. NO.: 5-575,515  
FILED: May 08, 1975 (19750508)

This is a division, of application Ser. No. 95,418, now U.S. Pat. No. 3,936,414, filed Dec. 4, 1970, which is a continuation-in-part of Ser. No. 837,911, filed June 30, 1969, now abandoned.

FULL TEXT: 1691 lines

#### ABSTRACT

This specification discloses that certain polychlorinated or polybrominated aromatic hydrocarbon compounds can be incorporated in thermosetting polymer compositions to make the thermosetting compositions flame retardant without injuring the desirable physical and electrical properties of the thermosetting compositions when in the thermoset state. These polyhalogenated aromatic hydrocarbons are apparently caused to react into the thermosetting composition, provided there is a minimum of 1 mol of a polyunsaturated monomer containing carbon to carbon unsaturation for each 3 mols of polychlorinated aromatic hydrocarbons. The thermosetting compositions generally contain a polyunsaturated monomer, a polyunsaturated polymer with carbon to carbon double bond unsaturation or a polyphenylene ether polymer which may be used in place of some or all of the polyunsaturated polymer, and a polyhalogenated aromatic hydrocarbon, containing at least 50% by weight combined chlorine or bromine and whose molecular weight is greater than 200. These thermosetting compositions can be heat cured or thermoset by a free radical initiator at a temperature of at least 130 degree(s) C. and can contain pigments, reinforcing fibers, release agents, coupling agents, and other incidental amounts of additives commonly used in thermosetting resin compositions.

...5460)  
50 45 40 60 65 60 -- -- 40 74 87 78  
Chlorinated biphenyl,  
68% chlorine (Aroclor 1268)  
-- -- -- -- -- 62 62 -- -- -- --  
Diallyl phthalate  
-- -- -- -- -- 40 -- 38 -- -- -- --  
Diallyl isophthalate  
50 55 60 40 35 -- 38 -- -- -- --  
Diallyl

3/4/5 (Item 4 from file: 652)  
00861778 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
POLYAMIDE-IMIDES

PATENT NO.: 3,988,374  
ISSUED: October 26, 1976 (19761026)  
INVENTOR(s): Brode, George L., Piscataway, NJ (New Jersey), US (United States of America)  
Kawakami, James H., Piscataway, NJ (New Jersey), US (United States of America)  
ASSIGNEE(s): Union Carbide Corporation, (A U.S. Company or Corporation), New York, NY (New York), US (United States of America)  
APPL. NO.: 5-182,526  
FILED: September 21, 1971 (19710921)  
FULL TEXT: 993 lines

A series of polyamide-imides which are both tractable and soluble in polar solvents as well as stable at high temperatures has been prepared by the condensation polymerization of aromatic diamines or oligomers containing oxygen, sulfone, and optionally alkylidene linkages with trimellitic acid, trimellitic acid anhydride or trimellitic anhydride acid chloride.

...was removed until the pot temperature reached 135 degree(s) C.

A concentrated solution of Aroclor 1268 (453 grams, 1.0 mole) in 500 ml. of hot toluene was added via an...

3/4/6 (Item 5 from file: 652)  
00819147 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
SEALING ADHESIVE STRANDS AND COMPOSITIONS THEREFOR

PATENT NO.: 3,945,975  
ISSUED: March 23, 1976 (19760323)  
INVENTOR(s): Strack, Donald Robert, Dayton, OH (Ohio), US (United States of America)  
ASSIGNEE(s): Protective Treatments, Inc, (A U.S. Company or Corporation), Dayton, OH (Ohio), US (United States of America)  
APPL. NO.: 5-101,084  
FILED: December 23, 1970 (19701223)

This application is a continuation-in-part of my applications Ser. No. 551,878, now abandoned, filed May 23, 1966 for GLAZING COMPOSITIONS; Ser. No. 611,129 filed Jan. 23 1967, for MOUNTING UNIT EMPLOYING DUAL GASKET CONSTRUCTION now U.S. Pat. No. 3,478,475; Ser. No. 607,022 filed Jan. 3, 1967 for SELF SUPPORTING, NON-LOAD BEARING RESILIENT TAPE SEALANT now U.S. Pat. No. 3,500,603, and Ser. No. 738,548, now abandoned, filed June 20, 1968 for ADHESIVE SEALING GLAZING COMPOSITION, and the disclosures of such applications are incorporated herein by reference.

FULL TEXT: 741 lines

#### ABSTRACT

New sealing adhesive bed compositions for glazing are form retaining and comprise elastomeric material initially mixed with plasticizers of low volatility and finely divided solids which either are of fibrous form or produce thixotropic mixtures with the plasticizers. A ratio range of from 3/4 to 21/2 parts non-volatile plasticizers to one part elastomeric material, and from 0.3 to 21/2 parts such finely divided solids to one part elastomeric material is present. Other kinds of finely divided solids and other ingredients may be present. The glazing strand is formed by extrusion, and the formed strand is not cured after extrusion. The tapes permanently deform under pressure when the glass is installed, have low spring back and exhibit little or no creep or cold flow in service.

... the elastomer art, such as polychlorinated polyphenyls known under the commercial names "Aroclor 1254" and "Aroclor 1268" and the ester plasticizers for example, dioctyl phthalate, dioctyl sebacate, butyl oleate, and liquid wood...

3/4/7 (Item 6 from file: 652)  
00812483 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
VEHICLE LAMP CONSTRUCTION

PATENT NO.: 3,939,337  
ISSUED: February 17, 1976 (19760217)

America)  
De Frayne, Garry O., Pontiac, MI (Michigan), US (United States of America)  
Walker, Paul L., Royal Oak, MI (Michigan), US (United States of America)

ASSIGNEE(s): Chrysler Corporation, (A U.S. Company or Corporation),  
Highland Park, MI (Michigan), US (United States of America)

APPL. NO.: 5-422,808

FILED: December 07, 1973 (19731207)

FULL TEXT: 256 lines

#### ABSTRACT

A lamp assembly wherein the lens portion is secured to the lamp housing by means of an electromeric tape. The bonding tape replaces the gasket, gasket cement and retaining screws conventionally employed in current lamp assemblies.

...polymer.

Examples of other suitable plasticizers are certain polychlorinated polyphenyls known commercially as "Aroclor" 1254, Aroclor 1268, low molecular weight polyisobutylene ("Vistanex" LM-MS), medium viscosity propylene polymer (Polypropene C-175 of...

... tackifiers in the compositions. Examples of suitable tackifiers are chlorinated biphenyls illustrated by Aroclor 1254, Aroclor 1268 (which Aroclors also function as plasticizers) and Aroclor 1260, non-reactive polymethylol phenol resin (commercially...

3/4/8 (Item 7 from file: 652)

00809550 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility

FLAME-RETARDANT RESIN COMPOSITIONS

PATENT NO.: 3,936,414

ISSUED: February 03, 1976 (19760203)

INVENTOR(s): Wright, Carl Leonard, Pasadena, MD (Maryland), US (United States of America)  
Beacham, Harry Hoyt, Severna Park, MD (Maryland), US (United States of America)

ASSIGNEE(s): FMC Corporation, (A U.S. Company or Corporation), Philadelphia, PA (Pennsylvania), US (United States of America)

APPL. NO.: 5-95,418

FILED: December 04, 1970 (19701204)

#### CROSS REFERENCE TO RELATED APPLICATION

This application is a continuation-in-part of U.S. patent application Ser. No. 837,911, filed June 30, 1969, entitled "Flame-Retardant Resin Compositions", now abandoned.

FULL TEXT: 1564 lines

#### ABSTRACT

This specification discloses that certain polychlorinated or polybrominated aromatic hydrocarbon compounds can be incorporated in thermosetting polymer compositions to make the thermosetting compositions flame retardant without injuring the desirable physical and electrical properties of the thermosetting compositions when in the thermoset state. These polyhalogenated aromatic hydrocarbons are apparently caused to react into the thermosetting composition, provided there is a minimum of 1 mol of a polyunsaturated monomer containing carbon to carbon unsaturation for each 3 mols of polychlorinated aromatic hydrocarbons. The thermosetting compositions generally contain a polyunsaturated monomer, a polyunsaturated polymer with carbon to carbon double bond unsaturation or a polyphenylene

polyunsaturated polymer, and a polyhalogenated aromatic hydrocarbon, containing at least 50% by weight combined chlorine or bromine and whose molecular weight is greater than 200. These thermosetting compositions can be heat cured or thermoset by a free radical initiator at a temperature of at least 130 degree(s) C, and can contain pigments, reinforcing fibers, release agents, coupling agents, and other incidental amounts of additives commonly used in thermosetting resin compositions.

...5460)

	50	45	40	60	65	60	--	--	40	74	87	78
Chlorinated biphenyl, 68% chlorine (Aroclor 1268 )	--	--	--	--	--	--	62	62	--	--	--	--
Diallyl phthalate	--	--	--	--	--	40	--	38	--	--	--	--
Diallyl isophthalate	50	55	60	40	35	--	38	--	--	--	--	--
Diallyl												

3/4/9 (Item 8 from file: 652)  
00772113 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
POLYAMIDE-IMIDES

PATENT NO.: 3,897,497  
ISSUED: July 29, 1975 (19750729)  
INVENTOR(s): Brode, George Lewis, Somerville, NJ (New Jersey), US (United States of America)  
Kawakami, James Hajime, Piscataway, NJ (New Jersey), US (United States of America)  
ASSIGNEE(s): Union Carbide Corporation, (A U.S. Company or Corporation), New York, NY (New York), US (United States of America)  
APPL. NO.: 5-456,805  
FILED: April 01, 1974 (19740401)

This is a division of Ser. No. 182,526 filed Sept. 21, 1971, now U.S. Pat. No. 3,817,921, which is in turn a continuation-in-part of Ser. No. 50,968 filed June 29, 1970, now abandoned.

FULL TEXT: 998 lines

#### ABSTRACT

A series of polyamide-imides which are both tractable and soluble in polar solvents as well as stable at high temperatures has been prepared by the condensation polymerization of aromatic diamines or oligomers containing oxygen, sulfone, and optionally alkylidene linkages with trimellitic acid, trimellitic acid anhydride or trimellitic anhydride acid chloride.

...was removed until the pot temperature reached 135 degree(s) C.

A concentrated solution of Aroclor 1268 (453 grams, 1.0 mole) in 500 ml. of hot toluene was added via an...

3/4/10 (Item 9 from file: 652)  
00770539 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
METHOD OF INHIBITING ALGAE EMPLOYING NITROGEN DERIVATIVES OF HALOGENATED BIPHENYLS

PATENT NO.: 3,895,932  
ISSUED: July 22, 1975 (19750722)

States of America)  
Shay, Edward Griffin, Suffern, NY (New York), US (United States of America)  
Adams, Phillip, Murray Hill, NJ (New Jersey), US (United States of America)  
Petrocci, Alfonso N., Glen Rock, NJ (New Jersey), US (United States of America)

ASSIGNEE(s): Millmaster Onyx Corporation, (A U.S. Company or Corporation),  
New York, NY (New York), US (United States of America)  
APPL. NO.: 5-349,630  
FILED: April 09, 1973 (19730409)

This is a continuation-in-part of application Ser. No. 38,513 filed May 18, 1970, now issued as U.S. Pat. No. 3,733,421 dated May 15, 1973 said application being itself a continuation-in-part of application Ser. No. 883,636, filed Dec. 9, 1969 and issued as U.S. Pat. No. 3,663,620 on May 16, 1972.

FULL TEXT: 193 lines

#### ABSTRACT

Microbiocidal bis-amino substituted halogenated biphenyls, especially adapted for use as algacides.

...range of polychlorinated biphenyls, e.g. "Aroclor 1260" (said to contain 60% of chlorine) and "Aroclor 1268" (said to contain 68% of chlorine). Others with other chlorine content, especially "Aroclors" 1232, 1242...

3/4/11 (Item 10 from file: 652)  
00769666 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
POLYAMIDE-IMIDES

PATENT NO.: 3,895,064  
ISSUED: July 15, 1975 (19750715)  
INVENTOR(s): Brode, George Lewis, Somerville, NJ (New Jersey), US (United States of America)  
Kawakami, James Hajime, Piscataway, NJ (New Jersey), US (United States of America)  
ASSIGNEE(s): Union Carbide Corporation, (A U.S. Company or Corporation),  
New York, NY (New York), US (United States of America)  
APPL. NO.: 5-456,836  
FILED: April 01, 1974 (19740401)

This is a division on Ser. No. 182,526 filed Sept. 21, 1971, now U.S. Patent 3,817,921, which is in turn a continuation-in-part of Ser. No. 50,968 filed June 29, 1970, now abandoned.

FULL TEXT: 1013 lines

#### ABSTRACT

A series of polyamide-imides which are both tractable and soluble in polar solvents as well as stable at high temperatures has been prepared by the condensation polymerization of aromatic diamines or oligomers containing oxygen, sulfone, and optionally alkylidene linkages with trimellitic acid, trimellitic acid anhydride or trimellitic anhydride acid chloride.

...was removed until the pot temperature reached 135 degree(s) C.

A concentrated solution of Aroclor 1268 (453 grams, 1.0 mole) in 500 ml. of hot toluene was added via an...

Utility  
POLYARYLIMIDES

PATENT NO.: 3,839,287  
 ISSUED: October 01, 1974 (19741001)  
 INVENTOR(s): Kwiatkowski, George T., Greenbrook, NJ (New Jersey), US  
 (United States of America)  
 Brode, George L., Somerville, NJ (New Jersey), US (United States of America)  
 ASSIGNEE(s): Union Carbide Corporation, (A U.S. Company or Corporation),  
 New York, NY (New York), US (United States of America)  
 APPL. NO.: 5-362,287  
 FILED: May 21, 1973 (19730521)  
 This is a continuation-in-part of Ser. No. 182,527 filed Sept. 21, 1971 now abandoned.  
 FULL TEXT: 655 lines

## ABSTRACT

Arylimides have been prepared from oligomer diamines and polyamines containing halogenated phenylene, oxygen, alkylidene and/or sulfone moieties and maleic anhydride. The resultant arylimides, in the form of B-stage resins can be used for the preparation of glass cloth prepreps, molding materials and adhesives which can be thermally converted to the thermoset state.

...was removed until the pot temperature reached 135 degree(s) C.

A concentrated solution of Aroclor 1268 (453 grams, 1.0 mole) in 500 ml. of hot toluene was added via an...

3/4/13 (Item 12 from file: 652)  
 00689466 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
FLAME-RESISTANT RESISTOR COATINGS

PATENT NO.: 3,804,669  
 ISSUED: April 16, 1974 (19740416)  
 INVENTOR(s): Bockstie, Jr. Lawrence G., Bradford, PA (Pennsylvania), US  
 (United States of America)  
 ASSIGNEE(s): Corning Glass Works, (A U.S. Company or Corporation), Corning,  
 NY (New York), US (United States of America)  
 APPL. NO.: 5-196,787  
 FILED: October 05, 1971 (19711005)

This is a division of application Ser. No. 652,412, filed July 11, 1967.

FULL TEXT: 233 lines

## ABSTRACT

Flame resistant polyimide resistory coatings containing chlorinated polyphenyl and antimony trioxide.

... from 18 to 66 percent chlorine. Useful chlorinated polyphenyl resins include Aroclor 1260, Aroclor 1262, Aroclor 1268, Aroclor 1270, Aroclor 4465, Aroclor 5442, Aroclor 2565, and Aroclor 5460, the most preferred being...

3/4/14 (Item 13 from file: 652)  
 00652180 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
-(NONACHLOROBIPHENYL)AMINOPROPYL-N'-DIMETHYLAMINE



ISSUED: September 18, 1973 (19730918)  
INVENTOR(s): Merianos, John J., Jersey City, NJ (New Jersey), US (United States of America)  
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APPL. NO.: 5-235,064  
FILED: March 15, 1972 (19720315)

This is a division of co-pending application Ser. No. 883,636, filed Dec. 9, 1969 and now U.S. Pat. No. 3,663,620.  
FULL TEXT: 448 lines

#### ABSTRACT

Microbiocidal ammonia and amino substituted halogenated biphenyls, as well as the nitrogen derivatives thereof such as quaternary ammonium compounds, amine oxides, imidazolines, amides, enamines, ampholytes, and the like.

The ammonia and amino halobiphenyls are prepared by reacting the ammonia or the amine and the halobiphenyl reactants in such a manner that the ratio of amine to halobiphenyl is preferably limited to between two and five mols of amine to each mol of halobiphenyl.

...range of polychlorinated biphenyls, e.g. "Aroclor 1260" (said to contain 60% of chlorine) and "Aroclor 1268" (said to contain 68% of chlorine). Others with other chlorine content, especially "Aroclors" 1232, 1242... triamine.

#### EXAMPLE 2

In the apparatus of Example 1, 500 grams (or one mol) of "Aroclor 1268" (assaying 70% Cl) and 310 grams (or three mols) of diethylene triamine were reacted in...

3/4/15 (Item 14 from file: 652)  
00555594 DIALOG File 652: U.S. Patents-Full Text (1971-79)

Utility  
COATED ABRASIVE BELT OVERLAP JOINT

PATENT NO.: 3,637,359  
ISSUED: January 25, 1972 (19720125)  
INVENTOR(s): Malloy, John F., Waterford, NY (New York), US (United States of America)  
Seiler, Charles J., Elnora, NY (New York), US (United States of America)  
ASSIGNEE(s): Norton Company, Troy, NY (New York), US (United States of America)  
APPL. NO.: 5-  
FILED: January 06, 1970 (19700106)

#### CROSS-REFERENCE TO RELATED APPLICATIONS

This application is a continuation-in-part of our application, Ser. No. 514,360 filed Dec. 16, 1965, for Coated Abrasive Belt Joining, now abandoned.

FULL TEXT: 663 lines

#### ABSTRACT

[54] FLAME-RETARDANT RESIN  
COMPOSITIONS

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[73] Assignee: FMC Corporation, Philadelphia, Pa.

[22] Filed: Dec. 4, 1970

[21] Appl. No.: 95,418

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 837,911, June 30,  
1969, abandoned.

[52] U.S. CL..... 260/40 R; 106/15 FP; 260/42.18;  
260/869; 260/874; 260/DIG. 24

[51] Int. CL<sup>1</sup>..... C08L 71/04

[58] Field of Search..... 260/869, 874, 37 R, 42.18,  
260/861; 106/15 FP

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Primary Examiner—Donald E. Czaja  
Assistant Examiner—S. M. Person

[57] ABSTRACT

This specification discloses that certain polychlorinated or polybrominated aromatic hydrocarbon compounds can be incorporated in thermosetting polymer compositions to make the thermosetting compositions flame retardant without injuring the desirable physical and electrical properties of the thermosetting compositions when in the thermoset state. These polyhalogenated aromatic hydrocarbons are apparently caused to react into the thermosetting composition, provided there is a minimum of 1 mol of a polyunsaturated monomer containing carbon to carbon unsaturation for each 3 mols of polychlorinated aromatic hydrocarbons. The thermosetting compositions generally contain a polyunsaturated monomer, a polyunsaturated polymer with carbon to carbon double bond unsaturation or a polyphenylene ether polymer which may be used in place of some or all of the polyunsaturated polymer, and a polyhalogenated aromatic hydrocarbon, containing at least 50% by weight combined chlorine or bromine and whose molecular weight is greater than 200. These thermosetting compositions can be heat cured or thermoset by a free radical initiator at a temperature of at least 130°C, and can contain pigments, reinforcing fibers, release agents, coupling agents, and other incidental amounts of additives commonly used in thermosetting resin compositions.

7 Claims, No Drawings

## FLAME-RETARDANT RESIN COMPOSITIONS

### CROSS REFERENCE TO RELATED APPLICATION

This application is a continuation-in-part of U.S. patent application Ser. No. 837,911, filed June 30, 1969, entitled "Flame-Retardant Resin Compositions", now abandoned.

### BACKGROUND OF THE INVENTION

#### A. Field of the Invention

This invention relates to thermosetting polymerizable compositions and particularly to flame-retardant thermosetting molding compositions.

#### B. Description of the of the Prior Art

Thermosetting polymerizable compositions, commonly termed thermosetting resins, are widely used in making castings, laminates, and molded articles. The high strength to weight ratios of thermoset resins when properly reinforced and cured and the resistance of these compositions to environmental conditions have suggested the application of thermoset compositions in numerous applications, including many applications where combustibility is a serious consideration.

A number of methods have been employed to reduce the rate of combustion of thermoset resin compositions. These methods have included the use of numerous flame-retardant additives, including inorganic additives, organic additives, a combination of inorganic and organic additives, the addition of a flame-retardant element that reacts and becomes part of the thermoset resin, and combinations of all these methods.

The use of inorganic fillers to impart flame retardance is attractive on a cost basis. Typical inert flame-retardant fillers retard the burning rate to the extent that they act as diluents of the combustible components. However, large amounts of fillers adversely affect the electrical properties of thermoset resin compositions.

Organic compounds containing halogen and phosphorus atoms such as chlorinated paraffins, chlorinated biphenyls, chlorinated aryl substituted alkyls, phosphorus containing hydrocarbons, and the like have been used for many years to impart flame resistance to plastic compositions including thermoset resins. The chlorinated materials are quite effective as flame retardants. The flame-retarding mechanism is believed to involve the release of hydrogen chloride at combustion temperatures which snuff out the flame by exclusion of oxygen. Only highly chlorinated organic materials have been considered, since the objective is to provide maximum flame resistance with a minimum of additive material. These highly chlorinated organics, including those which are solid, have a plasticizing action or thermoset compositions. Generally the plasticizing action of the chlorinated organic compounds lowers the heat deflection temperature and otherwise impairs the desirable properties of cured thermosetting resins.

Combinations of chlorinated organic additives with antimony trioxide have been known for a number of years. The effect of certain other metal oxides, such as magnesia or hydrated alumina, combined with antimony oxides in flameproofing chlorinated polymers, was noted by D. C. Thompson et al. in an article entitled "Flame Resistance of Neoprene" appearing in the August 1968 issue of *Rubber Age*. Combinations of hydrated alumina with antimony oxide and chlorinated organics are widely recognized as effective flame retar-

dants for resinous compositions including thermoset resin compositions.

Satisfactory flame resistance in thermoset polymers has been obtained by using reactive chlorinated intermediates which become a part of the cured thermoset system, thus avoiding the plasticizing effect experienced when using chlorinated paraffins, chlorinated polyphenyls and the like. An important advance in the chemistry of self-extinguishing or flame-retardant thermoset resins occurred as a result of the discovery of a commercial method of making chlorendic acid or anhydride in 1952. Chlorendic acid and anhydride have been esterified to form polyesters similar to those obtained when using phthalic anhydride. Additionally, chlorendic acid and anhydride have been used to prepare reactive monomers, such as diallyl chlorendate, which are useful in flameproofing thermosetting resins such as polyesters and diallylic phthalates without substantially reducing the desirable physical properties of the thermoset resin compositions.

The addition of a reactive flame-retardant additive to thermoset resins such as polyesters and diallylic phthalate resins is highly desirable, as this method of flameproofing offers flexibility in resin production, since it is possible to flameproof general-purpose resins rather than to make special flame-retardant grade resins. The chlorendic acid-based reactive flame-retardant materials are expensive and since 1952 no efficient and inexpensive reactive flame retardants for thermoset resins have been widely accepted. The principal object of this invention is the preparation of highly flame-retardant, inexpensive, thermosetting resin compositions.

### SUMMARY OF THE INVENTION

We have now discovered that certain polychlorinated and polybrominated aromatic hydrocarbon compounds can be incorporated in thermosetting polymer compositions to make the compositions flame retardant without injuring the desirable physical and electrical properties of the thermosetting compositions when in the thermoset state. We have found that this result is achieved by adding at least 1 mol of a polyunsaturated monomer with carbon to carbon unsaturation to the thermosetting composition for each 3 mols of polychlorinated or polybrominated aromatic hydrocarbon added to the thermosetting composition.

These thermosetting compositions generally comprise: (a) 100 parts by weight of the blend of about 10 to about 70 parts of a polyunsaturated monomer with carbon to carbon unsaturation, with about 90 to about 30 parts of a polymer of the class consisting of a polyunsaturated polymer with carbon to carbon unsaturation and polyphenylene ether polymers, and (b) about 5 to 50 parts by weight per 100 parts of a of a polychlorinated or polybrominated aromatic hydrocarbon containing at least 50% by weight of combined chlorine or bromine and whose molecular weight is greater than 200, in an amount not in excess of 3 mols of polyhalogenated aromatic hydrocarbon for each mol of the polyunsaturated monomer in a. These novel thermosetting compositions can also include pigments, fillers, reinforcing fibers, release agents, coupling agents and other incidental additives in amounts commonly used in thermosetting resin compositions.

We have also discovered that where high monomer levels cannot be tolerated, as in high-pressure molding compounds, it is possible to prereact the polychlorinated or polybrominated compound with mono-

mer prior to adding the polyhalogenated aromatic hydrocarbon in the thermosetting composition. The polyhalogenated aromatic compound is pre reacted with the monomer by heating 3 to 2 mols of the polyhalogenated aromatic hydrocarbon with 2 to 3 mols of the polyunsaturated monomer containing carbon to carbon unsaturation at a temperature of at least 130°C in the presence of a free radical initiator for a period of time sufficient to initiate an exothermic reaction between the polyhalogenated aromatic compound and the monomer, generally about 2 to about 10 minutes. These reaction products can be added to thermosetting compositions and thermoplastic compounds as flame-retardant additives.

Another phase of our invention is the discovery that certain of our novel compositions adhere strongly to a wide variety of substrates including metals. The compositions of this invention which are useful as thermosetting coatings generally comprise: (a) 10 to 50 parts by weight of a polyunsaturated monomer containing carbon to carbon unsaturation, (b) 5 to 30 parts by weight of a polychlorinated or polybrominated aromatic hydrocarbon containing at least 50% by weight of chlorine or bromine and having a molecular weight of at least 200, the ratio of monomer to polyhalogenated aromatic hydrocarbon being at least 1 to 3 respectively, (c) 20 to 60 parts by weight of a polyphenylene ether polymer, and (d) 0 to 30 parts by weight of a polyunsaturated polymer containing carbon to carbon unsaturation. Because it is not necessary to dissolve the polyphenylene ether polymer portion of these coating compositions, the compositions may be dispersed in a liquid for ease of application, thus making it possible to prepare very high solids coating compositions containing as much as 70% solids. The compositions are useful for coating articles to seal them against moisture, as coating compositions for impregnating glass fabric and the like for making laminates, and because they adhere well to metal, they are useful in adhering metal to metal and metal to plastics such as in preparing a copper clad glass laminate.

All of our novel thermosetting compositions can be thermoset by free radical initiators at temperatures of at least 130°C for a period of time sufficient to initiate an exothermic reaction during which the polychlorinated or polybrominated aromatic compound reacts into the other components of the compositions and loses its thermoplastic or plasticizing properties.

#### DESCRIPTION OF THE INVENTION AND THE PREFERRED EMBODIMENTS

The polychlorinated or polybrominated aromatic hydrocarbons containing at least 50% by weight combined chlorine and whose molecular weight is greater than 200, useful in practicing this invention, include, but are not limited to, compounds such as chlorinated and brominated biphenyls and terphenyls, trichlorobenzene and higher chlorinated benzenes and alkyl substituted benzenes, chlorinated naphthalenes, and the like.

Polyunsaturated polymers useful in practicing this invention include, but are not limited to, diallylic phthalate prepolymers and polyester resins. Diallylic phthalate prepolymers useful in practicing this invention include prepolymers made from the diallylic esters of ortho-, iso-, and terephthalic acids. These diallylic phthalates may be manufactured by polymerizing a monomeric material to produce a solution of soluble

prepolymer in monomer. Polymerization is carried to a point short of gelation. The prepolymer must then be separated from the unpolymerized monomer. This may be done by treatment with a solvent which dissolves the monomer and precipitates the prepolymer. Such a general process is described by Heilberger in U.S. Pat. No. 3,096,310. A conventional method of separating allylic prepolymer from monomer by precipitating the prepolymer in an unreactive liquid precipitant that is a solvent for the monomer and a nonsolvent for the prepolymer in a shearing zone is described by Willard in U.S. Pat. No. 3,030,341. Prepolymers may also be separated from unpolymerized monomer by distillation as disclosed by Mednick et al. in U.S. Pat. No. 3,285,836 issued May 28, 1968. The diallyl phthalate prepolymers are solids containing little or no monomer; they can be stored indefinitely in this form, since they require catalysts and either heat, actinic light or nuclear particle radiation to convert them to the insoluble or thermoset stage.

The polyester resins useful in practicing this invention, whether liquid or solid, should be of the reactive type, that is, at least about 50 mol percent of the dibasic acid portion of the polyester resin should be an unsaturated dibasic acid such as maleic or fumaric acid. If these polyester resins are diluted or cut with a monomer, the polyester resin should preferably be cut with a polyfunctional monomer. The alcohol moiety of the polyester is generally a saturated, difunctional glycol containing 2 to 8 carbon atoms which may be cyclic or acyclic.

A reactive polyester may be prepared by reacting equimolar amounts of maleic anhydride and diethylene glycol at a temperature about 200°C. Water is azeotropically removed. The distillate is analyzed from time to time for starting ingredients, and a sufficient amount of material may be added to the reactor to maintain the initial proportions of the reacting ingredients. After 8 hours' reaction at such a temperature, a polyester is obtained in the form of a viscous liquid with an acid number of about 18.

A solid reactive polyester can be prepared by reacting equimolar amounts of a mixture of maleic anhydride and isophthalic acid with a 10% excess of propylene glycol. Water is removed azeotropically. The reaction is continued by heating until the acid number of the reaction mixture falls below 5. Excess unreacted propylene glycol is removed under vacuum and a solid polyester is obtained. Polyesters made from chlorendic anhydride and equimolar amounts of maleic anhydride or fumaric acid are well known reactive flame-retardant polyesters that can be used in practicing this invention.

Polyfunctional monomers useful in practicing this invention include, but are not limited to, diallyl phthalate, diallyl isophthalate, diallyl chlorendate, diallyl maleate, triallyl cyanurate, triallyl isocyanurate, glycol dimethacrylate, divinyl benzene and the like. So far as it is known to the inventors, any polyfunctional unsaturated monomer may be used in practicing this invention.

The polyunsaturated materials and polyhalogenated aromatic hydrocarbons can be either compounded into a thermosetting composition, such as a molding compound, in place of part of the conventional thermosetting polymer, or can be pre reacted to form a kind of gel polymer by mixing about 3 to 2 mols of polyhalogenated compounds with 2 to 3 mols of a polyunsaturated

monomer and subjecting the mixture to a free radical initiator, by incorporating 1 to 5% by weight, preferably about 1.5 to 3% by weight, of a peroxide catalyst in such a composition.

The preferred ratio of polyhalogenated aromatic compounds to polyunsaturated monomer, when using the prereacted materials, is 1 mol of polyhalogenated aromatic compound to each mol of polyunsaturated monomer. The free radical initiation should take place at a temperature of at least 130°C and below the boiling point of the monomer. The reaction is exothermic and the temperature attained during the exothermic reaction period of a 100 g mass should be between about 200 and about 250°C under adiabatic conditions. Generally the reaction mixture must reach a temperature of at least 130°C for 2 to 5 minutes to initiate the exothermic reaction. Preferably the polyhalogenated aromatic hydrocarbon and the polyunsaturated monomer are selected so that a hard, grindable reaction product is produced. Fillers may be incorporated in the prereaction mixture, provided that the filler does not cause initiation of the reaction at less than 130°C or inhibit reaction about 130°C. Inert fillers can comprise up to 60% by weight of the total composition.

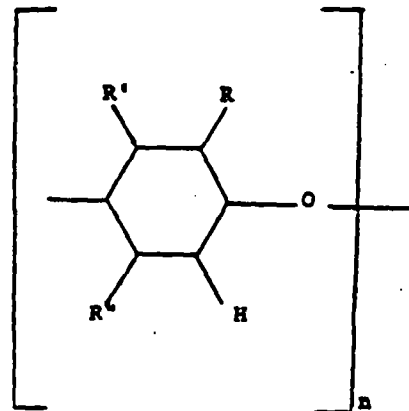
Typical polychlorinated aromatic hydrocarbons used in the practice of this invention are chlorinated biphenyls and terphenyls which are known to be so stable and chemically inactive that they are used as plasticizers, heat exchange fluids and the like. Diallyl phthalate resins and polyester resins, which are typical of the polyunsaturated materials used in practicing this invention, when cured, are known to be plasticized by chlorinated biphenyls and terphenyls. For example, ordinary molded diallyl phthalate articles containing chlorinated biphenyls do not have the characteristic "hot hardness" of diallyl phthalate molded articles; when removed from a hot mold, the molded articles containing a chlorinated biphenyl are soft, like hot rubber, and harden only on cooling. Physical mixtures of highly chlorinated biphenyls (some with melting points above 150°C) and an allylic prepolymer ball-milled together "ball up" or stick together in the ball mill (though either material alone will ball-mill in a conventional matter), thus showing solvation of resin by the polychlorinated aromatic compound. It is therefore quite surprising that, when there is at least 1 mol of a polyunsaturated monomer for every 3 mols of the polychlorinated aromatic compound, these polychlorinated aromatic hydrocarbons can be incorporated into a diallylic phthalate prepolymer or reactive polyester resin molding composition, which can be cured into articles that are essentially not plasticized by the polychlorinated aromatic compound.

The polyhalogenated aromatic hydrocarbon and polyunsaturated materials do not have to be prereacted. These materials may be incorporated into thermosetting resin compositions which are used for making moldings, castings, and laminates without substantially degrading the cured physical properties of the thermosetting resins, provided there is at least 1 mol of difunctional monomer for every 3 mols of polyhalogenated aromatic hydrocarbon in the thermosetting resin composition and the curing temperature is at least 130°C. Surprisingly, when cured to the thermoset stage, these novel thermosetting resin compositions exhibit excellent electrical and mechanical properties and are flame resistant. The polychlorinated or polybrominated aromatics have little or no plasticizing ef-

fect upon the cured thermoset compositions and there is substantially no loss of thermoset properties.

Polychlorinated and polybrominated aromatic hydrocarbons are commercially available and represent a low-cost halogen source for making resistant thermoset resin compositions. Surprisingly, compared to the more expensive flame-retardant compositions made with polyester or diallylic phthalate resins and diallyl chlorinate, the compositions of this invention have somewhat improved electrical properties. These novel compositions can be used with ordinary flame-retardant additives and synergists used in chlorinated flame-retardant resin compositions such as antimony trioxide and hydrated alumina.

Polyphenylene ether polymers can be used to replace some or all of the polyunsaturated polymers in these novel compositions, provided at least an additional 0.05 part by weight of polyunsaturated monomer is added to the composition for each part of polyphenylene ether polymer. Typical polyphenylene ether polymers have a repeating structural unit of the formula



wherein the oxygen atom of one unit is connected to the benzene nucleus of the adjoining unit,  $n$  is a positive integer and is at least 10,  $R$  is a monovalent substituent selected from the group consisting of hydrogen, hydrocarbon radicals free of tertiary alpha-carbon atom, halohydrocarbon radicals having at least two carbon atoms between the halogen atom and the phenol nucleus and being free of a tertiary alpha-carbon atom, hydrocarbonoxy radicals being free of a tertiary alpha-carbon atom, and halohydrocarbonoxy radicals having at least two carbon atoms between the halogen atom and phenol nucleus and being free of tertiary alpha-carbon atoms,  $R'$  and  $R''$  are both monovalent substituents which are the same as  $R$  and, in addition, halogen. All of the polyphenylene ether polymers currently available have been found to be useful in practicing this invention. A method of producing polyphenylene ether polymers by forming self-condensation products of phenylene by reacting oxygen with a phenol is described in U.S. Pat. No. 3,306,875 issued Feb. 28, 1967. We have used polyphenylene ether polymers available from the General Electric Company, such as Noryl and Grades 631-101, 691-111 and 631-111, and found them all to be useful in practicing this invention. As far as we know, all of the polyphenylene ether polymers known in the art are useful in practicing this invention.

The novel compositions of this invention employ a free radical initiator in sufficient amounts to convert the (polyunsaturated monomer, polyunsaturated polymer and polychlorinated aromatic hydrocarbon) compositions to the thermoset state upon the application of heat. Free radical initiation may be accomplished by electron beams or other sources of radiation including actinic light. Conveniently, peroxide catalysts which promote reaction between unsaturated compounds with carbon to carbon double bond unsaturation may be used. The peroxide catalyst does not have to exclude homopolymerization, but must be a catalyst that does not produce only homopolymers. We have found that when peroxide catalysts and heat are used to cure the compositions, peroxides which have a minimum 10 hour half life in benzene at a temperature in excess of 100°C should be used to some extent to catalyze the reactions of this invention i.e. 2-10 parts by weight per 100 parts of polymer-monomer-halogenated aromatic hydrocarbon mixture. Mixed catalysts may be used, but at least part of the catalyst must be a catalyst having a 10 hour half life at a temperature in excess of 100°C in benzene. We have successfully used dicumyl peroxide, tertiary butyl perbenzoate, 2,5-dimethyl-2,5-di(tert-butylperoxy)hexyne-3 and 2,5-dimethyl-2,5-di(tert-butylperoxy)hexane alone and in combination with benzoyl peroxide. We have found that dicumyl peroxide gives the best results in the novel compositions of this invention. Generally, catalysts well known in the art to cure allylic compositions are useful in practicing this invention.

A wide variety of water-insoluble, inert fillers may be used in preparing the molding compounds of this invention. Useful fillers include calcium carbonate, both precipitated and wet ground types, calcium silicate (wollastonite), silica, hydrated clays, calcined clays, chalk, calcium sulfate (anhydrous), barium sulfate, asbestos, glass (powdered), quartz, aluminum trihydrate, aluminum oxide, antimony oxide, magnesium oxide, inert iron oxides and groundstone such as granite, basalt, marble, limestone, sandstone, phosphate rock, travertine, onyx and bauxite. Additionally, inert fibrous materials may be used such as synthetic fibers, glass fibers, asbestos and cellulosic fibers. Up to 200 parts by weight of filler and/or fiber per 100 parts by weight of polyunsaturated polymerizable materials-polychlorinated aromatic compound may be used in these molding compositions.

Incidental additives widely incorporated in thermosetting molding compounds and laminates such as pigments, release agents, coupling agents, lubricants and other incidental additives may be incorporated in the compositions of this invention.

The novel molding compositions of this invention are prepared in conventional equipment and using techniques well known in the plastics industry to be useful in compounding allylic, epoxy, and polyester molding compounds. The molding compounds may be filled or unfilled and of the premixed, powdered, granular or dough type. The polyfunctional polymerizable materials, including polymer and monomer, a polyhalogenated aromatic hydrocarbon, free radical catalyst and an internal mold release, pigments, inhibitor, and so forth are simply mixed together in a heavy-duty mixer. The mixing may be done with or without the use of solvents. However, if solvents are used, they should be removed from the premixed compound before molding. The molding compositions can be molded under conditions

normally used for allylic molding compositions, that is, they may be molded at from about 130° to 180°C for about 1 to 60 minutes. Because of the varied viscosities of these novel molding compositions, the molding pressure may vary from near (0) zero to 10,000 pounds per square inch, depending on the composition.

There are two convenient ways to add the polychlorinated or polybrominated aromatic hydrocarbons to the polyunsaturated polymerizable materials. The preferred method of addition is to add the polyhalogenated aromatic hydrocarbon with the polyunsaturated materials as any other additive might be added in a molding compound or laminating solution. Although not the preferred method of addition, the polyhalogenated aromatic hydrocarbon may be added after it has been prereacted with a polyfunctional monomer. This is particularly useful where large amounts of monomer cannot be tolerated, as in high-pressure molding compounds. Direct addition of the polyhalogenated aromatic compound in preparation of the molding compound can be made by simply adding a polyunsaturated monomer and a polyhalogenated aromatic hydrocarbon to a molding compound prior to or during compounding in place of part of the polymer which would normally be used in the molding compound, providing there is at least 1 mol by weight of difunctional monomer present for each 3 mols of polyhalogenated aromatic compound.

The polyunsaturated polymerizable materials and polyhalogenated hydrocarbons have been prereacted to form solid reaction products by heating together a mixture of about 3 to 2 mols of polyhalogenated aromatic hydrocarbon with 2 to 3 mols of a polyunsaturated monomer in the presence of about 2% by weight of dicumyl peroxide. A number of filled and unfilled reaction products were prepared in a sigma blade mixer equipped with a steam jacket. Steam at a temperature of 160°C (100 pounds of pressure) was circulated through the jacket. The reactants, and in some cases filler and catalyst, were simply added to the sigma blade mixer with 160°C steam being supplied to the steam jacket and reacted together with agitation. Although the exact temperatures of the mixer blade and side walls were not known and accurate time measurements could not be made, reasonable initiation times, generally less than 10 minutes, were obtained. During the processing, the mixtures were highly fluid up to reaction and proceeded through reaction rapidly to a brittle rubber stage, finally breaking into small lumps. No particular advantage was observed for filled compositions. All of the reactants evaluated appeared to have about the same rate of reaction and the reaction appeared to be complete in a matter of 2 to 5 minutes after initiation. The hard reaction products were ball-milled for a period of 2 hours to produce a finely ground, uniform powder. The ratio of polyunsaturated monomer to polychlorinated aromatic compound was varied over ratios from 3 to 2 mols of monomer per 2 to 3 mols of polychlorinated aromatic compound. These products, due to their high chlorine content, were found to impart flame resistance to thermosetting compounds. These novel solid reaction products may be used in thermoplastics such as polyethylene, polyvinyl chloride acrylics and polystyrene, as well as rubber and other organic systems including thermosetting materials such as epoxy resins, polyurethane resins, polyimide resins and the like, where flame resistance is desirable.

These solid reaction products can also be added to unsaturated polyester resin compositions containing a monounsaturated monomer, for example a "styrene cut polyester". The physical properties of styrene-type polyester molding compounds containing the solid reaction products of this invention exceed the military specification requirements for polyester molding compounds as set forth in mil M-14F specification for MAI 60 Styrene-Polyester Molding compounds.

The materials of this invention permit compound versatility that has not previously been available. The materials can be used in processing compositions which are to be subjected to either low-pressure or high-pressure thermosetting conditions. Where it is desirable to incorporate these flame-retardant materials into high-pressure molding compounds such as in diallyl phthalate systems, the solid reaction products described above are preferred. These materials in the form of a fine powder are easily dispersed in an allylic resin composition in place of part of the allylic prepolymer. A diallylic phthalate molding composition containing the flame-retardant reaction products described above is processed in the same manner as conventional diallyl phthalate molding compounds. Up to 50% of the diallylic phthalate prepolymer can be replaced by these solid reaction products. Where it is desirable to achieve maximum flame retardancy in a molding compound or laminate with a minimum amount of halogen, it is advisable to incorporate antimony oxide along with the halogen-containing reaction product. Metal oxides such as hydrated alumina may be used with the antimony oxide. It is generally advisable to use a sufficient amount of the reaction product in a molding compound or laminate so that there is about 10 to 20% halogen based on the weight of the total organic content of the molding compound or laminate for high levels of flame resistance. However, less halogen is required where low levels of flame resistance can be tolerated.

Where low pressure, long flow laminating, impregnating and molding compositions are desired, the polyhalogenated aromatic hydrocarbon can be incorporated directly in allylic compositions or polyester compositions, provided there is at least 1 mol by weight of a polyunsaturated monomer for each 3 mols of polyhalogenated aromatic hydrocarbon and a peroxide catalyst which will not initiate the free radical curing at less than 100°C.

Polyphenylene ether polymers can be used to replace up to all of the polyunsaturated polymer in these novel compositions. When some or all of the polyunsaturated polymer is replaced with polyphenylene ether polymer, the compositions can be used as coating compositions, as glue lines for adhering materials to reinforced polymeric substrates or adhering two materials together, for use in reinforced laminates and as potting and encapsulating compositions. Although all of the polyunsaturated polymer can be replaced with polyphenylene ether polymer, it is preferred to retain at least some of the polyunsaturated polymer in the compositions, as the presence of the polyunsaturated polymer improves the handling characteristics of the compositions.

The novel compositions of this invention are readily used in preparing fiber reinforced laminates, generally fiber glass reinforced, by either the wet lay-up for prepreg techniques. Wet fiber glass lay-ups are prepared by making a liquid blend of the polymerizable materials, i.e., a monomer such as diallyl orthophthalate, a polymer containing carbon to carbon unsaturation or a

polyphenylene ether polymer, a polyhalogenated aromatic compound, a catalyst, and other modifying ingredients such as dyes, pigments, fillers, inhibitors, glass coupling agents and so forth, which is poured onto one or more layers of a fibrous non-woven glass mat or woven glass fabric, which has preferably been treated with a glass coupling agent, to impregnate the reinforcing glass; after impregnation, the product is laminated under heat and mild pressures according to procedures well known in the art to be useful for curing allylic resin laminates.

A typical slow cure is effected by placing the wet lay-up in a vacuum bag and applying a vacuum of 28 to 30 inches of mercury for about 1 to about 5 hours to remove bubbles; the evacuated lay-up is then preferably augmented by autoclave pressure at 30 to 50 psi for 30 minutes at 82°C, 60 minutes at 120°C, 30 minutes at 150°C, 15 minutes at 160°C, and then cured an additional 60 minutes at 160°C. Thin sections can be cured more rapidly; for example, 30 to 50 psi for 60 minutes at 160°C. The amount of glass in the lay-up can be as high as 80%, although the preferred amount of reinforcing glass is 50 to 75% by weight of the laminate.

The novel resin compositions of this invention can be employed in the usual process for manufacture of fibrous reinforced thermoset resin laminates using the prepreg technique. A polymerizable liquid, i.e., monomer such as diallyl orthophthalate and the like, a polyhalogenated aromatic hydrocarbon, a polyphenylene ether polymer, and, where desired, a polymer containing carbon to carbon unsaturation such as an allylic prepolymer or a reactive type polyester resin, a catalyst and modifying ingredients such as dyes, pigments, fillers, glass coupling agents, inhibitors and so forth are mixed together and used to impregnate a fibrous non-woven mat or a woven fabric; where glass mats or fabrics are used, it may be desirable to have the glass treated with a glass coupling agent. The use of some solvent is usually required in order to reduce the viscosity level of the resin composition to make it suitable for application to the mat or fabric with conventional, commercial saturating or impregnating equipment.

It is not necessary to dissolve the polyphenylene ether polymer in the impregnating compositions of this invention. Simple uniform dispersion of the polyphenylene ether polymer powder in the solvent-monomer-polymerizable resin mixtures suffices. Prepregs are generally most economically processed with 30 to 60 parts of the resin composition dispersed in 70 to 40 parts of a suitable solvent such as acetone, methylethyl ketone, methyl isobutyl ketone, toluene, xylene, chloroform, methylene chloride, trichloroethylene, perchloroethylene and mixtures thereof and other solvents known in the trade to be useful in preparing allylic prepregs.

The mat or fabric is impregnated with the solvent solution and then dried to remove the solvent. After impregnation and drying of the impregnated fabric, the laminate is laid up and cured with heat and mild pressure using cure cycles and conditions similar to those used in curing the wet lay-up type laminates. Roving, including glass roving, is similarly pre-impregnated for processing by filament winding techniques into pipe, other cylindrical shapes and hollow tapered and conical shapes. Products made by filament winding are generally cured at about 150°C in 60 minutes. The fiber content of the prepreg laminates varies from about 15 to about 40% by weight for low density fibers and up to

about 55 to 75% of the total weight of the cured laminate for glass mat or glass fabric laminates. The fiber content of filament wound constructions such as pipe, when made from impregnated glass roving, is generally about 70 to 80% of the total weight of the cured product.

Reinforced laminates of fibrous materials such as glass cloth, glass mats, synthetic fiber, cloth mats, paper and the like can be copper-clad to produce copper-clad laminates with excellent electrical properties to be used in preparing printed circuits and the like. Copper-clad laminates can be prepared by coating copper foil with a composition of this invention containing a polyphenylene ether polymer and then baking the coated copper foil at 160°C for about 15 minutes. The baked resin coated foil is then placed on resin impregnated fibrous materials such as glass cloth which has been impregnated with the novel resin compositions of this invention, unsaturated polyester resins, diallylic phthalate prepolymers and the like, and then the "laminate" is pressed at 50 to 2,000 psi at 100° to 170°C for at least 5 minutes to convert the resinous materials to the thermoset state. If desired, the coated copper foil can be laminated to almost any previously cured base. As indicated above, dicumyl peroxide is the preferred catalyst for producing the copper-clad laminates of this invention. The resulting copper-clad laminate has excellent adhesion of the copper to the base material and has excellent electrical properties. Quite surprisingly, when tested according to NEMA Standards Publication LJ-1-1966, but at up to 200°C rather than 25°C standard, these copper-clad laminates retained essentially all of their electrical properties as measured at room temperature. Reinforced laminates made from those compositions of this invention which contain polyphenylene ether polymers adhere very well to the type of copper foil used in making printed circuit boards without precoating the foil. The compositions containing a polychlorinated aromatic hydrocarbon apparently "wet" the metal surface better than the similar compositions without the polychlorinated aromatic hydrocarbon disclosed in our copending application Ser. No. 682,326, filed Nov. 13, 1967.

Those compositions of this invention which contain polyphenylene ether resins as a portion of the system are useful as thermosetting coatings. They adhere strongly to a wide variety of substrates involving metals, ceramic bases and other resinous substances while providing excellent electrical insulating characteristics. They may be used as conformal coatings for electronic devices or as primer coats to bond additional electrical insulation to the devices. Many molding compounds used in the encapsulation of electronic components do not form good bonds to the devices and to the wire leads, thereby permitting access of moisture. When such devices or leads are first coated with the compositions of this invention, and then subjected to mold encapsulation, hermetically sealed systems are obtained which resist moisture even on prolonged exposure to boiling in water or high pressure steam.

The coatings may be formulated with all polymer components in solution. Useful solvents comprise chlorinated hydrocarbons and aromatic hydrocarbons. Alternatively, the coatings may be formulated as finely divided dispersions of the polymer system in liquid organic media. Useful dispersion media include organic

ketones and esters. Dispersed coatings are preferred for most applications, since they may be formulated at high polymer solids levels yet are capable of application by brushing, spraying or roller coating. Coatings containing up to 80% solids can be applied by knife coating techniques, yet they flow out to smooth adherent insulating varnishes when subjected to oven baking at temperatures of 120°C or higher.

The test methods appearing in the following list were followed in testing the molded and laminated test specimens made from the various compositions disclosed in the examples.

15	A. Flexural Strength*	ASTM	D-790
	B. Modulus of Elasticity	"	D-790
	C. Tensile Strength	"	D-638
	D. Izod Impact	"	D-256
	E. Compressive Strength	"	D-695
	F. Deflection Temperature	"	D-648
	G. Water Absorption	"	D-570 (a)
20	H. Specific Gravity	"	D-792
	I. Dielectric Constant	"	D-150
	J. Dissipation Factor**	"	D-150
	K. Volume and Surface Resistivity	"	D-257
	L. Flame Resistance (Burning and Ignition Tests Method 1 and 2)	"	D-229
25	M. Hardness	"	D-785
	N. Hardness (Rockwell)	"	D-785
	O. Shear Strength (Modified for Flat Beams)	"	D-2344

\*The flexural strengths at 150°C for the unfilled resin systems were obtained after 4 hour conditioning at 150°C. Each individual specimen was held to 4 hour conditioning to avoid any ambiguity that may be caused by post-curing in the test oven. Only maximum flexural yield was measured.

\*\*The wet test is conducted on samples which were conditioned by immersing the samples for 24 hours at 23°C in distilled water, removing the samples, blotting them dry and then testing the samples as soon as practical according to the test method.

The following examples, illustrating the novel products disclosed herein, are given without any intention that the invention be limited thereto. All parts and percentages are by weight unless otherwise noted.

#### EXAMPLE I

A series of samples were prepared which were simple mixtures of chlorinated terphenyl containing 60% chlorine or a chlorinated biphenyl containing 68% chlorine with different monomers and 2% by weight of dicumyl peroxide based on the total weight of the chlorinated aromatic compound and monomer. The samples were placed in a paper cup and a thermocouple connected to a recording thermocouple bridge was inserted in each of the samples. The samples were placed in an oven maintained at a temperature of 160°C. Various concentrations of the chlorinated aromatics with various monomer concentrations were prepared. Some of the samples tested were subjected to a 16 hour acetone Soxhlet extraction at the reflux temperature of acetone. The composition details, the reaction initiating temperature, the reaction peak exotherm temperature, the reaction time to peak exotherm, and the results of the reaction are contained in Table I.

Soxhlet extraction studies were made on some of the compositions. Mixtures of the unreacted chlorinated aromatics and the monomers yield clear solutions in chloroform and hot acetone. The chlorinated aromatics can only be partially extracted from the reaction product with monomers, and that fraction which is extracted is different from the original crystalline chlorinated aromatic in that it tends to be a resinous gum.



TABLE I

	BATCH REACTION AT 160°C. MONOMER STUDY											
	1	2	3	4	5	6	7	8	9	10	11	12
Chlorinated terphenyl, 60% chlorine (Aroclor 3460)	50	45	40	60	65	60	—	—	40	74	87	78
Chlorinated biphenyl, 68% chlorine (Aroclor 1268)	—	—	—	—	—	—	42	62	—	—	—	—
Diallyl phthalate	—	—	—	—	—	40	—	38	—	—	—	—
Diallyl isophthalate	50	55	60	40	35	—	38	—	—	—	—	—
Diallyl chloroendate	—	—	—	—	—	—	—	—	60	—	—	—
Triallyl cyanurate	—	—	—	—	—	—	—	—	—	26	—	—
Triallyl phosphate	—	—	—	—	—	—	—	—	—	—	13	—
Diallyl maleate	—	—	—	—	—	—	—	—	—	—	—	22
Divinyl benzene (55%)	—	—	—	—	—	—	—	—	—	—	—	—
Ethylene Glycol Dimethacrylate	—	—	—	—	—	—	—	—	—	—	—	—
Dicumyl peroxide	2	2	2	2	2.5	2	2	2	2	2	2	2
Initiation Temp., °C.	130	130	130	135	155	125	145	140	135	125	135	122
Peak Exotherm Temp., °C.	223	211	227	218	273	222	225	216	205	245	248	230
Time to Exotherm, min.	3	5	7	4	5	3	3	3	3	4	4	3
16 hr. Acetone Soxhlet Extraction												
Nature of Solution												
Clear												
Cloudy				1			1	1				
Dried Extract												
Powder												
Gum				1			1	X				

	Comparison					
	13	14	C-1	C-2	C-3	C-4
Chlorinated terphenyl, 60% chlorine (Aroclor 3460)	70	70	100	—	—	50
Chlorinated biphenyl, 68% chlorine (Aroclor 1268)	—	—	—	—	100	—
Diallyl phthalate	—	—	—	80	—	50
Diallyl isophthalate	—	—	—	—	—	—
Diallyl chloroendate	—	—	—	—	—	—
Triallyl cyanurate	—	—	—	—	—	—
Triallyl phosphate	—	—	—	—	—	—
Diallyl maleate	—	—	—	—	—	—
Divinyl benzene (55%)	30	—	—	—	—	—
Ethylene glycol dimethacrylate	—	30	—	—	—	—
Dicumyl peroxide	2	2	2	2	2	—
Initiation temp., °C.	125	115	none	110	none	none
Peak exotherm temp., °C.	235	200	none	250	none	none
Time to exotherm, min.	3	4	—	5	—	—
16 hr. Acetone Soxhlet Extraction						
Nature of Solution						
Clear			1	1		
Cloudy					1	
Dried Extract						
Powder			1		1	
Gum				1		

## EXAMPLE 2

Diallyl phthalate prepolymer, prereacted poly-chlorinated aromatic compound with monomer, molding compounds were prepared. The prereacted poly-chlorinated aromatic hydrocarbon and monomer reaction products were prepared in 3½ lb. batches. Chlorinated biphenyls, containing 68% combined chlorine (Aroclor-1268 from Monsanto Chemical Company), and a diallylic phthalate monomer were heated together in a ratio of 62 parts of chlorinated biphenyls to 38 parts of diallylic phthalate monomer with 2 parts by weight of dicumyl peroxide per 100 parts of Aroclor plus monomer. The reaction products were made in a 1-gallon sigma blade mixer equipped with a steam jacket. The steam to the jacket was at 160°C and the internal temperature of the blade and side walls was about 120°C. Although accurate reaction time measurements could not be made, reasonably short reaction times of about 10 minutes were observed in preparing these reaction products. The reactants were

highly fluid up to the point of gelation, from which point the reaction proceeded rapidly to a brittle stage, breaking up thereafter into small lumps. The prereacted materials were ball-milled for a 2-hour period in a 1½ gallon jar mill to produce 1,000 g of ground product. Reaction products were prepared using diallyl phthalate and diallyl isophthalate monomer and are designated Reaction Product No. 1 and Reaction Product No. 2 respectively in Table II. The reaction products were mixed with diallyl phthalate prepolymer and diallyl isophthalate prepolymer along with some catalyst, monomer and fillers to make molding compounds. The composition details and physical properties of samples molded from the molding compounds are found in Table II. For comparison, the table includes qualifying specifications for diallyl phthalate molding compounds for military use under MIL M-14F and MIL-19833. (The largest market for the diallyl phthalate molding compounds in the United States is for military applications.)

TABLE II

MOLDING COMPOUNDS			MIL M-14F Specification Requirements for SDGF Molding Compounds
Formulation			
Diallyl phthalate prepolymer (Dapon 35, FMC Corp.)	74		
Diallyl isophthalate prepolymer (Dapon M, FMC Corp.)		70	
Diallyl phthalate monomer	3		
Diallyl isophthalate monomer		3	
Reaction Product No. 1 <sup>100</sup>	26		
Reaction Product No. 2 <sup>100</sup>		30	
tert Butyl perbenzoate	3	3	
Calcium Stearate	2	2	
Antimony oxide	15	18	
Wollastonite	30	20	
Glass (K <sup>100</sup> strands)	90	100	
Properties			
Ignition time (sec.)	93	98	90 (min.)
Burning time (sec.)	78	76	90 (max.)
Heat Deflection Temp. °C.	175	270	160 (min.)
Load (ft. lbs.) impact	0.66	0.60	0.30 (min.)
Compressive (psi) Strength	24,620	26,500	16,000 (min.)
Flexural (psi) Strength	11,462	11,200	9,000 (min.)
Modulus (psi) of E			
Elasticity	1.70 x 10 <sup>6</sup>	1.72 x 10 <sup>6</sup>	—
Water absorption (%)	0.12	0.11	0.40 (max.)
Specific Gravity	1.902	1.897	—
D.C. 10 <sup>3</sup> /10 <sup>6</sup> cm	4.50/4.39	4.36/4.24	4.6/4.4 (max.)
D.C. 10 <sup>3</sup> /10 <sup>6</sup> (wet)	4.52/4.38	4.41/4.29	4.7/4.5 (max.)
D.F. 10 <sup>3</sup> /10 <sup>6</sup> cm	.005/.005	.005/.005	.009/.015 (max.)
D.F. 10 <sup>3</sup> /10 <sup>6</sup> (wet)	.006/.005	.006/.005	.013/.017 (max.)
Volume Resist. (ohm)	1.0 x 10 <sup>10</sup>	0.8 x 10 <sup>10</sup>	5 x 10 <sup>9</sup> (min.)
720 hr. at 70°C.			
100% R.H.			

<sup>100</sup> 62/38 reaction product of chlorinated biphenyl with diallyl phthalate monomer.  
<sup>100</sup> 62/38 reaction product of chlorinated biphenyl with diallyl isophthalate monomer.  
<sup>100</sup> Dielectric constant.  
<sup>100</sup> Dissipation Factor.

## EXAMPLE 3

A portion of the diallyl phthalate monomer-chlorinated biphenyl Reaction Product, Reaction Product No. 1 of Example 2, was incorporated into a diallyl phthalate polyester molding compound. Test specimens were molded from the compound for 3 minutes at 150°C at 2,000 psi (pounds per square inch). The composition of the molding compound, the physical and electrical properties of the molded test specimens and the MIL-M-14F specification requirements for MAI 30 diallyl phthalate polyester molding compounds are shown in Table III.

TABLE III

MAI-30 (DAP-POLYESTER) COMPOUNDS			MIL M-14 F Specification Requirements for MAI-30 (DAP-Polyester) Molding Compounds
Formulation			
Diallyl phthalate monomer	35		
Unsat isophthalate polyester resin, reactive type.	35 <sup>100</sup>		
Reaction product No. 1	30 <sup>100</sup>		
Glass (K <sup>100</sup> strands)	57		
Hydrated alumina	48		
Wollastonite	48		
Antimony oxide	18		
Calcium stearate	2		
tert Butyl perbenzoate	2		
Physical Properties			
Compressive	21,200	20,000 (min.)	
D.C. 10 <sup>3</sup> /10 <sup>6</sup>	4.93/4.54	6.3/6.2 (max.)	
D.C. 10 <sup>3</sup> /10 <sup>6</sup> (wet)	5.15/4.63	6.4/6.4 (max.)	

TABLE III-continued

MAI-30 (DAP-POLYESTER) COMPOUNDS			MIL M-14 F Specification Requirements for MAI-30 (DAP-Polyester) Molding Compounds
Formulation			
D.F. 10 <sup>3</sup> /10 <sup>6</sup>	.010/.009	.015/.012 (max.)	
D.F. 10 <sup>3</sup> /10 <sup>6</sup> (wet)	.012/.011	.017/.015 (max.)	
Flexural (psi)	16,700	14,000 (min.)	
Flexural Modulus (psi)	2.0 x 10 <sup>6</sup>	—	
Heat Deflection Temp. °C	272	200 (min.)	
Impact (ft. lbs.)	4.37	3.0 (min.)	
Water Absorption (%)	0.17	0.5 (max.)	
Ignition time (sec.)	105	100 (min.)	
Burning time (sec.)	17	75 (max.)	
Rockwell Hardness (M)	108	—	
Specific Gravity	1.93	—	
Vol. Resistivity (ohm.cm.)	1.2 x 10 <sup>10</sup>	—	
Surface Resistivity (ohm.)	2.8 x 10 <sup>10</sup>	—	

<sup>100</sup> Equimolar amounts of isobutyl anhydride and isophthalic were heated together with a 10% excess of diethylene glycol. Water was removed azeotropically and the diethylene glycol was removed by heating at about 200°C. was continued until the acid number of the polyester under vacuum and the polyester resin recovered.

<sup>100</sup> 62/38 Reaction Product of chlorinated biphenyl with diallyl phthalate monomer from Example 2.

## EXAMPLE 4

Example 3 was repeated except that a styrene polyester was used rather than a DAP polyester with a portion of the diallyl phthalate-chlorinated biphenyl gel of Example 2. The molding compositions were molded for 3 minutes at 150°C at 2,000 psi. The composition of the molding compound, physical and electrical properties of the molded samples, and, for comparison, the MIL

M-14 F specification requirements for MAI 60 flame-resistant styrene polyesters are shown in Table IV.

TABLE IV

STYRENE-POLYESTER MOLDING COMPOUNDS MIL M-14 F Specification Requirements for MAI 60 (Styrene- Polyester) Molding Compounds		
Formulation		
Monomer, styrene	24.3	
Polyester Resin <sup>1</sup>	43.3	
Reaction Product No. 1 <sup>2</sup>	30	
Glass (1/4" strands)	67	
Hydrated alumina (Hydral 710)	48	
Wollastonite	48	
Antimony oxide	18	
Calcium stearate	2	
tert Butyl perbenzoate	2	
Properties		
Compressive (psi)	23,150	18,000 (min.)
D.C. 10 <sup>2</sup> /10 <sup>4</sup>	4.77/4.53	6.0/5.7 (max.)
D.C. 10 <sup>2</sup> /10 <sup>4</sup> (wet)	3.04/4.31	7.0/6.0 (max.)
D.F. 10 <sup>2</sup> /10 <sup>4</sup>	.012/.008	.03/.03 (max.)
D.F. 10 <sup>2</sup> /10 <sup>4</sup> (wet)	.016/.013	.08/.05 (max.)
Flexural (psi)	17,700	12,000 (min.)
Flexural modulus (psi)	2.0 x 10 <sup>6</sup>	—
Heat Deflection Temp. °C	272	200 (min.)
Impact (ft. lbs.)	7.45	6.0 (min.)
Water Absorption (%)	0.17	1.5 (max.)
Ignition time (sec.)	95	90 (min.)
Burning time (sec.)	58	90 (max.)
Rockwell Hardness (M)	107	—
Specific Gravity	1.898	—

<sup>1</sup> Reactive polyester resin from Example 1 made by reacting equimolar portions of maleic and isophthalic acids with a 10% excess of diethylene glycol.

<sup>2</sup> 42/38 Reaction Product No. 1 of chlorinated biphenyl with diallyl phthalate monomer from Example 2

## EXAMPLE 5

High pressure laminates containing diallylic phthalate polychlorinated aromatic reaction products were prepared using polychlorinated aromatic diallylic phthalate reaction products made according to Example 2. Sixty parts of polychlorinated aromatic compound (Aroclor 5460), a chlorinated terphenyl containing 60% by weight of chlorine, was reacted with 40 parts of diallylic phthalate monomer and 2 parts of dicumyl peroxide as described in Example 2, and is labeled Reaction Product No. 3 in Table V. A similarly prepared reaction product, using 40 parts of diallyl isophthalate monomer in place of diallyl phthalate monomer, is labeled Reaction Product No. 4 in Table V. A glass cloth laminate was prepared as follows: A "resin blend" of the polychlorinated aromatic diallylic phthalate reaction product with the corresponding diallylic phthalate prepolymer and a small amount of diallylic phthalate monomer and 2 parts of dicumyl peroxide catalyst per 100 parts of reactants as shown in Table V was dispersed, in the amount of 100 parts by weight of the resin blend, in 100 parts by weight of acetone. Woven glass cloth was impregnated with this dispersion and allowed to dry at least 48 hours in air at room temperature. The impregnated glass cloth, often termed prepreg, was cut into 12 by 12 inch squares and stacked 18 plies deep with the warp yards parallel. The pre-impregnated lay-up was laminated in a flat bed press for 30 minutes at 80°C at contact pressure, 30 minutes at 120°C at 300 psi and 1 hour at 160°C at 300 psi. Two samples were prepared, one using diallyl phthalate monomer and another sample using diallyl isophthalate prepolymer and diallyl isophthalate monomer. The composition details of the resin mixture used

to impregnate the glass fabric, the resin content of the prepreps prepared from the resin systems and the physical properties of the cured laminates are found in Table V.

TABLE V

HIGH PRESSURE DIALLYL PHTHALATE CHLORINATED TERPHENYL GFL LAMINATES		
Formulation		
Reaction Product No. 3	50	
Reaction Product No. 4		50
Diallyl phthalate prepolymer (Dapon 35)	45	
Diallyl isophthalate prepolymer (Dapon M)		45
Diallyl phthalate monomer	5	
Diallyl isophthalate monomer		5
Dicumyl peroxide	2	2
Physical Properties		
Burning <sup>1</sup> time (sec.)	12.7	32
Compressive (psi)	49,500	44,900
Rockwell Hardness (M)	118	122
Flexural (psi) R.T.	67,800	76,200
150°C	13,700	—
Modulus (psi) R.T.	2.43 x 10 <sup>6</sup>	2.92 x 10 <sup>6</sup>
150°C	0.97 x 10 <sup>6</sup>	—
Tensile (psi)	49,100	50,400
Specific Gravity	1.822	1.858
Shear (psi)	4,010	3,400
Flexural (psi) 2 hr. boil	67,900	72,700
Modulus (psi) 2 hr. boil	2.48 x 10 <sup>6</sup>	2.89 x 10 <sup>6</sup>
Flexural (psi) after aging:		
1 day	68,200	72,800
4 days	70,900	75,300
10 days	65,600	77,800
D.C. 10 <sup>2</sup> /10 <sup>4</sup>	4.46/4.35	4.26/4.22
D.C. 10 <sup>2</sup> /10 <sup>4</sup> (wet)	4.52/4.38	4.27/4.18
D.F. 10 <sup>2</sup> /10 <sup>4</sup>	.0033/.0054	.0039/.0042
D.F. 10 <sup>2</sup> /10 <sup>4</sup> (wet)	.0063/.0061	.0042/.0048
Vol. Resist. (ohm. cm.)	5.2 x 10 <sup>10</sup>	12.9 x 10 <sup>10</sup>
Surface Resist. (ohm.)	1.3 x 10 <sup>10</sup>	1.19 x 10 <sup>10</sup>
Insulation Resist. (ohm.)		
10 day cycle		
72/70/100	1.25 x 10 <sup>10</sup>	6.3 x 10 <sup>10</sup>
72/25/100	5.60 x 10 <sup>11</sup>	3.6 x 10 <sup>10</sup>
100/70/100	0.3 x 10 <sup>10</sup>	1.9 x 10 <sup>10</sup>

<sup>1</sup>ASTM D229, Part 1

## EXAMPLE 6

A molding compound was prepared directly from a mixture of a chlorinated terphenyl (Aroclor 5460 containing 60% combined chlorine by weight) and diallyl phthalate prepolymer (Dapon 35) and diallyl phthalate monomer. The following composition was compounded as an "SDG-F Type" flame-resistant molding compound designed to meet Military Specification MIL M-14 F.

	Parts by weight
Diallyl phthalate prepolymer (Dapon 35)	73
Diallyl phthalate monomer	9
Chlorinated terphenyl (60% combined chlorine)	18
Chopped glass (1/4" strands)	90
Clay, hydrated	30
Antimony oxide	15
Calcium stearate	1
Tertiary butyl perbenzoate	3
Acetone	160

All the ingredients in the above were mixed together in a heavy-duty mixer until the glass and the fillers were thoroughly wetted. The composition was spread on trays and the acetone was allowed to evaporate at room temperature. The composition was then compounded on a two-roll rubber mill at a temperature of 80°-90°C. The mill sheet was cooled and ground into a molding powder. Test pieces molded at 150°C at 2,000 psi for a

minimum of 1.5 minutes showed full cure as measured by development of hot hardness and resistance to boiling chloroform. The test data in the following table show the properties of the above molded compositions compared to the MIL M-14 F specification requirements for diallyl phthalate molding compounds.

TABLE VI

CHLORINATED TERPHENYL AROCLOR/MONOMER/DAPON SDG-F COMPOUND		
	Example 6	Comparison ***
Ignition time (sec.)	97	90 (min.)
Burning time (sec.)	70	90 (max.)
Heat Deflection Temp. °C	165	160 (max.)
Rockwell Hardness (M)	115	—
Impact (ft. lb.)	0.58	0.30 (min.)
Flexural strength, psi	12,120	9000 (min.)

the laminates were placed in a vacuum bag and evacuated to remove air from the polymerizable liquid resin mixture. The lay-up was laminated while maintaining the vacuum at pressures at 15 psi or less, as in the very early stages of cure it was very difficult to maintain pressure on the polymerizable liquid. Full cure was obtained after about 15 minutes at 150°C as measured by hot hardness, hot strength, and solvent resistance in boiling acetone. This laminate construction was also cured in a vacuum bag by exposure to a 3 MEV (million electron volts) electron beam in multiple passes of 5 megarads each until a room temperature Barcol hardness of 60 was developed. The polymerizable liquid details and the physical properties of the cured laminates are set forth in Table VII.

TABLE VII

VACUUM BAG LAMINATES		
Formulation		
Diallyl phthalate prepolymer (Dapon 35)	50	
Diallyl isophthalate prepolymer (Dapon M)		50
Diallyl phthalate monomer	20	
Diallyl isophthalate monomer		20
Chlorinated terphenyl (Aroclor 5460)	30	30
Dicobalt peroxide	2	2
Burning time (ASTM D229, Pt. I)	16 sec.	27 sec.
Specific Gravity	2.104	2.040
Percent Resins	28.0	32.7
Rockwell Hardness (M)	117	121
Flex. strength, psi, 25°C	94,000	101,700
Flex. strength, psi, 150°C	25,410	(260°C) 6,050 ***
Modulus, psi, 25°C	$4.08 \times 10^6$	$3.86 \times 10^6$
Modulus, psi, 150°C	$2.49 \times 10^6$	(260°C) $1.47 \times 10^6$ ***
Compressive Str., psi	49,110	48,740
Tensile Str., psi	63,460	62,110
Shear, psi	3710	2040
Flex., 2 hr. H <sub>2</sub> O/100°C	93,390	88,810
Modulus, 2 hr. H <sub>2</sub> O/100°C	$4.18 \times 10^6$	$3.26 \times 10^6$
Flexural (psi) after aging:		
1 day	101,900	95,400
4 days	97,660	98,870
10 days	93,900	88,580
D.C. $10^6/10^4$	4.94/4.85	4.72/4.65
D.C. $10^6/10^4$ (wet)	5.00/4.84	4.81/4.65
D.F. $10^6/10^4$	.0034/.0049	.0068/.0040
D.F. $10^6/10^4$ (wet)	.0088/.0057	.0091/.0048
Vol. Resistivity, (ohm cm.)	$4.17 \times 10^{14}$	$3.56 \times 10^{14}$
Surf. Resistivity, (ohm)	$5.08 \times 10^{14}$	$2.36 \times 10^{14}$

\*\*\* Test run at 260°C

Modulus, psi	$1.72 \times 10^6$	—
Compressive strength, psi	23,060	16,000 (min.)
Water absorption (%)	0.08	0.30
Specific gravity	1.880	—
D.C. $10^6/10^4$	4.44/4.35	4.6/4.4 (max.)
D.C. $10^6/10^4$ (wet)	4.47/4.37	4.7/4.5 (max.)
D.F. $10^6/10^4$	.0044/.0043	.009/.015 (max.)
D.F. $10^6/10^4$ (wet)	.0054/.0047	.013/.017 (max.)
Vol. Resist., (ohm cm.)	$6.76 \times 10^{14}$	—
Surf. Resist., (ohm)	$7.95 \times 10^{14}$	—
Vol. Resist. (ohm) (720 hrs. 70°C at 100% relative humidity)	$1.97 \times 10^{14}$	$5 \times 10^9$

\*\*\* MIL M-14F Specification requirement properties for glass filled flame-retardant diallyl phthalate molding compound.

## EXAMPLE 7

Wet lay-up or vacuum bag laminates were prepared using a diallylic phthalate prepolymer, a diallylic phthalate monomer and chlorinated terphenyl (Aroclor 5460). The wet lay-up was prepared by making a liquid blend of the monomer, chlorinated terphenyl and diallylic phthalate resin and catalyst. The liquid polymerizable resin composition was poured into several layers of woven glass fabric. After impregnation,

## EXAMPLE 8

A premixed molding compound containing a portion of the polyester resin described in Example 3 and a polychlorinated aromatic compound was prepared. The formulations listed in Table VIII were mixed in a heavy duty mixer until the glass and fillers were thoroughly wetted with the polyester resin-monomer-chlorinated terphenyl mixture. The compositions of this invention were compared with similarly compounded conventional diallyl phthalate polyesters. The composition details and physical properties are set forth in the following Table. The test specimens were molded at 1000 psi at 150°C for 3 minutes.

TABLE VIII

PREMIX MOLDING COMPOUNDS		
Formulation		
Polyester resin, Reactive isophthalate type ***	50	41
Diallyl phthalate monomer	30	41
Chlorinated terphenyl (60% chlorine)	0	18
4" Chopped glass fibers	57	57

TABLE VIII-continued

PREMIUM MOLDING COMPOUNDS		
Formulation		
Wollastonite	55	55
Hydrated alumina (Hydral 710)	55	55
tert Butyl perbenzoate	2	2
Zinc stearate	1	1
Antimony oxide	18	18
Physical properties		
Ignition time, sec.	103	102
Burning time, sec.	244	31
Deflect. temp. °C	283	270
Rockwell Hardness (M)	102	103
Load Impact (ft.lbs.)	4.90	6.90
Specific Gravity	1.935	1.953
Water absorption, (%)	0.37	0.25
Compressive str., psi	20,360	21,450
Flex. strength, psi	15,860	18,450
Modulus, psi	$2.0 \times 10^6$	$2.01 \times 10^6$
D.C. $10^3/10^4$	5.56/4.85	5.11/4.66
D.C. $10^3/10^4$ (wet)	6.25/5.08	5.46/4.79
D.F. $10^3/10^4$	.023/.016	.014/.010
D.F. $10^3/10^4$ (wet)	.041/.025	.018/.016
Vol. Resistivity, ohm-cm.	$6.82 \times 10^{14}$	$8.74 \times 10^{14}$
Surf. Resistivity ohms	$7.43 \times 10^{14}$	$5.13 \times 10^{14}$

<sup>100</sup> Reactive polyester resin from Example 3 made by reacting equimolar portions of maleic and isophthalic acids with a 10% excess of diethylene glycol.

## EXAMPLE 9

A low-pressure laminate containing diallyl isophthalate monomer and prepolymer, a chlorinated terphenyl and polyphenylene ether polymer was prepared in which the following formulation was used to saturate glass cloth from which the laminate was prepared.

	Parts by Weight
Polyphenylene ether resin (General Electric Grade 691-111)	25
Chlorinated terphenyl containing 60% combined chlorine (Aroclor 5460)	15
Diallyl isophthalate prepolymer (Dapon M)	30
Diallyl isophthalate monomer	30
Dicumyl peroxide (recrystallized)	3
Acetone	130

The diallyl isophthalate prepolymer and polyphenylene ether polymer were ball-milled together until 70% of the product was -200 mesh (U.S. Standard Sieve Series 1940). The chlorinated terphenyl-diallyl isophthalate monomer and dicumyl peroxide were dissolved in the acetone solvent and the ball-milled powder was added to this solution to make up a saturating mixture. Woven glass cloth, type 181, was impregnated with this mixture and allowed to dry at least 48 hours at room temperature. The dry impregnated glass cloth was cut into 12 by 12 inch squares, stacked 18 plies deep with the warp yarns parallel. The pre-impregnated lay-up was made in a flat bed press for 30 minutes at 80°C at contact pressure, 30 minutes at 120°C at 300 psi and 1 hour at 160°C at 300 psi. The laminated sample had a resin content of 44% and was found to be non-burning in ASTM Flammability Test D-229, Part 1. The following physical properties were obtained on the laminated sample:

Laminate Physical and Electrical Properties

Resin content	44%
Specific Gravity	1.752
Rockwell Hardness (M)	120
Compressive strength, psi	50,400
Tensile strength, psi	43,700
Flexural strength, psi at 23°C	67,300

-continued

Laminate Physical and Electrical Properties

Flexural strength, psi at 200°C	13,700
Flexural modulus at 23°C, psi	$3.0 \times 10^6$
Flexural modulus at 200°C, psi	$1.3 \times 10^6$
Tensile shear strength, psi	3,530

## EXAMPLE 10

A vacuum bag laminate was prepared from a composition containing no polyunsaturated polymer. Thirty parts of polyphenylene ether polymer was ball-milled 1 hour together with 10 parts of trihydrate of aluminum oxide (Hydral 710 from Alcoa). The ball-milled materials were mixed with a solution containing 90 parts of diallyl isophthalate monomer, 17.5 parts of chlorinated terphenyl containing 60% by weight combined chlorine (Aroclor 5460) and 4.5 parts of dicumyl peroxide catalyst (DiCup R) with stirring at 60° to 80°C. This mixture was used to saturate 12 plies of 181 glass cloth 12 inches square. The impregnated cloth was stacked 12 plies deep with the warp yarns parallel. The impregnated lay-up was placed in a vacuum bag which was then evacuated until essentially all of the air was removed. The lay-up was then laminated in a flat bed press, maintaining the vacuum, throughout the lamination, for 1 hour at 93°C, 1 hour at 121°C and 1 hour at 160°C. The cured laminate had good "hot" hardness when removed from the vacuum bag at about 150°C and when cooled was a tough flexible laminate.

## EXAMPLE 11

A copper-clad laminate was prepared by roller coating onto 1 oz. copper foil (1 oz. per sq. ft.), a standard copper used in the preparation of printed circuit boards, the following coating composition:

Ingredients	Parts by weight
Polyphenylene ether polymer (General Electric Company Grade 631-101)	60
Diallyl isophthalate prepolymer (FMC Corp., Dapon M)	10
Diallyl isophthalate monomer	10
Chlorinated terphenyl, 60% chlorine (Aroclor 5460), Monsanto Chemical Corp.	20
Dicumyl peroxide	5
Chloroform	150
Trichloroethylene	130

After solvent removal and baking for 5 minutes at 165°C, the average film thickness of the coating on the copper was 1.5 mil. An 18 ply, low pressure, glass cloth laminate was prepared for laminating as described in Example 9. The treated copper foil was placed on one side of the laminate, with the treated side of the foil facing the laminate, and the laminate was then cured as described in Example 9.

The bond and peel strength of the copper foil to the laminate base after laminating exceeded 10 lbs. per inch. This bond strength was essentially unchanged after immersion in a solder bath at 260°C for 2 minutes.

## EXAMPLE 12

A copper-clad glass cloth laminate was prepared using the following coating composition to saturate glass cloth as described in Example 9:

Three hundred twenty parts of polyphenylene ether polymer (General Electric Grade 691-111), 80 parts of

diallyl isophthalate prepolymer (Dapon M) and 50 parts of hydrated alumina (Hydral 710) were ground together in a ceramic ball mill until 70% of the mixture passed through a 200 mesh sieve (U.S. Standard Sieve Series, 1940). Forty-five parts of this powdered mixture was added with agitation to a solution of 30 parts of diallyl phthalate monomer, 30 parts of chlorinated terphenyl (Aroclor 5460), 10 parts of diallyl isophthalate prepolymer (Dapon M), and 5 parts of recrystallized dicumyl peroxide (DiCup R — Hercules Powder Company) in 100 parts of methyl ethyl ketone.

The saturated glass cloth was cut into 12 inch squares prepared for laminating as described in Example 9. A 12 inch square of 1 oz. copper foil was placed on one side of the saturated glass cloth lay-up which was then laminated (cured) as described in Example 9 to produce a copper-clad laminate. When laminated, the copper foil had a bond strength to the glass cloth base in excess of 8 lbs. per inch peel strength at 90°. This bond strength was maintained, and the laminate maintained its integrity, when immersed for 2 minutes in solder at 260°C.

#### EXAMPLE 13

A silicon diode was coated by dipping the diode into the polymerizable coating composition of Example 11. Only about one-eighth inch of the metal leads (Covar) to the diode, nearest to the body of the diode, was coated, thereby leaving part of the leads uncoated to facilitate soldering. The coated diode was baked in an air circulating oven at 170°C for 2 minutes to remove solvent and polymerize the coating. The resulting coating was between 1 and 2 millimeters thick.

The coated diode was then mold encapsulated using the following encapsulating composition:

	Parts by weight
Diallyl phthalate prepolymer (Dapon D - FMC Corp.)	45
Glass, 1/4" chopped fibers	25
Clay, calcined	30
Calcium stearate	0.5
Tertiary butyl perbenzoate	1.5

The encapsulation was done by transfer molding at a ram pressure of 1,000 psi at 160°C for a cure time of 3 minutes. The encapsulated silicon diode showed no loss of electrical performance after 100 hours' immersion in boiling water.

A second silicon diode was coated as described above. This second coated diode was mold encapsulated as described above using a bisphenol A diglycidol ether type epoxy polymer encapsulating composition (Polyset 300 available from Morton Chemical Company). The wet electrical performance of the coated-encapsulated diode was improved as compared to a similar diode that was not coated prior to encapsulation with the epoxy encapsulating composition.

#### EXAMPLE 14

Vacuum bag laminates were prepared using diallyl isophthalate prepolymer and monomer, brominated biphenyl and in one example a polyphenylene ether polymer. Woven glass cloth, type 181, was impregnated with the mixtures set forth below and allowed to dry for 48 hours at 25°C. The dry impregnated glass cloth was cut into 12 inch squares, stacked 18 plies

deep with the warp yarns parallel. The impregnated lay-up was molded by the vacuum-bag technique. Full cure was obtained after 15 minutes at 150°C.

TABLE IX

Formulation		
Diallyl isophthalate monomer	250	350
Diallyl isophthalate prepolymer	650	310
Hexabromobiphenyl	200	150
Dicumyl peroxide	30	30
Gamma methacryloxypropyl-trimethylsilane	5	5
Hydroquinone	0.3	0.3
Aluminum trihydrate	70	40
Methyl ethyl ketone	400	400
Polyphenylene ether blend ***	—	285
Physical Properties		
Resin content, weight %	29.8	35.1
Rochwell hardness (M)	120	122
Compressive strength, psi	26,400	44,500
Flexural strength, psi at 25°C	63,800	83,900
psi at 205°C	31,100	22,000
after 2 hours in boiling water	47,800	76,900
Flexural modulus, psi at 25°C	2.7 x 10 <sup>6</sup>	2.8 x 10 <sup>6</sup>
psi at 205°C	2.6 x 10 <sup>6</sup>	1.8 x 10 <sup>6</sup>
after 2 hours in boiling water	2.6 x 10 <sup>6</sup>	2.9 x 10 <sup>6</sup>
Tensile strength, psi	52,600	55,300
Shear strength, psi at 25°C	3,010	5,400
psi at 205°C	1,740	1,470
Moisture absorption, 48 hours under water at 50°C, weight %	.44 <sup>aa</sup>	.08
Specific gravity	1.974	1.911
Dielectric constant 10 <sup>3</sup> /HZ dry	4.53	4.30
10 <sup>6</sup> /HZ dry	4.46	4.33
Burning test (ASTM D-433)	non-burning	non-burning

<sup>aa</sup> 200 parts of polyphenylene ether polymer, 57 parts of diallyl isophthalate prepolymer, 28 parts of aluminum trihydrate, ball-milled together for 12 hours.

<sup>ab</sup> Sample contained some small voids.

Obviously, these examples can be multiplied indefinitely in view of the possible permutations and combinations of resins, polychlorinated aromatic hydrocarbons, fillers and additives known in the art to be useful in preparing molding compounds and laminates. The principle of this invention has been explained and exemplified so that it can readily be practiced by those skilled in the art. The best mode contemplated by the inventors has been set forth. Clearly, within the scope of the appended claims, the invention may be practiced by those skilled in the art having benefit of this disclosure, otherwise than as specifically described and exemplified herein.

1. A thermosetting composition comprising:

1. 10 to 50 parts by weight of a polyunsaturated monomer;

2. 5 to 30 parts by weight of a polychlorinated or polybrominated aromatic hydrocarbon containing at least 50% by weight chlorine or bromine and having a molecular weight of at least 200;

3. 20 to 60 parts by weight of a polyphenylene ether polymer;

4. 0-30 parts by weight of a polyunsaturated polymer having carbon to carbon unsaturation and

5. 2-10 parts by weight per 100 parts of all of (1) plus (2) plus (3) plus (4) of a peroxide catalyst which has a 10 hour half-life in benzene at a temperature in excess of 100°C.

2. The composition of claim 1 further comprising a fluid in which the thermosetting composition is dispersed.

3. The composition of claim 1 in which the fluid is a solvent selected from the group consisting of chlorinated hydrocarbon solvents and aromatic hydrocarbon solvents.

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4. The composition of claim 1 in which the polychlorinated or polybrominated aromatic hydrocarbon is selected from the group consisting of chlorinated or brominated biphenyls, chlorinated or brominated terphenyls, trichlorobenzene, and chlorinated naphthalenes.

5. The composition of claim 1 in which the polyunsaturated monomer is selected from the group consisting of diallyl phthalate, diallyl isophthalate, diallyl chloridate, diallyl maleate, triallyl cyanurate, triallyl iso-

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cyanurate, glycol dimethylacrylate, and a divinyl benzene.

6. The composition of claim 1 in which the peroxide catalyst is selected from the group consisting of dicumyl peroxide, tertiary butyl perbenzoate, 2,5-dimethyl-2,5-di(tertiary-butylperoxy) hexyne-3 and 2,5-dimethyl-2,5-di(tertiary-butylperoxy) hexane.

7. The composition of claim 1 further comprising an inert filler selected from the group consisting of inorganic fillers, fibrous fillers, fibrous mats, woven fabric and non-woven fabric.

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# CHEMOSPHERE

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## CHLORINATED DIOXINS AND RELATED COMPOUNDS 1990—Part 1



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## DATA BASE DESCRIPTION

Three data bases were available for this study. The first and largest data base contained chemical information (PCDD and PCDF data) only; the second contained demographic information on the donors; and the third, which was provided after all other analyses were completed, provided the military/civilian status on each individual. The three data bases were merged using a common sample identifier.

The analytical results, expressed in picogram per gram (pg/g) or equivalently in parts per trillion (ppt), were reported on a total of 17 selected analytes. These are the 2,3,7,8-substituted tetra- through octachloro-PCDD and PCDF congeners, which are considered to be the most toxic. Because two analytes, 1,2,3,4,7,8- and 1,2,3,6,7,8-HxCDD, coeluted, data were available on a total of 16 analytes. For each sample, the lipid-based concentration of each of the selected 16 analytes was given if the analyte was detected. When the analyte was not detected, the limit of detection was reported (also equivalent to lipid concentration). Throughout all analyses, half the limit of detection value was used for those samples with levels below detection limits. This approach was taken to conform with the approach chosen by VA statisticians. When the analyte was detected at a trace level, that is, between the limit of detection and the limit of quantitation, the reported concentration was used in the analyses.

Table 1 summarizes the number of nondetects, traces (TR), and positive quantifiable (PQ) levels for each analyte in the 197 NHATS specimens. An upper limit of 25% for the total number of specimens with levels below detection limit for a given analyte (i.e., at least 75% PQ+TR) was selected as the cutoff point for that analyte to be included in subsequent multivariate analyses. By that rule, a total of five analytes (marked by an \* in Table 1) were excluded from analyses. Table 2 provides distributional statistics for all 16 analytes.

Next, the following demographic information was available for each donor:

- Geographic region (North Central, North East, South, or West), determined by the location of the individual's hospital
- Race of individual (Caucasian, Black, Oriental, American Indian, or other)
- Age of individual (years) at time of specimen collection (i.e., at time of death or surgery)
- Specimen collection year;
- Year of birth; and
- Body mass index (kg/m<sup>2</sup>).

The third data base provided military/civilian status on 195 of the 197 NHATS specimens: 36 Vietnam veterans, 79 non-Vietnam veterans, and 80 civilians. Table 3 displays the distribution of the donors in the three groups across two demographic categories.

**Table 3. Distribution of 195 NHATS Specimens Across Military/Civilian Status, Geographic Region, and Race Groups**

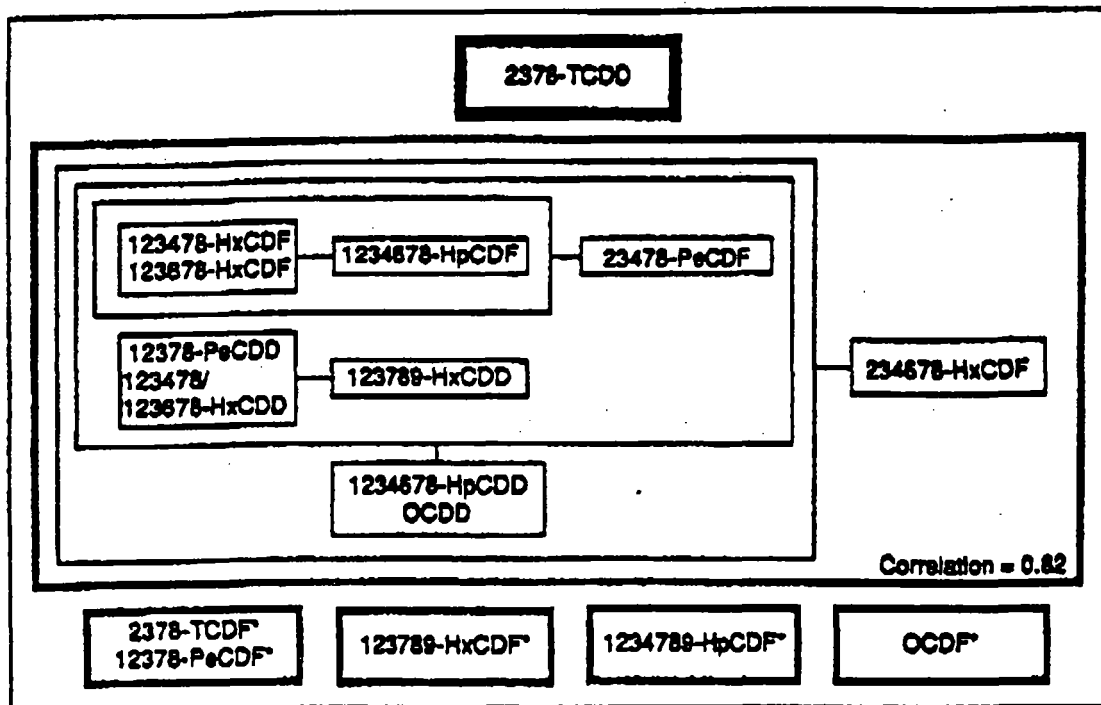
Military/Civilian Status	Geographic Region	Caucasian	Non-Caucasian	Total *1	
				Number	% of 195
Vietnam Veteran	North Central	6	1	7	3.6
	North East	4	2	6	3.1
	South	10	5	15	7.7
	West	7	1	8	4.1
	All	27	9	36	18.5
Non-Vietnam Veteran	North Central	18	2	21	10.8
	North East	16	1	17	8.7
	South	29	6	35	17.9
	West	8	1	9	4.6
	All	69	10	79	40.5
Civilian	North Central	8	2	11	5.6
	North East	22	3	27	13.8
	South	23	11	34	17.4
	West	6	2	8	4.1
	All	60	20	80	41.0
All Specimens	Total	156	39	195	
		80%	20%		

Note \*1: Demographics were unavailable for 2 of the 197 NHATS specimens

#### CORRELATION ANALYSIS OF CONCENTRATION LEVELS

Since both the cluster analysis and the PCA were based on the correlations between variables, correlation coefficients between each pair of analytes were computed. All correlations were positive, and with a few exceptions, most are statistically significantly different from zero at the 99% confidence level. However, due to the large number of specimens (197), even moderately small correlations will be statistically significant. Table 4 lists all correlation coefficients equal to or greater than 0.50. For all practical purposes, anything below that figure (0.50, equivalent to a predictive power of 25%) can be considered negligible in terms of linear association and predictive power.

Of the 120 possible pairs of analyte concentrations, slightly more than half (66) yield correlation coefficients above 0.50. Of these, about half (29) are above 0.80 (predictive power of 64%), with 1,2,3,4,7,8-HxCDF and 1,2,3,6,7,8-HxCDF being the most highly correlated pair of analytes with a coefficient of 0.95. Of the correlations above 0.50, the 2,3,7,8-TCDD shows overall the lowest correlation with most other analytes. Using 0.80 as a cutoff value, one could say that 2,3,7,8-TCDD is not strongly correlated with any of the other analytes.



\*Analytes with high occurrences (over 25%) of non-detects

Notes: The relative position of the analytes in this figure is for illustrative purposes only.

Each rectangle outlines a cluster. One cluster may consist of sub-clusters and individual analytes.

The 6 clusters outlined by heavy dark lines are those formed when a 0.80 cutoff is selected for the similarity measure between clusters.

The 10 analytes within the large rectangle are all highly inter-correlated.

The 4 rectangles at the bottom contain those analytes detected in less than 75% of the specimens.

The cluster on the top shows 2378-TCDD, which does not significantly correlate with any other analyte or group of analytes.

Figure 1. Cluster analysis results of all 16 analytes based on the correlations of the log-transformed concentrations.

#### PRINCIPAL COMPONENT ANALYSIS RESULTS FOR ANALYTES AND DEMOGRAPHIC VARIABLES

PCA was performed on the global data set containing analytical and demographic data. Of the 16 analytes in the data base, those with less than 25% of nondetects were excluded; a total of 11 analytes remained. The demographic variables considered at first were individual's age at sampling, specimen collection year, and body mass index. The inclusion of body mass index reduced the number of specimens to 169, since body mass index was available for only that number. The results, in terms of significant principal components, showed that body mass index did not contribute to explaining any of the variation in the data set. Thus, the decision was made to eliminate that variable from subsequent analyses. A total of 11 analytes (log-transformed concentrations) and 2 demographic variables, age at sampling and specimen collection year, and a total of 195 samples were kept for the final PCA.

The major PCA results are listed below:

- A large proportion (77%) of the variability in the total data set, as determined by the values of the NHATS specimens on all the variables, is explained by the combination of two principal components.
- The first and predominant principal component (explaining 64% of the total variability) is a combination of the higher chlorinated PCDDs and PCDFs, with each analyte having about equal contribution to this principal component. This is consistent with the results of the cluster analysis.
- The second, and by far less important principal component (13% of total variability explained), consists mainly of age at time of death or surgery and specimen collection year.
- A third principal component (6.4% of total variance) consists of lower chlorinated PCDDs and PCDFs, including 2,3,7,8-TCDD.

The profiles of the three principal components are presented in Table 5, along with the variable loadings and their individual contributions (percent variance) to the variance explained by the given principal component. Only those variables with a variance contribution above average (1/13 or 7.7%, where 13 is the number of variables included in the PCA) are shown within each principal component.

Table 5. Principal Component Analysis Results

Variable (ordered by % variance)	Variable Loading	Variance Explained (%)	Cumulative Variance (%)
<b>First Principal Component (64.4% of total variance explained)</b> <i>"Higher Chlorinated Furans and Dioxins"</i>			
123478-HxCDF	0.94	10.33	10.33
123789-HxCDD	0.92	10.02	20.35
123678-HxCDF	0.91	9.99	30.34
123478/123678-HxCDD	0.90	9.71	40.05
OCDD	0.88	9.20	49.25
1234578-HpCDF	0.87	8.98	58.23
1234578-HpCDD	0.86	8.89	67.12
12378-PeCDD	0.85	8.71	75.83
23478-PeCDF	0.83	8.28	84.11
<b>Second Principal Component (12.7% of total variance explained)</b> <i>"Age and Collection Year"</i>			
Age at Sampling	0.92	51.68	51.68
Collection Year	0.84	42.47	94.15
<b>Third Principal Component (6.39% of total variance explained)</b> <i>"Lower Chlorinated Furans and Dioxins"</i>			
234578-HxCDF	0.43	22.09	22.09
1234578-HpCDD	0.37	18.85	38.94
12378-PeCDD	-0.37	18.73	55.67
2378-TCDD	-0.37	18.62	72.30

Of the possible three two-dimensional principal component plots, only two are shown below. Figure 2 depicts principal components 1 vs. 2 (this plot displays about 77% of the variation in the data); Figure 3 depicts principal components 1 vs. 3 (displaying about 71% of the variation). The military/civilian status was used as the labeling code in both plots. Neither plot provides any clear stratification of the specimens, demonstrating that the group of Vietnam veterans in this study exhibits no difference from the two other groups of non-Vietnam veterans and civilians.

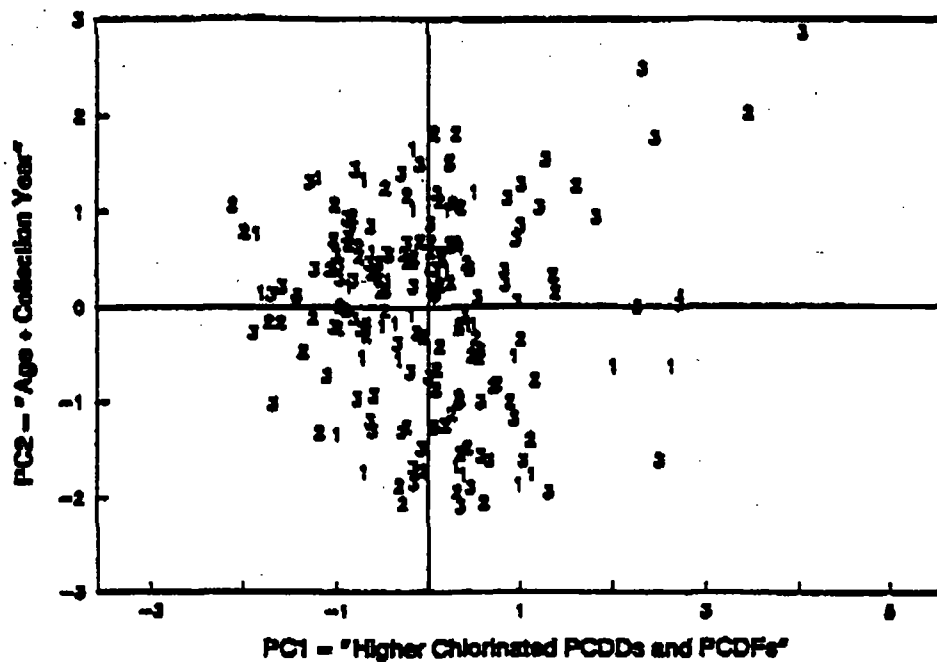
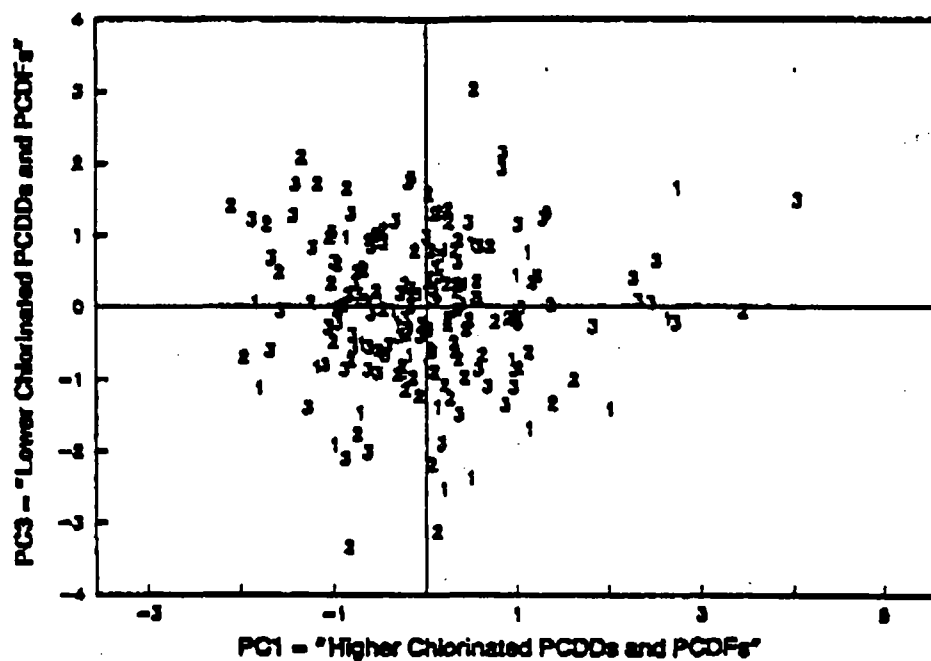


Figure 2. Plot of principal component 2 vs. 1—all 195 specimens coded by military/civilian status



1=Vietnam Veterans (36); 2=Non-Vietnam Veterans (79); 3=Civilians (80)

Figure 3. Plot of principal component 3 vs. 1—all 195 specimens coded by military/civilian status

### CONCLUSIONS

The most important outcome from the analyses performed in this study is that there is no visible stratification of the NHATS specimens in groups as defined by their military service or civilian status with respect to either the PCDD and PCDF levels or the demographic variables. In other words, based on the set of matched NHATS specimens, Vietnam veterans in this study do not exhibit higher levels of PCDDs and PCDFs, including 2,3,7,8-TCDD, than either the non-Vietnam veterans or the control group of civilians.

The results of these multivariate statistical analysis efforts provide evidence of the interrelationships of PCDDs and PCDFs that could be possible indicators of sources of exposure. That is, the source of 2,3,7,8-TCDD may be completely different from those of the other PCDDs and PCDFs. Further statistical evaluations of body burden data may require more comprehensive data bases that identify body burden half-life of the higher chlorinated PCDDs and PCDFs vs. 2,3,7,8-TCDD, as well as the potential contributions arising from commercial products and environmental matrices (e.g., incinerator emissions, ambient air, water, fish, etc.)

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# SULFIDES

Boring No.		2	2	2	2	2	3	3	3	3	3	11	
Sample No.		92-1949	92-1950	92-1951	92-1952	92-1954	92-1861	92-1862	92-1863	92-1864	92-1871	92-1911	
FIELD I.D.		0200.08	0208.11	0238.48	0292.102	EQBLK02	0307.15	0315.18.5	0331.43	0355.63	003	1100.06	
		Soil	Soil	Soil	Soil	Water	Soil	Soil	Soil	Soil	Water	Soil	
Chemical Name	ACTION LEVEL	Det. Limit (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/L)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/L)	Conc. (mg/Kg)
Sulfides			82	50	<31	<31	<1.0	<62	<62	<39	<40	<1.0	<35

# **SULFIDES**

Boring No.			11	11	11	17	17	18	18	18	22	22	27
Sample No.			92-1912	92-1913	92-1914	92-1768	92-1769	92-1747	92-1748	92-1749	92-1962	92-1963	92-1753
FIELD I.D.			1106.11	1140.46	1132.38	1702.12	1715.24	1800.10	1824.34	1814.20	2209.15	2241.49	2702.12
			Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil
Chemical Name	ACTION LEVEL	Det. Limit (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)
Sulfides			48	<31	<29	33	41	<46	<35	<42	<33	<32	37



**SULFIDES**

Boring No.		27	28	28	28	31	31	35	35	37	37	46
Sample No.		92-1755	92-1759	92-1760	92-1761	92-1775	92-1776	92-1797	92-1798	92-1984	92-1985	92-1802
FIELD I.D.		2734.38	2802.11.5	2827.37	2840.50	3100.10	3114.24	3500.10	3534.42	1005.11	1019.27	4600.08
		Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil
Chemical Name	ACTION LEVEL	Det. Limit (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)
Sulfides			35	57	40	40	<34	<37	52	33	36	<32
												<31

**SULFIDES**

Boring No.	46	51	51	51	51	51	62	62	62	66	66	
Sample No.	92-1803	92-1897	92-1898	92-1899	92-1900	92-1953	92-1955	92-1956	92-1957	92-1855	92-1856	
FIELD I.D.	4622.32	5100.10	5110.18	5136.46	5146.54.5	EQBLK51	6200.10	6212.22	6228.36	6600.08	6612.22	
	Soil	Soil	Soil	Soil	Soil	Water	Soil	Soil	Soil	Soil	Soil	
Chemical Name	ACTION LEVEL	Det. Limit (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/L)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)
Sulfides		36	<30	<34	<28	<28	<1.0	<26	<30	<29	<37	250

**SULFIDES**

Boring No.	66	66
Sample No.	92-1857	92-1870
FIELD I.D.	6628.38	EQBLK66
	Soil	Water

Chemical Name	ACTION LEVEL	Det. Limit (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/L)
Sulfides			66	<1.0

SULFIDES

Boring No.			Hydrant	A4	A4	E-5	E-5	E-5	E-5	E-5	F4	F4
Sample No.			92-2130	92-2707	92-2708	92-2777	92-2778	92-2779	92-2780	92-2795	92-2677	92-2678
FIELD I.D.				A40004	A42227	E55282	E53844	E51016	E50208	ELQBLKE5	F40.502	F45664
			Water	W. SOIL	W. SOIL	W. SOIL	W. SOIL	W. SOIL	W. SOIL	Water	W. SOIL	W. SOIL
Chemical Name	ACTION LEVEL	Det. Limit (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/L)	Conc. (mg/Kg)	Conc. (mg/Kg)
Sulfides			<2	<13	<16	<35	<36	<29	<34	<1.0	<0.050	<0.050

CYANIDE

Boring No.			2	2	2	2	2	3	3	3	3	3	11
Sample No.			92-1949	92-1950	92-1951	92-1952	92-1954	92-1861	92-1862	92-1863	92-1864	92-1871	92-1911
FIELD I.D.			0200.08	0208.11	0238.48	0292.102	EQBLK02	0307.15	0315.18.5	0331.43	0355.63	EQBLK03	1100.08
			Soil	Soil	Soil	Soil	Water	Soil	Soil	Soil	Soil	Water	Soil
Chemical Name	ACTION LEVEL	Det. Limit (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (Ug/Lg)	Conc. (mg/Kg)
Cyanide	0.2		8.30	1.70	0.62	0.99	10.00	1.00	1.00	0.65	0.68	10.00	3.30

CYANIDE

Boring No.			11	11	11	17	17	18	18	18	22	22	27
Sample No.			92-1912	92-1913	92-1914	92-1768	92-1769	92-1747	92-1748	92-1749	92-1962	92-1963	92-1753
FIELD I.D.			1106.11	1140.46	1132.38	1702.12	1715.24	1800.10	1824.34	1814.20	2209.15	2241.49	2702.12
			Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil
Chemical Name	ACTION LEVEL	Det. Limit (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)
Cyanide	0.2		3.00	0.62	0.59	0.55	0.67	6.90	0.59	0.70	0.67	0.64	2.90

CYANIDE

Boring No.			27	28	28	28	31	31	35	35	37	37	46
Sample No.			92-1755	92-1759	92-1760	92-1761	92-1775	92-1776	92-1797	92-1798	92-1984	92-1985	92-1802
FIELD I.D.			2734.38	2802.11.5	2827.37	2840.50	3100.10	3114.24	3500.10	3534.42	1005.11	1019.27	4600.08
			Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil	Soil
Chemical Name	ACTION LEVEL	Det. Limit (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)
Cyanide	0.2		0.58	4.80	0.67	0.66	0.57	0.62	3.40	0.55	0.65	0.65	0.52

CYANIDE

Boring No.			46	51	51	51	51	51	62	62	62	66	66
Sample No.			92-1803	92-1897	92-1898	92-1899	92-1900	92-1953	92-1955	92-1956	92-1957	92-1855	92-1856
FIELD I.D.			4622.32	5100.10	5110.18	5136.46	5146.54.5	EQBLK51	6200.10	6212.22	6228.36	6600.08	6612.22
			Soil	Soil	Soil	Soil	Soil	Water	Soil	Soil	Soil	Soil	Soil
Chemical Name	ACTION LEVEL	Det. Limit (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (Ug/Lg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)
Cyanide	0.2		0.59	0.60	0.66	0.55	0.55	10.00	0.52	0.61	0.58	0.61	3.20



**CYANIDE**

<b>Boring No.</b>	<b>66</b>	<b>66</b>
<b>Sample No.</b>	<b>92-1857</b>	<b>92-1870</b>
<b>FIELD I.D.</b>	<b>6626.36</b>	<b>EQBLK66</b>
	<b>Soil</b>	<b>Water</b>

<b>Chemical Name</b>	<b>ACTION LEVEL</b>	<b>Det. Limit (mg/Kg)</b>	<b>Conc. (mg/Kg)</b>	<b>Conc. (mg/Kg)</b>
<b>Cyanide</b>	<b>0.2</b>		<b>0.65</b>	<b>10.00</b>

## CYANIDE

## WELL SOIL

Boring No.			Hydrant	A4	A4	E-5	E-5	E-5	E-5	E-5	F4	F4
Sample No.			92-2130	92-2707	92-2708	92-2777	92-2778	92-2779	92-2780	92-2795	92-2877	92-2678
FIELD I.D.				A40004	A42227	E55262	E53844	E51016	E50208	ELQBLKE5	F40.502	F45664
			Water	W. SOIL	W. SOIL	W. SOIL	W. SOIL	W. SOIL	W. SOIL	W Soil	W. SOIL	W. SOIL
Chemical Name	ACTION LEVEL	Det. Limit (mg/Kg)	Conc. (Ug/L)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)	Conc. (mg/Kg)
Cyanide	0.2		<10	0.95	<0.05	<0.010	<0.010	<0.010	<0.010	<0.01	<0.050	<0.050

PCDD's & PCDF's

Boring No.	2	2	2	2	2	3	3	3	3	3	11
Sample No.	92-1949	92-1950	92-1951	92-1952	92-1954	92-1861	92-1862	92-1863	92-1864	92-1871	92-1911
FIELD I.D.	0200.08	0208.11	0238.48	0292.102	EQBLK02	0307.15	0315.18.5	0331.43	0355.63	EQBLK03	1100.06
	Soil	Soil	Soil	Soil	Water	Soil	Soil	Soil	Soil	Water	Soil
Chemical Name	ACTION LEVEL	Det. Limit (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)
2378-TCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND
12378-PeCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND
123478-HxCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND
123678-HxCDD			0.16	ND	ND	ND	ND	ND	ND	ND	ND
123789-HxCDD			0.18	ND	ND	ND	ND	ND	ND	ND	ND
1234678-HpCDD			2.30	0.29	ND	ND	ND	ND	ND	ND	1.70
OCDD			4.60	0.96	ND	0.42	ND	0.35	0.50	0.13	0.20
2378-TCDF			2.40	0.25	ND	ND	ND	ND	ND	ND	3.20
12378-PeCDF			1.50	0.25	ND	ND	ND	ND	ND	ND	1.60
23478-PeCDF			5.10	1.10	ND	ND	ND	ND	ND	ND	7.40
123478-HxCDF			7.40	1.70	ND	ND	ND	ND	ND	ND	9.60
123678-HxCDF			1.50	0.27	ND	ND	ND	ND	ND	ND	1.60
234678-HxCDF			4.50	1.10	ND	ND	ND	ND	ND	ND	6.00
123789-HxCDF			ND	ND	ND	ND	ND	ND	ND	ND	ND
1234678-HpCDF			13.10	4.20	ND	ND	ND	ND	ND	ND	14.80
1234789-HpCDF			ND	ND	ND	ND	ND	ND	ND	ND	ND
OCDF			0.73	0.23	ND	ND	ND	ND	ND	ND	0.62
TOTAL TCDD			0.52	ND	ND	ND	0.67	ND	ND	ND	0.06
TOTAL PeCDD			0.58	ND	ND	ND	ND	ND	ND	ND	ND
TOTAL HxCDD			2.60	ND	ND	ND	ND	ND	ND	ND	0.11
TOTAL HpCDD			2.30	0.46	ND	ND	ND	ND	ND	ND	2.90
TOTAL TCDF			8.10	0.51	ND	ND	ND	ND	ND	ND	9.30
TOTAL PeCDF			21.30	3.70	ND	ND	ND	ND	ND	ND	25.70
TOTAL HxCDF			30.40	6.40	ND	ND	ND	0.08	ND	ND	35.10
TOTAL HpCDF			14.40	4.50	ND	ND	ND	ND	ND	ND	16.70

## PCDD's &amp; PCDF's

Boring No. Sample No. FIELD I.D.			11 92-1912 1106.11 Soil	11 92-1913 1140.46 Soil	11 92-1914 1132.38 Soil	17 92-1768 1702.12 Soil	17 92-1769 1715.24 Soil	18 92-1747 1800.10 Soil	18 92-1748 1824.34 Soil	18 92-1749 1814.20 Soil	22 92-1962 2209.15 Soil	22 92-1963 2241.49 Soil	27 92-1753 2702.12 Soil
Chemical Name	ACTION LEVEL	Det. Limit (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)
2378-TCDD			ND	ND	ND	ND	ND	ND	ND		ND	ND	ND
12378-PeCDD			ND	ND	ND	ND	ND	ND	ND		ND	ND	ND
123478-HxCDD			ND	ND	ND	ND	ND	ND	ND		ND	ND	ND
123678-HxCDD			ND	ND	ND	ND	ND	ND	ND		ND	ND	ND
123789-HxCDD			ND	ND	ND	ND	ND	ND	ND		ND	ND	ND
1234678-HpCDD			0.55	ND	ND	ND	ND	1.30	ND		ND	ND	0.83
OCDD			1.10	0.14	0.16	0.10	ND	4.10	ND		0.09	0.49	2.20
2378-TCDF			0.36	ND	ND	0.01	ND	3.40	ND		ND	ND	0.41
12378-PeCDF			0.43	ND	ND	ND	ND	2.40	ND		ND	ND	0.27
23478-PeCDF			1.30	ND	ND	ND	ND	7.80	0.02		ND	ND	1.20
123478-HxCDF			4.10	ND	ND	0.36	ND	14.40	ND		ND	ND	1.50
123678-HxCDF			0.78	ND	ND	0.04	ND	1.90	ND		ND	ND	0.22
234678-HXCDF			2.50	ND	ND	0.18	ND	8.30	ND		ND	ND	0.82
123789-HxCDF			ND	ND	ND	ND	ND	ND	ND		ND	ND	ND
1234678-HpCDF			16.20	ND	ND	1.40	0.03	37.60	0.13		0.01	ND	3.60
1234789-HpCDF			ND	ND	ND	ND	ND	0.19	ND		ND	ND	ND
OCDF			0.92	ND	ND	0.05	ND	1.20	ND		ND	ND	1.10
TOTAL TCDD			ND	ND	ND	ND	ND	ND	ND		ND	ND	0.09
TOTAL PeCDD			ND	ND	ND	ND	ND	ND	ND		0.02	ND	ND
TOTAL HxCDD			ND	ND	ND	ND	ND	ND	ND		ND	ND	ND
TOTAL HpCDD			0.88	ND	ND	ND	ND	1.90	ND		ND	ND	1.30
TOTAL TCDF			1.30	ND	ND	0.03	ND	10.40	ND		ND	ND	0.79
TOTAL PeCDF			8.00	ND	ND	0.22	ND	34.90	0.06		ND	ND	5.00
TOTAL HxCDF			19.60	ND	ND	1.80	ND	58.40	ND		ND	ND	5.30
TOTAL HpCDF			18.30	ND	ND	1.50	0.03	43.90	0.15		0.01	ND	4.30

## PCDD's &amp; PCDF's

Boring No. Sample No. FIELD I.D.	27 92-1755 2734.38 Soil	28 92-1759 2802.11.5 Soil	28 92-1760 2827.37 Soil	28 92-1761 2840.50 Soil	31 92-1775 3100.10 Soil	31 92-1776 3114.24 Soil	35 92-1797 3500.10 Soil	35 92-1798 3534.42 Soil	37 92-1984 1005.11 Soil	37 92-1985 1019.27 Soil	46 92-1802 4600.08 Soil
Chemical Name	ACTION LEVEL	Det. Limit (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)
2378-TCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND
12378-PeCDD			ND	ND	ND	ND	0.11	ND	ND	ND	ND
123478-HxCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND
123678-HxCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND
123789-HxCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND
1234678-HpCDD			ND	0.37	ND	ND	0.20	ND	ND	ND	ND
OCDD			ND	0.79	0.13	ND	3.90	0.32	0.07	ND	2.30
2378-TCDF			ND	ND	ND	ND	0.16	ND	0.26	ND	ND
12378-PeCDF			ND	0.09	ND	ND	0.12	ND	0.25	ND	ND
23478-PeCDF			ND	0.38	ND	ND	0.47	ND	1.00	ND	ND
123478-HxCDF			ND	0.65	ND	ND	1.40	ND	5.10	ND	ND
123678-HxCDF			ND	0.10	ND	ND	0.22	ND	0.85	ND	ND
234678-HxCDF			ND	0.41	ND	ND	0.87	ND	3.30	ND	ND
123789-HxCDF			ND	ND	ND	ND	ND	ND	ND	ND	ND
1234678-HpCDF			ND	2.40	ND	ND	7.20	0.04	16.10	0.03	0.007
1234789-HpCDF			ND	ND	ND	ND	ND	ND	ND	ND	ND
OCDF			ND	0.14	ND	ND	0.61	ND	1.20	ND	ND
TOTAL TCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND
TOTAL PeCDD			ND	ND	ND	ND	ND	0.11	ND	ND	ND
TOTAL HxCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND
TOTAL HpCDD			ND	0.54	ND	ND	0.37	ND	ND	ND	ND
TOTAL TCDF			ND	0.33	ND	ND	0.43	ND	1.10	ND	ND
TOTAL PeCDF			ND	1.60	ND	ND	2.40	ND	5.80	ND	ND
TOTAL HxCDF			ND	2.60	ND	ND	6.50	ND	27.60	ND	ND
TOTAL HpCDF			ND	2.70	ND	ND	8.10	0.04	18.60	0.03	0.008

PCDD's & PCDF's

Boring No.	46	51	51	51	51	51	62	62	62	66	66
Sample No.	92-1803	92-1897	92-1898	92-1899	92-1900	92-1953	92-1955	92-1956	92-1957	92-1855	92-1856
FIELD I.D.	4622.32	5100.10	5110.18	5136.46	5146.54.5	EQBLK51	6200.10	6212.22	6228.36	6600.08	6612.22
	Soil	Soil	Soil	Soil	Soil	Water	Soil	Soil	Soil	Soil	Soil
Chemical Name	ACTION LEVEL	Det. Limit (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)
2378-TCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND
12378-PeCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND
123478-HxCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND
123678-HxCDD			ND	ND	0.12	ND	ND	ND	ND	ND	ND
123789-HxCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND
1234678-HpCDD			ND	ND	5.50	ND	ND	ND	ND	ND	ND
OCDD			0.01	0.72	30.60	ND	ND	ND	0.97	0.37	0.91
2378-TCDF			ND	ND	ND	ND	ND	ND	ND	0.04	ND
12378-PeCDF			ND	ND	ND	ND	ND	ND	ND	ND	ND
23478-PeCDF			ND	ND	ND	ND	ND	ND	ND	ND	ND
123478-HxCDF			ND	0.03	0.07	ND	ND	ND	ND	0.62	ND
123678-HxCDF			ND	ND	ND	ND	ND	ND	ND	0.07	ND
234678-HxCDF			ND	ND	ND	ND	ND	ND	ND	0.32	ND
123789-HxCDF			ND	ND	ND	ND	ND	ND	ND	ND	ND
1234678-HpCDF			ND	0.07	1.30	ND	ND	ND	ND	1.60	ND
1234789-HpCDF			ND	ND	0.04	ND	ND	ND	ND	ND	ND
OCDF			ND	ND	4.80	ND	ND	ND	ND	0.19	ND
TOTAL TCDD			ND	ND	ND	ND	0.88	ND	ND	ND	ND
TOTAL PeCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND
TOTAL HxCDD			ND	ND	0.21	ND	ND	ND	ND	ND	ND
TOTAL HpCDD			ND	ND	8.70	ND	ND	ND	ND	ND	ND
TOTAL TCDF			ND	ND	ND	ND	ND	ND	ND	0.23	ND
TOTAL PeCDF			ND	ND	ND	ND	ND	ND	ND	1.10	ND
TOTAL HxCDF			ND	0.10	1.20	ND	ND	ND	ND	2.60	ND
TOTAL HpCDF			ND	0.08	6.00	ND	ND	ND	ND	1.70	ND

PCDD's & PCDF's

Boring No.	66	66
Sample No.	92-1857	92-1870
FIELD I.D.	6626.36	EQBLK66
	Soil	Water

Chemical Name	ACTION LEVEL	Det. Limit (ppb)	Conc. (ppb)	Conc. (ppb)
2378-TCDD			ND	ND
12378-PeCDD			ND	ND
123478-HxCDD			ND	ND
123678-HxCDD			ND	ND
123789-HxCDD			ND	ND
1234678-HpCDD			ND	ND
OCDD			ND	ND
2378-TCDF			ND	ND
12378-PeCDF			ND	ND
23478-PeCDF			ND	ND
123478-HxCDF			ND	ND
123678-HxCDF			ND	ND
234678-HxCDF			ND	ND
123789-HxCDF			ND	ND
1234678-HpCDF			ND	ND
1234789-HpCDF			ND	ND
OCDF			ND	ND
TOTAL TCDD			ND	ND
TOTAL PeCDD			ND	ND
TOTAL HxCDD			ND	ND
TOTAL HpCDD			ND	ND
TOTAL TCDF			ND	ND
TOTAL PeCDF			ND	ND
TOTAL HxCDF			ND	ND
TOTAL HpCDF			ND	ND

## PCDD's &amp; PCDF's

## WELL SOIL

Boring No. Sample No. FIELD I.D.			Hydrant 92-2130	A4 92-2707 A40004 W. SOIL	A4 92-2708 A42227 W. SOIL	E-5 92-2777 E55262 W. SOIL	E-5 92-2778 E53844 W. SOIL	E-5 92-2779 E51016 W. SOIL	E-5 92-2780 E50208 W. SOIL	E-5 92-2795 ELQBLKE5 W Soil	F4 92-2677 F40.502 W. SOIL	F4 92-2678 F45664 W. SOIL
Chemical Name	ACTION LEVEL	Det. Limit (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppb)	Conc. (ppm)	Conc. (ppb)	Conc. (ppb)
2378-TCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
12378-PeCDD				ND	ND	ND	ND	ND	ND	ND	ND	ND
123478-HxCDD				ND	ND	ND	ND	ND	ND	ND	ND	ND
123678-HxCDD				ND	ND	ND	ND	ND	ND	ND	ND	ND
123789-HxCDD				ND	ND	ND	ND	ND	ND	ND	ND	ND
1234678-HpCDD				ND	ND	ND	ND	ND	0.360	1.400	0.059	ND
OCDD				0.073	ND	ND	ND	0.380	1.200	11.000	0.660	ND
2378-TCDF				ND	ND	ND	ND	0.053	0.680	ND	0.120	ND
12378-PeCDF				ND	ND	ND	ND	ND	0.560	ND	0.067	ND
23478-PeCDF				ND	ND	ND	ND	0.830	1.400	4.300	0.400	ND
123478-HxCDF				ND	ND	ND	ND	0.100	1.500	ND	0.990	ND
123678-HxCDF				ND	ND	ND	ND	0.016	0.270	ND	0.140	ND
234678-HXCDF				ND	ND	ND	ND	0.073	0.930	ND	0.720	ND
123789-HxCDF				ND	ND	ND	ND	ND	ND	ND	ND	ND
1234678-HpCDF				ND	ND	ND	ND	0.210	2.200	ND	2.900	ND
1234789-HpCDF				ND	ND	ND	ND	ND	ND	ND	ND	ND
OCDF				ND	ND	ND	ND	0.360	0.310	ND	0.310	ND
TOTAL TCDD			ND	ND	ND	ND	ND	ND	0.058	ND	ND	ND
TOTAL PeCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
TOTAL HxCDD			ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
TOTAL HpCDD				ND	ND	ND	ND	ND	0.670	1.400	0.059	ND
TOTAL TCDF			ND	ND	ND	ND	ND	0.120	2.400	ND	0.300	ND
TOTAL PeCDF			ND	ND	ND	ND	ND	0.300	6.000	4.200	1.300	ND
TOTAL HxCDF			ND	ND	ND	ND	ND	0.410	5.600	39.000	3.900	ND
TOTAL HpCDF				ND	ND	ND	ND	0.240	2.700	1.800	3.500	ND



**FILE COPY**

Raymark Industries, Inc.  
RCRA Section 3013 Order  
Docket No. 87-1057  
ELI #91-545-10

AROCOR 1268 VS. PCDD/PCDF TEF PHASE II A DATA TABLE			
LAB ID	FIELD ID	PCB - 1268	TEF
92-1747	18 00.10	190 ppm *	7.22
92-1748	18 24.34	ND < 0.1 ppm	0.0113
92-1749	18 14.20	BDL < 0.1 ppm	ND
92-1753	27 02.12	120 ppm *	0.956
92-1755	27 34.38	ND < 0.1 ppm	ND
92-1759	28 02.11.5	40 ppm *	0.339
92-1760	28 27.37	ND < 0.1 ppm	0.00013
92-1761	28 40.50	ND < 0.1 ppm	ND
92-1768	17 02.12	4 ppm *	0.0732
92-1769	17 15.24	0.2 ppm *	0.0003
92-1775	31 0.10	50 ppm *	0.585
92-1776	31 14.24	0.6 ppm *	0.0004
92-1797	35 00.10	190 ppm *	1.681
92-1798	35 34.42	ND < 0.1 ppm	0.00037
92-1802	46 00.08	ND < 0.1 ppm	0.00237
92-1803	46 22.32	ND < 0.1 ppm	0.00001
92-1855	66 00.08	30 ppm *	0.121
92-1856	66 12.22	ND < 0.1 ppm	0.00091
92-1857	66 26.36	ND < 0.1 ppm	ND
92-1861	03 07.15	1 ppm *	0.00035
92-1862	03 15.18.5	3 ppm *	0.0005
92-1863	03 31.43	ND < 0.1 ppm	0.00013

<b>AROCLOR 1268 VS. PCDD/PCDF TEF PHASE II A DATA TABLE</b>			
<b>LAB ID</b>	<b>FIELD ID</b>	<b>PCB - 1268</b>	<b>TEF</b>
92-1864	03 55.63	ND < 0.1 ppm	0.0002
92-1870	EQBLK66	ND < 1.0 ppb	ND
92-1871	EQBLK03	ND < 1.0 ppb	ND
92-1897	51 00.10	BDL < 1.0 ppm	0.00442
92-1898	51 10.18	BDL < 1.0 ppm	0.1228
92-1899	51 36.46	ND < 0.1 ppm	ND
92-1900	51 46.54.5	ND < 0.1 ppm	ND
92-1911	11 00.06	90 ppm *	5.99
92-1912	11 06.11	150 ppm *	1.62
92-1913	11 40.45	ND < 0.1 ppm	0.00014
92-1914	11 32.38	ND < 0.1 ppm	0.00016
92-1949	02 00.08	30 ppm *	4.4
92-1950	02 08.11	30 ppm *	0.94
92-1951	02 38.48	ND < 1.0 ppm	ND
92-1952	02 92.102	ND < 1.0 ppm	0.00042
92-1953	EQBLK51	ND < 1.0 ppb	ND
92-1954	EQBLK02	ND < 1.0 ppb	ND
92-1955	62 00.10	ND < 1.0 ppm	ND
92-1956	62 12.22	2 ppm *	0.00097
92-1957	62 28.36	ND < 1.0 ppm	0.00037
92-1962	22 09.15	BDL < 0.1 ppm	0.00019
92-1963	22 41.49	ND < 0.1 ppm	0.00049
92-1984	37 05.11	1 ppm *	ND
92-1985	37 19.27	ND < 0.1 ppm	0.00008
92-2130	Hydrant	ND < 1.0 ppb	ND

**AROCLOR 1268 VS. PCDD/PCDF TEF  
PHASE II A DATA TABLE**

<b>LAB ID</b>	<b>FIELD ID</b>	<b>PCB - 1268</b>	<b>TEF</b>
92-2677	F4 0.5.02	18.9 ppm *	0.431
92-2678	F4 56.64	ND < 0.1 ppm	ND
92-2707	A4 00.04	ND < 0.1 ppm	0.000073
92-2708	A4 22.27	ND < 0.1 ppm	ND
92-2777	E5 52.62	ND < 0.1 ppm	ND
92-2778	E5 38.44	ND < 0.1 ppm	ND
92-2779	E5 10.16	69.5 ppm *	0.442
92-2780	E5 02.08	48.6 ppm *	1.093
92-2795	EQBLKE5	ND < 1.0 ppb	0.00217
92-3176	GWE1-1	ND < 1.0 ppb	ND
92-3177	GWE4-1	ND < 1.0 ppb	ND
92-3178	FdBLK	ND < 1.0 ppb	
92-3195	GWE3	ND < 1.0 ppb	ND
92-3196	FdBLK	ND < 1.0 ppb	
92-3228	GWE5	ND < 1.0 ppb	ND
92-3232	GWE2	ND < 1.0 ppb	ND
92-3234	FdBLK	ND < 1.0 ppb	
92-3242	GWA3	ND < 1.0 ppb	ND
92-3244	FdBLK	ND < 1.0 ppb	
92-3250	GWA5	ND < 1.0 ppb	ND
92-3252	GWA2	ND < 1.0 ppb	ND
92-3254	FdBLK	ND < 1.0 ppb	
92-3303	GWF1-1	3.5 ppb	ND
92-3305	GWE6	ND < 1.0 ppb	ND
92-3307	FdBLK	ND < 1.0 ppb	

<b>AROCLOR 1268 VS. PCDD/PCDF TEF PHASE II A DATA TABLE</b>			
<b>LAB ID</b>	<b>FIELD ID</b>	<b>PCB - 1268</b>	<b>TEF</b>
92-3325	GWF2	ND < 1.0 ppb	ND
92-3329	GWF3	ND < 1.0 ppb	ND
92-3332	GWF4	ND < 1.0 ppb	ND
92-3335	FdBLK	ND < 1.0 ppb	
92-3343	EQBLK	ND < 1.0 ppb	
92-3491	BD1/DP22 #8	ND ppm	0.065493
92-3502	BD7/DP43 #5	4.06 ppm	ND
92-3504	BD29/30DP3/9 #4a	27.7 ppm	0.002634
93-0042	BD16/DP21 #4b	62.8 ppm	ND
93-0049	DB1/DP2 #2	ND ppm	0.000651
93-0051	BD1/DP22 #3a	ND ppm	ND
93-0117	BD6/DP6 #1	ND ppm	0.000628
93-0119	BD6/DP2 #7	ND ppm	ND
93-0233	BD1/DP22 #3b	ND ppm	ND
93-0234	BD4/DP42 #6	ND ppm	ND
93-0235	BD5a/DP5 #9	ND ppm	ND

\* Approximate concentration

RJD/jd  
036RM



**ENVIRONMENTAL LABORATORIES, INC.**

142 TEMPLE STREET  
NEW HAVEN, CT 06510  
(203) 789-1280  
FAX (203) 789-8281

April 21, 1993

Mr. Arthur Wing  
U.S. Environmental Protection Agency  
60 Westview Street  
Lexington, MA 02173

RE: Raymark Industries, Inc.  
CERCLA Order Docket No. I921072  
Extension Request Number 2  
ELI #93-563-16

Dear Mr. Wing:

Environmental Laboratories, Inc. (ELI) is writing in behalf of Raymark Industries, Inc. to request an extension to our April 30, 1993 deadline. The April 30, 1993 deadline is an extension to the original Work Order which was modified as a result of discoveries in Lagoon 4 and in conjunction with analytical data identified in the RCRA Phase IIA remedial investigation. The discovery of dioxin/furan has resulted in delays which necessitates this request.

ELI is providing a schedule of activities to complete the required CERCLA Work Order. The schedule is based upon various assumptions regarding approvals by the EPA for modifications to Work Plans and upon the completion and data validation of additional dioxin/furans sample analysis. ELI proposes to complete the placement of topsoil and seeding of settling basins 1, 2 and 3 in conjunction with the closure of Lagoon 4. These work tasks are expected to be completed by May 11, 1993. ELI anticipates that the analytical analysis for the waste pile will not be completed until May 3, 1993. Upon completion of the dioxin/furan analysis, the waste pile will be removed.

ELI is currently completing the TCLP/Hazardous Waste characterizations of the interior samples (i.e. pits, sumps and tanks). Once the analysis and a QA/QC review have been completed, the data will be submitted. If the material present is found to be hazardous in nature, it will be manifested and removed to an approved landfill. ELI anticipates that the removal of hazardous material in pits, sumps and tanks will be completed by May 31, 1993.



Mr. Arthur Wing  
U.S. Environmental Protection Agency

- 2 -

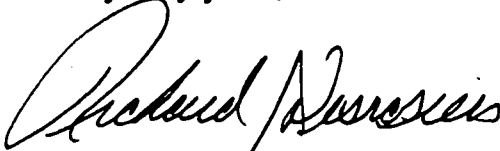
April 21, 1993  
ELI #93-563-16

The hydrologic analysis will be completed once approval is received on the modified sample locations. ELI anticipates that the one year storm event will occur within the next sixty days dependant upon local weather conditions. The hydrologic analysis will be completed as rapidly as possible based upon weather conditions. ELI does not anticipate physical delays within the hydrologic analysis. The hydrologic analysis does not have as rigid completion date (as outlined within the Order) as the settling basins 1, 2 and 3 and Lagoon 4, the waste pile, or the removal of internal hazardous material. Raymark does understand the urgency of the hydrologic analysis and is willing to expedite this study wherever possible.

Raymark Industries, Inc., will proceed as rapidly as possible to complete the required tasks outlined in the Work Plan. We request that the EPA approve an additional sixty days (June 30, 1993) for the completion of the above Work Order requirements. This sixty day time frame should allow for the final placement of topsoil and seeding at settling basins 1, 2 and 3; the temporary closure to Lagoon 4; the removal of hazardous material, and the removal of the waste pile. ELI requests approval to this extension so that we may continue to initiate these projects in a timely manner.

If you have any questions regarding this extension, please do not hesitate to contact us.

Very truly yours,



Richard J. Desrosiers  
Project Manager

RJD/gdb  
034RL/1

Enclosure

cc: Mr. Thomas O'Connor, Connecticut DEP  
Mr. Michael Hill, U.S. EPA, Region I  
Mr. Robert Moody, Raymark Industries, Inc.  
Mr. Mark Zimmerman, Updike, Kelly and Spellacy, P.C.  
Mr. Robert G. Wasp, P.E., Environmental Laboratories, Inc.

REVISED GENERAL SCHEDULE  
DOCKET NO. I-92-1072  
RAYMARK INDUSTRIES, INC.

April		May				June			July
19	26	3	10	17	24	31	7	14	21
									28
									6

**Tanks/Sumps/Pits**

Chemical Analysis QA/QC  
Material Removal



**Settling Basins 1,2,3**

Topsoil and Seed  
Outside Fence Areas



**Lagoon 4**

EPA Approval  
Order Materials  
Remove Trees and Brush  
Install Liners  
Install 2 Inch Stone  
Protect Inlets/Outlets



**Waste Pile**

PCCD/PCDF Analysis  
Removal of Waste Pile  
Decontaminate Pad  
Collect and Analyze Pad



**Hydrologic Study**

EPA Sampling Approval  
Sample 1 Year Storm



**ENVIRONMENTAL LABORATORIES, INC.**

142 TEMPLE STREET  
NEW HAVEN, CT 06510  
(203) 789-1260  
FAX (203) 789-8261

April 19, 1993

Mr. Michael Hill  
U.S. Environmental Protection Agency  
Region I  
JFK Federal Building  
Mail Code HEE-CAN6  
Boston, MA 02203-2211

RE: Raymark Industries, Inc.  
RCRA Order Docket No. I-87-1057  
PCB Aroclor - 1268 Sample Results  
ELI No. 91-545-10

Dear Mr. Hill:

Pursuant to your April 16, 1993 letter to Raymark Industries, Inc., Environmental Laboratories, Inc. (ELI) is enclosing the results of the Aroclor-1268 data. As you are aware, the Appendix IX analyte list included within the Raymark Work Plan does not include Aroclor 1268 or 1262. Therefore, these Aroclor analytes were not analyzed during the Phase IIA site investigation, but will be included in all subsequent analyses.

The tabulated Aroclor-1268 data attached has been flagged by our laboratory as estimated values at best. These results are submitted as supplemental data in conjunction with the Phase IIA analytical data package provided to your office. The method of analysis is provided with the case narrative attached with the sample results.





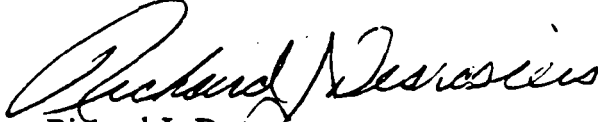
Mr. Michael Hill  
ELI #91-545-10

- 2 -

U.S. Environmental Protection Agency  
April 19, 1993

If you have any questions regarding this data, please do not hesitate to contact us. ELI is currently assessing if the Aroclor-1262 data can be analyzed for using similar methods. Once we have assessed this analyte, we will forward the data under separate cover.

Sincerely,



Richard J. Desrosiers  
Project Manager/Senior Hydrogeologist

RJD/gdb  
034RL/2

Enclosure

cc: Mr. Robert Moody, Raymark Industries, Inc.  
Mr. Mark Zimmerman, Updike, Kelly and Spellacy, P.C.  
Mr. Robert G. Wasp, P.E., Environmental Laboratories, Inc.



**ENVIRONMENTAL LABORATORIES, INC.**

142 TEMPLE STREET  
NEW HAVEN, CT 06510  
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Mr. Robert F. Moody  
Raymark Industries, Inc.  
75 East Main Street  
Stratford, CT 06497

## **CASE NARRATIVE**


The following table contains information regarding concentrations of PCB-1268 associated with samples collected from Raymark Industries. This is in addition to the analytical reports submitted by ELI on January 15, 1993 and March 26, 1993.

The concentrations listed were determined using EPA Method 8080 analyses. Since this Aroclor is not listed in Appendix IX and therefore not requested for this project, it was not reported in the original results package.

The values reported here are only estimated. Concentrations for sample numbers 92-1747 through 92-2795 were calculated using a standard analyzed at a later date. Concentrations for sample numbers 92-3176 through 93-0235 were calculated using a standard analyzed at the time of sample analysis.

All values are reported as wet weight.

A copy of the chromatogram for standard Aroclor 1268 has been supplied.

  
ELAINE A. CARINO  
LABORATORY DIRECTOR



**ELI SAMPLE  
NUMBER**

**PCB - 1268**

**COLLECTION  
DATE**

**EXTRACTION  
DATE**

**ANALYSIS  
DATE**

92-1963	ND < 0.1	6/26/93	7/7/92	7/15/92
92-1984	1 ppm	6/30/93	7/8/92	7/8/92
92-1985	ND < 0.1 ppm	6/30/93	7/8/92	7/8/92
92-2130	ND < 1.0 ppb	7/16/93	7/22/93	7/23/92
92-2677	18.9 ppm	9/9/92	9/15&30/92	11/11&13/92
92-2678	ND < 0.1 ppm	9/9/92	9/15&30/92	11/11/&13/92
92-2707	ND < 0.1 ppm	9/14/92	9/15&30/92	11/11&13/92
92-2708	ND < 0.1 ppm	9/14/92	9/15&30/92	11/11&13/92
92-2777	ND < 0.1 ppm	9/17/92	9/30&10/1/92	11/11&13/92
92-2778	ND < 0.1 ppm	9/17/92	9/30&10/1/92	11/11&13/92
92-2779	69.5 ppm	9/17/92	9/30&10/1/92	11/11&13/92
92-2780	48.6 ppm	9/17/92	9/30&10/1/92	11/11&13/92
92-2795	ND < 1.0 ppb	9/22/92	10/2/92	11/11/92
92-3176	ND < 1.0 ppb	11/5/92	11/17/92	12/7/92
92-3177	ND < 1.0 ppb	11/5/92	11/17/92	12/7/92
92-3178	ND < 1.0 ppb	11/5/92	11/17/92	12/7/92
92-3195	ND < 1.0 ppb	11/9/92	11/16/92	12/7/92
92-3196	ND < 1.0 ppb	11/9/92	11/16/92	12/7/92
92-3228	ND < 1.0 ppb	11/10/92	11/17/92	12/7&12/92
92-3232	ND < 1.0 ppb	11/10/92	11/17/92	12/7&12/92
92-3234	ND < 1.0 ppb	11/10/92	11/17/92	12/7&12/92
92-3242	ND < 1.0 ppb	11/11/92	11/17/92	12/8/92
92-3244	ND < 1.0 ppb	11/11/92	11/17/92	12/8/92
92-3250	ND < 1.0 ppb	11/12/92	11/17/92	12/8/92
92-3252	ND < 1.0 ppb	11/12/92	11/17/92	12/8/92
92-3254	ND < 1.0 ppb	11/12/92	11/17/92	12/8/92
92-3303	3.5 ppb	11/16/92	11/17/92	12/8/92
92-3305	ND < 1.0 ppb	11/16/92	11/17/92	12/8/92
92-3307	ND < 1.0 ppb	11/16/92	11/17/92	12/8/92
92-3325	ND < 1.0 ppb	11/18/92	11/30/92	12/9/92
92-3329	ND < 1.0 ppb	11/18/92	11/30/92	12/9/92
92-3332	ND < 1.0 ppb	11/18/92	11/30/92	12/9/92
92-3335	ND < 1.0 ppb	11/18/92	11/30/92	12/9/92
92-3343	ND < 1.0 ppb	11/19/92	11/30/92	12/9/92
92-3491	ND ppm	12/7/92	12/14/92	12/28&1/13/93
92-3502	4.06 ppm	12/8/92	12/14/92	12/29&1/12/93
92-3504	27.7 ppm	12/8/92	12/14/92	12/29&1/12/93
93-0042	62.8 ppm	1/6/93	1/14/93	1/27&2/1/93
93-0049	ND ppm	1/7/93	1/14/93	1/28/93
93-0051	ND ppm	1/7/93	1/14/93	1/28/93
93-0117	ND ppm	1/14/93	1/21/93	1/28&2/1/93
93-0119	ND ppm	1/14/93	1/21/93	1/28/93
93-0233	ND ppm	1/21/93	1/27/93	1/29&2/1/93
93-0234	ND ppm	1/21/93	1/27/93	1/29&2/1/93
93-0235	ND ppm	1/21/93	1/27/93	1/29&2/1/93

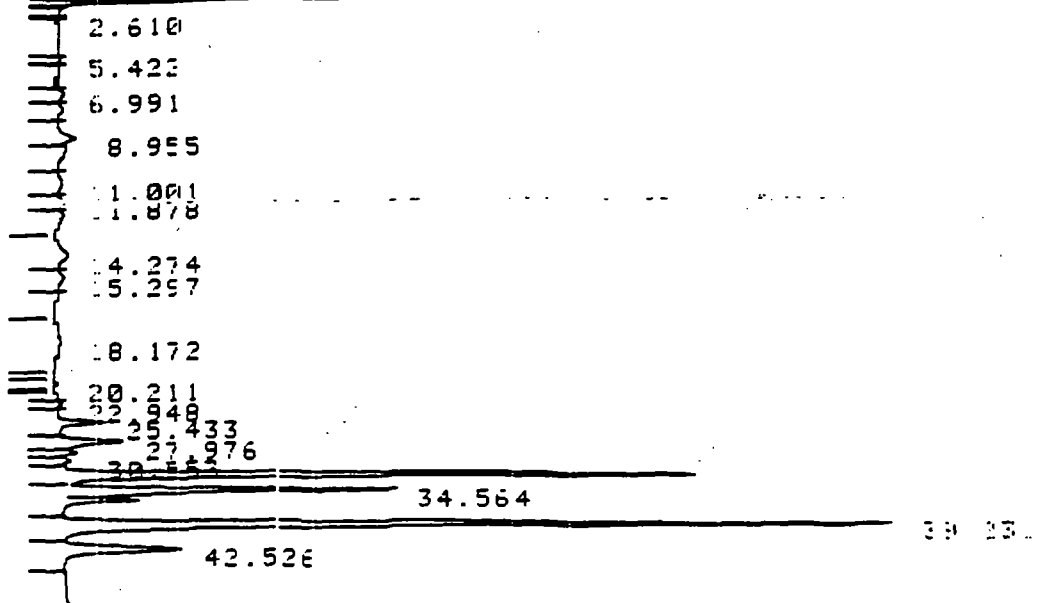
No. 2-2371 (b)  
10 pads

OH?

SUPELCO, INC. Ballston Spa, NY 12020

RT SPEED 0.1 CM/MIN  
IN: 6- ZERO: 15% 5 MIN/TICK  
STAT: INJECT

Avodor 1268 1.0 ppm



TITLE: PCBs & PESTICIDES

17:3- 1 DEC 92

CHANNEL NO: 1

SAMPLE: 1268 1.0 PPM

METHOD: 8080CONF

PEAK NO	PEAK NAME	RESULT PPM	TIME (MIN)	AREA COUNTS	SEP CODE
1		19.2140	0.472	1071410	BV
2		1.8904	X25.433	105415	VV
3		3.0002	X27.976	167633	VV
4		22.9508	X32.514	1280230	VV
5		12.3611	X34.564	689283	VV
6		2.9503	X35.866	164516	VV
7		3.5708	X39.231	1760900	VV
8		6.0403	42.526	336821	VB

RF = 0.22199

TOTALS:

99.9959

5576210

MULTIPLIER: 1.00000

NOTES:

3% SP-2100  
160°-1-2°/MIN TO 210°-4-20°/MIN TO 230°-19  
RANGE 10, 3 UL, FLOW 60

92-3018 20.0 g/5 mL 1/133



**ENVIRONMENTAL LABORATORIES, INC.**

142 TEMPLE STREET  
NEW HAVEN, CT 06510  
(203) 789-1280  
FAX (203) 789-8281

April 13, 1993

Mr. Mike Hill  
U.S. Environmental Protection Agency  
Region I  
90 Canal Street 3<sup>rd</sup> Floor  
Boston, Massachusetts 02203

RE: Raymark Industries, Inc.  
RCRA Section 3013 Order  
Docket 87-1057  
ELI #91-545-10

Dear Mr. Hill:

In accordance with Section 7.0 of the detailed Work Plan for the above referenced site, Environmental Laboratories, Inc. (ELI) is submitting the attached site specific analyte list and sampling methodology for approval. The specific analyte list is based upon the data generated on-site from the quarterly analyses, course reports and from the soil boring and monitoring well installation under this order.

The site specific analyte list is generally similar to the Appendix IX list of chemical parameters for soils. The specific analyses will include: metals (17), asbestos, sulfides, cyanide, PCDD/PCDF, specific volatile organics, chlorinated herbicides, PCB's, organophosphorus pesticides and all the semi-volatiles. No organochlorine pesticides will be analyzed as part of the soil analyte test. The groundwater data detected similar chemical constituents. The specific analyses will include: metals (12), total/dissolved, asbestos (only in the fill), sulfides, cyanides, specific volatile organics, chlorinated herbicides and selective semi-volatile will be analyzed. PCDD/PCDF, organochlorine pesticides, organophosphorus pesticides and PCB will not be analyzed as part of the groundwater analyte test.



Mr. Mike Hill  
U.S. Environmental Protection Agency  
Region I

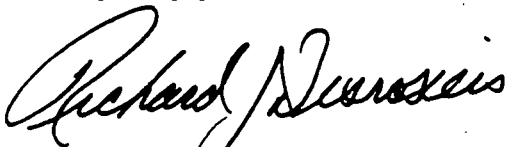
- 2 -

April 13, 1993  
ELI #91-545-10

The attached specific analyte list is being submitted for approval in all subsequent chemical analysis as required under the RCRA Section 3013 Order related to the additional soil and groundwater analysis; the off-site analysis required in Ferry Creek (i.e sediment, surface water and biological) studies. ELI anticipates that Phase IIB will be initiated in mid-May. ELI request approval to the site specific analyte list so we may initiate subcontractor bids and to ensure that the QA/QC protocols are in place to ensure the quality of the data.

If you have any questions regarding the attached list, please do not hesitate to contact us.

Very truly yours,



Richard J. Desrosiers  
Project Manager/Senior Hydrogeologist

RJD/jd  
033RL

Attachment

cc: Mr. Robert F. Moody - Raymark Industries, Inc.  
Mr. Mark Zimmerman, Esq. - Updike, Kelly and Spellacy, P.C.  
Mr. Robert G. Wasp, P.E. - Environmental Laboratories, Inc.

## PHASE IIB SITE SPECIFIC ANALYTE LIST SOLIDS

### METALS (All soils)

Antimony	Beryllium	Cobalt	Mercury	Silver	Vanadium
Arsenic	Cadmium	Copper	Nickel	Thallium	Zinc
Barium	Chromium	Lead	Selenium	Tin	

### ASBESTOS (Fill and Peat only)

Chrysotile	Cellulose	Matrix	Synthetic
------------	-----------	--------	-----------

### SULFIDES (All soils) Method 9030

Sulfides

### CYANIDE (All soils) Method 9012

Cyanide

### DD/PCDF (Fill only) Method 8280

2378-TCDD	2378-TCDF	1234678-HpCDF	Total TCDF
12378-PeCDD	12378-PeCDF	1234789-HpCDF	Total PeCDF
123478-HxCDD	23478-PeCDF	OCDF	Total HxCDF
123678-HxCDD	123478-HxCDF	Total TCDD	Total HpCDF
123789-HxCDD	123678-HxCDF	Total PeCDD	
1234678-HpCDD	234678-HxCDF	Total HxCDD	
OCDD	123789-HxCDF	Total HpCDD	

### VOLATILE ORGANICS (All soils) Method 8240

Acetone	Chlorobenzene	1,2-Dichloroethane	Methyl methacrylate	Trichloroethene
Benzene	Chloroform	1,1-Dichloroethene	4-Methyl-2-pentanone	Tetrachloroethene
Bromodichloromethane	1,2-Dichlorobenzene	Trans-1,2-Dichloroethene	Toluene	Vinyl Chloride
2-Butanone	1,4-Dichlorobenzene	Ethylbenzene	1,1,1-Trichloroethane	Xylene
Carbon disulfide	1,1-Dichloroethane	2-Hexanone	1,1,2-Trichloroethane	

### CHLORINATED HERBICIDES (All soils) Method 8150

2,4-D	Dinoseb	2,4,5-T	2,4,5-TP (Silvex)
-------	---------	---------	-------------------

### NOCHLORINE PESTICIDES AND PCB'S\* Method 8080

CB-1016	PCB-1232	PCB-1248	PCB-1260
PCB-1221	PCB-1242	PCB-1254	PCB-1268

\* Organochlorine pesticides were not detected in Phase IIB. These analytes will not be analyzed in Phase IIB.

# PHASE IIB SITE SPECIFIC ANALYTE LIST SOLIDS (Continued)

## ORGANOPHOSPHOROS PESTICIDES (All soils) Method 8140

Diallate  
Dimethoate  
Disulfoton

Famphur  
Methy Parathion  
Parathion

Phorate  
Pronamide  
Sulfotep

Thioazin

## SEMI-VOLATILE (All soils) Method 8270

Acenaphthene  
Acenaphthylene  
Acetophenone  
2-Acetylaminofluorene  
4-Aminobiphenyl  
Aniline  
Anthracene  
Aramite  
Benzo(a)anthracene  
Benzo(b)fluoranthene  
Benzo(k)fluoranthene  
o(g,h,i)perylene  
benzo(a)pyrene  
Benzyl alcohol  
Bis(2-chloroethoxy)methane  
Bis(2-chloroethyl)ether  
Bis(2-chloroisopropyl)ether  
Bis(2-ethylhexyl)phthalate  
4-Bromophenyl phenyl ether  
Butyl benzyl phthalate  
4-Chloroaniline  
Chlorobenzilate  
4-Chloro-3-methylphenol  
2-Chloronaphthalene  
2-Chlorophenol  
4-Chlorophenyl phenyl ether  
Chrysene  
cis-Diallate  
trans-Diallate  
Dibenz(a,h)anthracene  
Dibenzofuran

Di-n-butylphthalate  
3,3-Dichlorobenzidine  
2,4-Dichlorophenol  
2,6-Dichlorophenol  
Diethyl phthalate  
Dimethoate  
Dimethylaminoazobenzene  
7,12-Dimethylbenz(a)anthracene  
3,3'-Dimethylbenzidine  
alpha-, alpha-  
Dimethylphenethylamine  
2,4-Dimethylphenol  
Dimethylphthalate  
1,3-Dinitrobenzene  
4,6-Dinitro-2-methylphenol  
2,4-Dinitrophenol  
2,4-Dinitrotoluene  
2,6-Dinitrotoluene  
Dinoseb  
Di-n-octylphthalate  
Diphenylamine  
Disulfoton  
Ethyl methanesulfonate  
Famphur  
Fluoranthene  
Fluorene  
Hexachlorobenzene  
Hexachlorobutadiene  
Hexachlorocyclopentadiene  
Hexachloroethane  
Hexachlorophene

Hexachloropropene  
Indeno(1,2,3-cd)pyrene  
Isodrin  
Isophorone  
Isosafrole  
Kepone  
Methapyrilene  
3-Methylcholanthrene  
Methylmethanesulfonate  
2-Methylnaphthalene  
Methyl parathion  
2-Methylphenol (o-cresol)  
3-Methylphenol (m-cresol)  
4-Methylphenol (p-cresol)  
Naphthalene  
1,4-Naphthoquinone  
1-Naphthylamine  
2-Naphthylamine  
2-Nitroaniline  
3-Nitroaniline  
4-Nitroaniline  
Nitrobenzene  
2-Nitrophenol  
4-Nitrophenol  
Nitroquinoline-1-oxide  
N-Nitrosodibutylamine  
N-Nitrosodiethylamine  
N-Nitrosodimethylamine  
N-Nitrosodiphenylamine  
N-Nitrosodi-n-propylamine  
N-Nitrosomethylethylamine

N-Nitrosomorpholine  
N-Nitrosopiperidine  
N-Nitrosopyrrolidine  
5-Nitro-o-toluidine  
Parathion  
Pentachlorobenzene  
Pentachloroethane  
Pentachloronitrobenzene  
Pentachlorophenol  
Phenacetin  
Phenanthrene  
Phenol  
1,4-Phenylenediamine  
Phorate  
2-Picoline  
Pronamide  
Pyrene  
Pyridine  
Safrole  
Sulfotep  
1,2,4,5-Tetrachlorobenzene  
2,3,4,6-Tetrachlorophenol  
Thionazine  
o-Toluidine  
1,2,4-Trichlorobenzene  
2,4,5-Trichlorophenol  
2,4,6-Trichlorophenol  
1,3,5-Trinitrobenzene  
0,0,0-Triethyl phosphorothioate



## PHASE IIB SITE SPECIFIC ANALYTE LIST LIQUIDS

### METALS (Total dissolved)

Arsenic	Cadmium	Cobalt	Lead	Selenium	Zinc
Barium	Chromium	Copper	Nickel	Vanadium	

### ASBESTOS (All liquids)

Chrysotile	Cellulose	Matrix	Synthetic
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### SULFIDES (All liquids) Method 9030

Sulfides

### CYANIDE (All liquids) Method 9012

Cyanide

### D/PCDF (Fill only) Method 8280

Not Detected - Analytes will not be analyzed in Phase IIB

### VOLATILE ORGANICS (All liquids) Method 8240

Benzene	Chloroethane	Trans-1,2-Dichloroethene	Ethylbenzene	1,1,1-Trichloroethane
Bromodichloromethane	Chloroform	1,2-Dichloroethane	Methylene Chloride	Trichloroethene
2-Butanone	1,1-Dichloroethane	1,1-Dichloroethylene	Tetrachloroethene	Vinyl Chloride
Chlorobenzene	1,1-Dichloroethene	Dichloromethane	Toluene	Xylene

### CHLORINATED HERBICIDES (All liquids) Method 8150

2,4-D	Dinoseb	2,4,5-T	2,4,5-TP (Silvex)
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### ORGANOCHLORINE PESTICIDES AND PCB'S\* Method 8080

- No organochlorine pesticides were detected. Organochlorine pesticides will be excluded from Phase IIB analyte list.
- PCB's were not detected in Phase IIA. PCB's known to be present in the soils are typically insoluble in water. PCB's will be excluded from the Phase IIB analyte list.

### ORGANOPHOSPHORUS PESTICIDES Method 8140

Organophosphorus pesticides were detected in Phase IIA. These analytes will be excluded from the Phase IIB analyte list.

**PHASE IIB SITE SPECIFIC ANALYTE LIST  
LIQUIDS**

**SEMI-VOLATILE (All liquids) Method 8270**

Benzyl alcohol  
4,6-Dinitro-2-methylphenol  
2,4-Dinitrophenol

2,4-Dinitrotoluene  
2-Methylphenol (o-cresol)  
4-Methylphenol (p-cresol)

Naphthalene  
2-Nitrophenol  
4-Nitrophenol

N-Nitrosodibutylamine  
Phenol  
P-Phenylenediamine



April 29, 1993

Mr. Richard Desrosiers  
Environmental Laboratories, Inc.  
142 Temple Street  
New Haven, CT 06510

Subject: Review of PCDD/PCDF Analytical Data from Raymark Industries, Inc.  
Site, MRI Project No. 3387A

Dear Mr. Desrosiers:

Midwest Research Institute has reviewed the analytical data and background information pertaining to the Raymark Industries, Inc. We have reviewed the data with respect to assisting Environmental Laboratories, Inc. (ELI) in assessing the origination of PCDD/PCDF residues on site. ELI has provided to MRI a number of items which include historical data and perspective of production efforts at the Raymark Industries site, data tables (hardcopy and Lotus 123 format) for the targeted analytes, and hardcopy results for each of the samples for the PCDDs and PCDFs to allow review of the chromatographic patterns.

In approaching this effort, MRI has reviewed all information supplied by ELI pertaining to the site history, confirmed the calculations for the determination of TEF, assessed the potential for statistical evaluation of the results, reviewed the chromatographic patterns, reviewed available literature, and contacted a number of other scientists with experience in the area of PCDDs, PCDFs, and PCBs.

Our conclusions based on the data received to date reflect that the levels of PCDDs and PCDFs detected in the samples from the Raymark Industries site reflect contribution from a number of sources, which may include the background contribution from general atmospheric transport, residues from Aroclor 1268, and some of the phenoxy herbicides (2,4-D, 2,4,5-T and 2,4,5-TP) detected in the field samples. Data on the patterns of PCDD and PCDF contaminants have been reviewed. Of primary importance in our review of the data is the chromatographic pattern(s) of the homologs of the PCDDs and PCDFs.

## **PCDD/PCDF BACKGROUND LEVELS IN THE STATE OF CONNECTICUT**

MRI has previously worked with the State of Connecticut to determine background levels of PCDDs and PCDFs in soil and sediment from several locations throughout the state to develop a baseline and evaluation of the impact of municipal waste incineration. These data have demonstrated a background of PCDDs and PCDFs in the state. Table 1 presents a summary of data from sites located in Bridgeport, Connecticut. The data base from which this information was extracted contains data on up to 971 samples (77 soils, 344 sediments, and 550 fish) collected from 1987 to 1990 from areas within or adjacent to nine different municipalities. MRI has contacted the Connecticut Department of Environmental Protection to obtain approval for the release of our final report, "Multivariate Statistical Analysis of Dioxin and Furan Levels in Fish, Sediment and Soil Collected near Resource Recovery Facilities" (Final Report for PSA Contract 91-1526).

The data from the general background samples are different than those reported in the ELI sample set. The background contaminant levels for the general environmental conditions are in the 0.002 to 0.356 ng/g levels based on the TEF approach. Also, the patterns from the general environmental samples in Connecticut reflected chromatographic patterns that represented combustion type sources, although statistical analysis of the data base indicated that some of the specific isomers may originate from alternative sources.

## **PCDDS/PCDFS FROM AROCLORS**

As per our past telephone discussions, the Aroclor 1268 may be an important contributor to the levels of PCDFs observed in the sample. The chromatographic patterns observed for the TCDFs, PeCDFs, and HxCDFs reflect some similarities with other Aroclors that have been studied in round robin investigations by the Electric Power Research Institute (EPRI) and in investigations of thermal degradation products from dielectric fluids conducted by MRI. Unfortunately, the samples in both cases focused on Aroclors that were typically used in transformers. Aroclor 1268 was not used for the same purpose as it is a material of higher molecular weight and hence was not suitable as a fluid. At this time we have not been able to identify any research efforts or investigations pertaining to the levels of PCDFs in Aroclor 1268.

Some examples of chromatographic patterns for Aroclor 1254 that has been thermally degraded are presented in the series of Figures presented as attachments to this letter. This data was generated by MRI as an investigation of the thermal degradation of dielectric fluids (Thermal Degradation Products from Dielectric Fluids, M. D. Erickson, et al., EPA-560/5-84-009 December 1984). The chromatographic patterns for the tetra, penta, and hexachlorinated dibenzofurans demonstrate some similarities with the data in the soil samples showing positive results. The other striking factor is the relative difference in the PCDF to PCDD ratios. The PCDFs are greater than the

PCDDs by factors of up to 20 or more in some of the field samples from the Raymark Industries site.

Additional data on PCDFs and PCDDs in Aroclor mixtures were obtained from the EPRI report on PCDFs and PCDDs in Utility Transformers and Capacitors (EPRI EL/EA-4858, December 1986). MRI has spoken with the EPRI project officer on that work, Mr. Gil Addis, and two of the principal investigators from IIT Research Institute and Battelle Columbus Laboratories. The effort on this work focused on Aroclors used within the utility industry. Some of the pertinent data from that effort follows.

**Aroclor 1016.** Aroclor 1016 did not exhibit any PCDDs or PCDFs in the samples analyzed in this study. Approximate detection limits appear to be on the order of 10 ng/g of sample.

**Aroclor 1242.** Aroclor 1242 demonstrated responses to the TCDF, PeCDF, HxCDF, and OCDF. The PCDDs were not detected in this particular matrix. The total PCDF values ranged from approximately 130 ng/g for the OCDF up to 870 ng/g for the TCDFs. The TCDFs were the predominant responses for this mixture.

**Aroclor 1260.** Aroclor 1260 also demonstrated responses to the PCDFs but not the PCDDs. The PCDF homolog concentrations were as follows OCDF (4150 ng/g) > HpCDF (1985 ng/g) > HxCDF (1400 ng/g) > PeCDF (1040 ng/g) > TCDF (600 ng/g).

**Askarel Load Center Network Transformer.** This sample contained 70% Aroclor 1260, 29% trichlorobenzenes, and 1% tetrachlorobenzene from a transformer of over 30 years' service. PCDDs were not detected in the sample to any significant extent. The data demonstrated a different order of predominance of the PCDFs: HxCDF (2300 ng/g) > HpCDF (1590 ng/g) > PeCDF (1530 ng/g) > TCDF (760 ng/g) > OCDF (560 ng/g).

Some effort was made to correlate the Aroclor 1268 concentrations to the total PCDDs and PCDFs using the I-TEF values. However, a graphical representation of the data did not result in any clear or coincident relationship of these values. The correlation of data may be affected by the homogeneity of the matrices analyzed.

Using the information obtained from the EPRI round robin study on various dielectric fluids an estimate of the contaminant levels of PCDFs in the soil samples resulting from the Aroclor 1268 was developed as follows.

If one assumes that the TCDF concentration of 8.1 ng/g in field sample 92-1949 is attributed to the Aroclor 1268 (determined at ~ 30 µg/g of soil), the contaminant level of the TCDF in the Aroclor would originally be 270 µg/g or

$$(8.1 \times 10^{-9} / 30 \times 10^{-6}) = 0.27 \times 10^{-3} \text{ g/g or } 270 \text{ µg/g}$$

This contaminant level is considerably higher than found in any other PCB mixture reported in the EPRI study. Some data on the thermal degradation products of PCBs indicate that conversion efficiencies of up to 2% can be achieved. Hence without information on the residual levels of PCDFs and PCDDs in the Aroclor 1268, it must be considered that some thermal degradation of the product may have occurred under chemical processing conditions or that the contribution is from other sources outside the manufacturing processes and procedures.

### **PCDDS/PCDFS FROM PHENOXY HERBICIDES**

Our review of the data tables indicate some potential association of the PCDD/PCDF levels with the presence of phenoxy herbicides in a number of the samples. However, as with the PCB data, there does not appear to be a strict statistical correlation with any one of the herbicide residues pertaining to the overall TCDD TEA values. Attachment 2 to this letter summarizes available published literature through approximately 1984 ("Guidelines for Determination of Halogenated Dibenzo-p-dioxins and Dibenzofurans in Commercial Products," EPA560/5-87/007, September 1987).

Other sources of information in commercial products, particularly the journal *Chemosphere*, have been reviewed. While additional information has been published regarding PCDD/PCDF levels, the information is consistent with citations from the late 1970s and early 1980s demonstrating the major contaminants are PCDDs. In many products, particularly 2,4,5-T, the primary contaminant is 2,3,7,8-TCDD which was not present in the field samples.

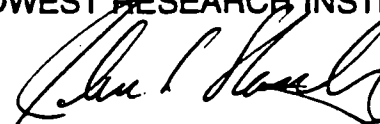
### **STATISTICAL TREATMENT OF DATA**

Our original telephone discussions and proposal indicated that if feasible, MRI would proceed with statistical assessment of data (using multivariate techniques termed cluster analysis and principal component analysis). However, given the extent of the data base and the available timeframe, MRI has not proceeded with the effort. Having reviewed the data base, it would be beneficial to discuss current monitoring efforts and the advantage of adding the most recent data to any such statistical treatment. One area that will need to be addressed is to add detection limit data to the current data tables.

If there are any questions regarding this information, please call. A hardcopy of this letter and contents will be forwarded under separate cover.

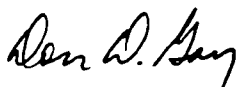
Sincerely,

MIDWEST RESEARCH INSTITUTE



John S. Stanley, Ph.D.  
Section Head  
Analytical Chemistry

Approved:



Don D. Gay, Ph.D.  
Director  
Chemical Sciences Department

**ATTACHMENT 1**

**Background Levels of PCDDs/PCDFs  
in the  
Bridgeport, Connecticut Area**



## BRIDGEPORT SUMMARY (pg/g, dry weight basis)

Analyte	Lab ID	30264	30265	30266	30267	30268	30271	30272	30273
TCDF		238	929	404	29.5	218	59.1	39.5	68
TCDD		49.9	288	115	5.63	38.5	5.57	5.88	11.2
PeCDF		236	1330	470	30.9	405	90.5	58.6	105.4
PeCDD		168	718	151	5.96	79.4	22.4	10.8	20.2
HxCDF		373	1610	370	24.5	365	108	57.8	97.2
HxCDD		430	1670	434	25.9	380	118	84.4	146
HpCDF		552	1990	574	40.6	635	214	160	234
HpCDD		1843	5587	1340	95.6	2039	599	323	476
2378TCDF		47.7	206	65.8	5.08	39.7	15.8	10.8	18.3
2378TCDD		13.9	51.5	6.23	ND( .193 mpc)	ND( 1.24 mpc)	0.762	ND( .412)	ND( .383 mpc)
12378PeCDF	ND( 22.7 mpc)		82.9	22	1.24	13.4	5.16	3.95	6.95
23478PeCDF		20.1	96.9	27.2	1.77	18.3	6.51	4.88	8.42
12378PeCDD		19.6	87.8	13.3	0.78	7.38	2.77	1.4	2.25
123478HxCDF		59.2	277	46.4	3.04	25.2	11.8	5.95	9.77
123678HxCDF		18.4	94.4	18.1	1.26	14.5	5.15	2.56	4.33
234678HxCDF		28.6	134	28.9	2.24	26.7	8.49	4.34	7.71
123789HxCDF	ND( .488)		ND( 9.13 cdl)	ND( 1.72)	ND( .578 mpc)	ND( .647)	ND( .325)	ND( .203)	ND( .964 mpc)
123478HxCDD	ND( 10.3 mpc)		42	11.1	ND( .581 mpc)	10.4	2.25	2.08	2.98
123678HxCDD		41.4	150	35.6	1.99	36.4	8.62	5.99	8.82
123789HxCDD		36.3	146	32.8	2.15	30.6	8.15	5.33	8.5
1234678HpCDF		253	1100	273	18.7	252	93	67.6	101
1234789HpCDF		21	70.5	17	ND( 1.19 mpc)	15.1	3.92	2.36	4.04
1234678HpCDD		773	2330	565	42	831	187	122	192
12346789OCDF		478	1480	665	70.5	772	300	259	380
12346789OCDD		7160	16700	5210	364	6650	1720	1220	1860
I-TE		86.7	358	80.1	5.50	60.6	20.3	13.6	21.9
TOC (mg/kg)		31,900	87,500	43,700	5,120	50,100	34,000	23,000	38,000
Grain Size:									
Sand (%)		45	55	28	82	34	74	60	15
Silt (%)		45	38	58	8	52	21	35	75
Clay(%)		10	9	14	10	14	5	2	10

**BRIDGEPORT SUMMARY (pg/g, dry weight basis)**

Analyte	Lab ID	30274	30275	30276	30277	30278	30279	30280	30281
TCDF		70	64.1	60	56.8	14.6	26.6	60.6	90.6
TCDD		9.12	10.9	1.08	1.61	2.7	2.86	4.86	10.8
PeCDF		150	93.4	103	71.9	15.4	35.3	71.9	110
PeCDD		43.9	37.9	30.9	23.9	4.91	7.83	20.7	34.6
HxCDF		354	78	122	89.6	4.11	22.2	22	52
HxCDD		365	182	140	102	13.4	21.6	36.4	45.6
HpCDF		743	261	155	121	9.24	16.8	24.9	30
HpCDD		2540	757	723	557	39.7	85.5	89.3	126
2378TCDF		22.2	17.2	17.4	14.7	4.2	6.55	20.3	28.4
2378TCDD		2.18	1.66	ND( .483 mpc)	ND( .736)	ND( .631)	ND( .459)	ND( .754)	ND( .434 mpc)
12378PeCDF		12.3	5.87	4.92	4.2	0.98	1.78	4.38	5.41
23478PeCDF		9.02	5.69	10.1	6.07	1.57	2.71	5.05	7.89
12378PeCDD		11.1	5.94	ND( 3.92 mpc)	3.2	ND( .299 mpc)	0.718	1.28	2.01
123478HxCDF		30.7	14.6	11.5	9.77	1.78	3.22	5.58	7.3
123678HxCDF		17.5	7.01	5.62	4	0.78	0.211	1.91	3.55
234678HxCDF		25.6	11.2	9.08	7.35	1.68	3.07	4.55	5.74
123789HxCDF	ND( .592)		ND( .259)	ND( .546)	ND( .355)	ND( .0844 mpc)	0.537	ND( .303)	ND( .51 mpc)
123478HxCDD	12.8		ND( 1.96 mpc)	5.93	ND( 3.23 mpc)	ND( .146 mpc)	1.08	1.22	ND( .727)
123678HxCDD	58.7		21.2	13.6	14.6	ND( .465 mpc)	1.4	2.56	2.16
123789HxCDD	47.3		16.2	13.2	11.5	1.47	2.5	3.57	4.34
1234678HpCDF	318		109	69.6	57.3	4.74	9.73	15.4	17.7
1234789HpCDF	22.2		4.85	5.28	4.29	0.757	0.999	ND( 1.53 mpc)	1.72
1234878HpCDD	1310		319	338	260	17.7	35.1	37.2	53.4
12346789OCDF	603		236	120	98	9.44	10.7	38.7	18.5
12346789OCDD	10600		2580	4530	3560	519	1210	799	1970
I-TE		67.1	27.1	28.5	22.0	4.30	7.42	12.1	17.8
TOC (mg/kg)		130,000	93,000	100,000	88,000	35,000	150,000	220,000	240,000
Grain Size:									
Sand (%)		45	46	28	64	90	99	94	90
Silt (%)		53	53	72	36	8	1	6	10
Clay(%)		2	1	0	0	0	0	0	0

**BRIDGEPORT SUMMARY (pg/g, dry weight basis)**

Analyte	Lab ID	30282	30283	32586	32587	32588	32589	32590	32591
TCDF		2.04	1.58	40.9	272	8.67	37.7	57.1	159
TCDD		ND( .255)	ND( .233)	4.02	46.9	0.516	2.97	3.13	13.8
PeCDF		8.6	6.83	73.5	399	17.2	91.2	173	479
PeCDD		2.11	ND( .338)	27.6	121	6.03	23.5	29.3	55
HxCDF		10.9	7.57	73.7	213	15.6	64.9	140	370
HxCDD		12.9	4.93	59.3	233	23.6	48.8	62.4	181
HpCDF		17.5	13.1	80.2	255	16.9	40	149	198
HpCDD		65.9	44.1	303	909	129	265	286	846
2378TCDF		0.943	ND( .323 mpc)	8.87	57.4	2.21	8.01	11.1	18.2
2378TCDD		ND( .255)	ND( .233)	ND( .298 mpc)	1.74	ND( .43)	ND( .386 mpc)	ND( .343 mpc)	0.955
12378PeCDF		0.493	ND( .118 mpc)	3.55	23.4	0.798	3.22	4.51	6.64
23478PeCDF		0.535	0.324	4.74	25.8	ND( 1.35 mpc)	4.54	6.63	10.5
12378PeCDD		0.366	ND( .0952 mpc)	2	6.65	0.614	1.36	2.07	5.53
123478HxCDF		0.952	ND( .314 mpc)	7.9	ND( 61.3 mpc)	2.76	7.13	11.6	17.5
123678HxCDF		ND( .567 mpc)	0.274	3.9	ND( 19.2 mpc)	1.37	3.19	5.43	13.6
234678HxCDF		0.859	0.679	5.77	30.1	1.92	5.33	9.63	25.8
123789HxCDF		ND( .0677 mpc)	ND( .0597 mpc)	ND( .35)	ND( 1.02)	ND( .18 mpc)	ND( 2.36 cdl)	ND( .565)	ND( 1.68)
123478HxCDD		ND( .223 mpc)	ND( .0191 mpc)	2.04	ND( 1.33)	0.824	1.86	ND( 2.34 mpc)	ND( 2.05 mpc)
123678HxCDD		2.05	1.3	5.8	26.9	1.9	3.17	6.84	23.5
123789HxCDD		1.02	0.744	5.36	20.6	2.22	4.28	ND( 5.92 mpc)	19.1
1234678HpCDF		6.79	5.13	37.9	138	9.11	20.5	71.6	85.8
1234789HpCDF		0.562	ND( .437 mpc)	2.16	14.3	0.761	1.88	4.87	6.62
1234678HpCDD		33.4	23.6	142	377	53.6	100	137	394
12346789OCDF		15.5	12.1	48.9	86.2	13.1	45	110	142
12346789OCDD		448	436	3420	4790	3130	4450	2520	6150
I-TE		2.87	2.04	15.8	60.3	6.84	15.2	18.2	38.2
TOC (mg/kg)		3,600	16,000	21,000	75,000	84,000	64,000	53,000	74,000
Grain Size:									
Sand (%)		90	100	53	- (I)	- (I)	44	58	48
Silt (%)		1	0	41	-	-	50	42	48
Clay (%)		0	0	6	-	-	8	2	4

## BRIDGEPORT SUMMARY (pg/g, dry weight basis)

Analyte	Lab ID	32592	31460		
TCDF		101	151	ND(0.5);	1.58 - 929
TCDD		8.41	29.5	ND(0.5);	0.516 - 288
PeCDF		211	119	ND(0.5);	6.83 - 1330
PeCDD		41.4	166	ND(0.5);	2.11 - 718
HxCDF		143	524	ND(1.25);	4.11 - 1610
HxCDD		86.6	525	ND(1.25);	4.93 - 1670
HpCDF		97.4	745	ND(1.25);	9.24 - 1990
HpCDD		386	3610	ND(1.25);	39.7 - 5587
2378TCDF		25.3	44	ND(0.5);	0.943 - 206
2378TCDD	ND( .878 mpc)		12.8	ND(0.5);	0.762 - 51.5
12378PeCDF	11.1	ND( 25.6 mpc)		ND(0.5);	0.493 - 82.9
23478PeCDF	9.1	7.63		ND(0.5);	0.324 - 96.9
12378PeCDD	3	19.9		ND(0.5);	0.366 - 87.8
123478HxCDF	16.4	72.4		ND(1.25);	0.952 - 277
123678HxCDF	6.95	28.5		ND(1.25);	0.211 - 94.4
234678HxCDF	11.8	41.8		ND(1.25);	0.679 - 134
123789HxCDF	ND( .297)	1.84		ND(1.25);	0.537 - 1.84
123478HxCDD	ND( .969 mpc)	15.5		ND(1.25);	0.824 - 42
123678HxCDD	9.66	58.4		ND(1.25);	1.300 - 150
123789HxCDD	8.16	42.9		ND(1.25);	0.744 - 146
1234678HpCDF	49.7	317		ND(1.25);	4.74 - 1100
1234789HpCDF	3.6	23.5		ND(1.25);	0.562 - 70.5
1234678HpCDD	168	1560		ND(1.25);	17.7 - 2330
12346789OCDF	53.9	638		ND(2.5);	9.440 - 1480
12348789OCDD	3850	> 9700		ND(2.5);	364 - 16700
I-TE		26.1	87.1	0.83 (i);	2.040 - 358
TOC (mg/kg)		71,000	49,500	ND(1,040)	3600 - 240000
Grain Size:					
Sand (%)		71	47	-	15 - 100
Silt (%)		28	43	-	0 - 75
Clay(%)		0	10	-	0 - 14

## **ATTACHMENT 2**

### **Example Chromatograms from Thermally Degraded PCB Mixtures**

**(Taken from EPA 560/5-84-009, December 1984,  
Thermal Degradation Products from Dielectric Fluids)**

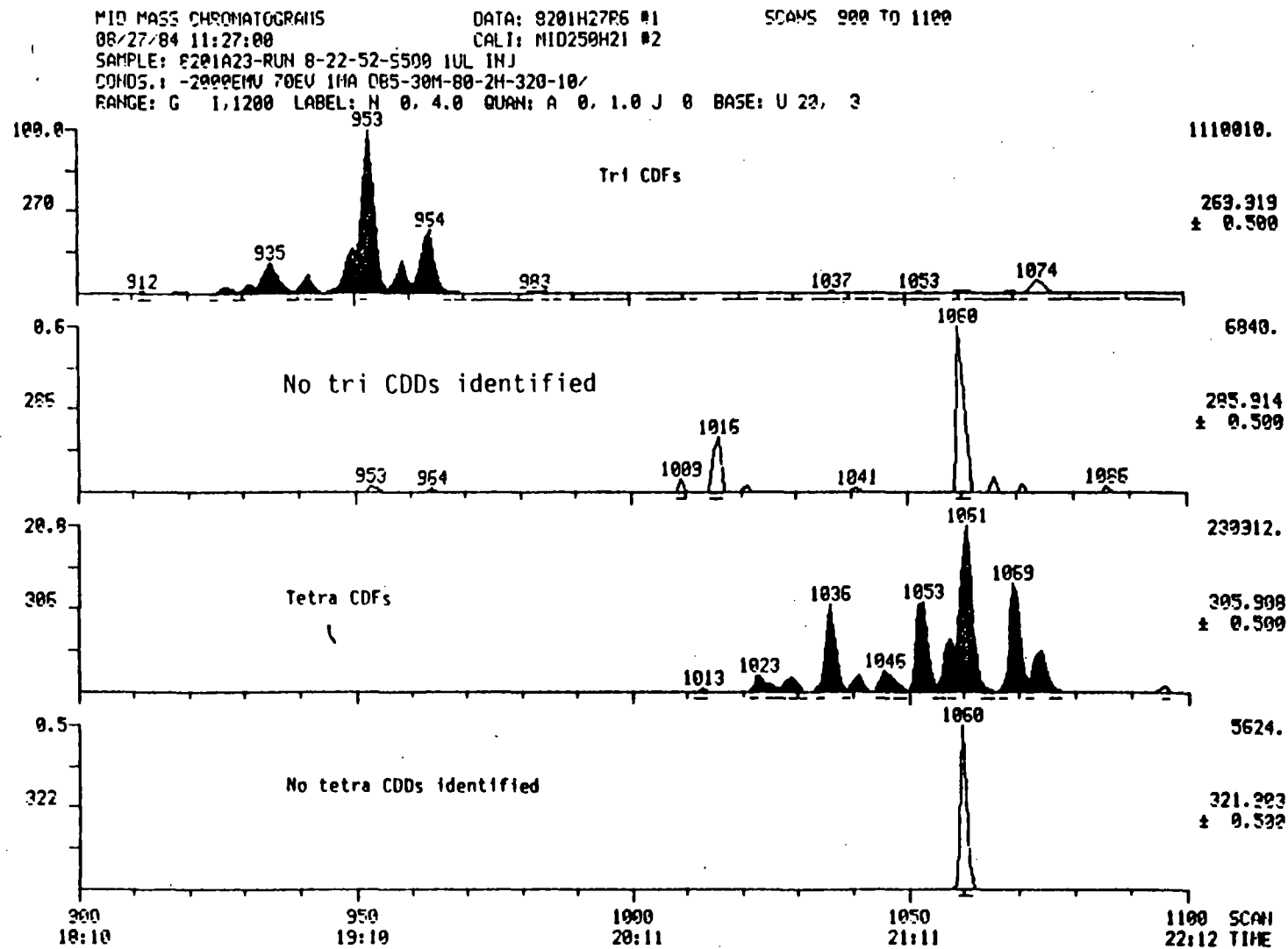


Figure 11. Tri and tetra CDFs and CDDs in sample 8-22-52-S500.

S. Time 0.1 @ 500mg/kg PCB

MID MASS CHROMATOGRAMS DATA: 820112X4 #1 SCANS 1050 TO 1300  
 09/12/84 17:16:00 CALI: MID315112X1 #3  
 SAMPLE: 8-22-52-S500 1UL PCDD/F/BN CL5-5  
 COND.S.: MG=3.5 70EV BC=3 DB5-15M 100-1H-325-10/ 45 SEC SPLTL.  
 RANGE: G 1.1574 LABEL: N 0. 4.0 QUAN: A 0. 1.0 J 0 BASE: U 20. 3

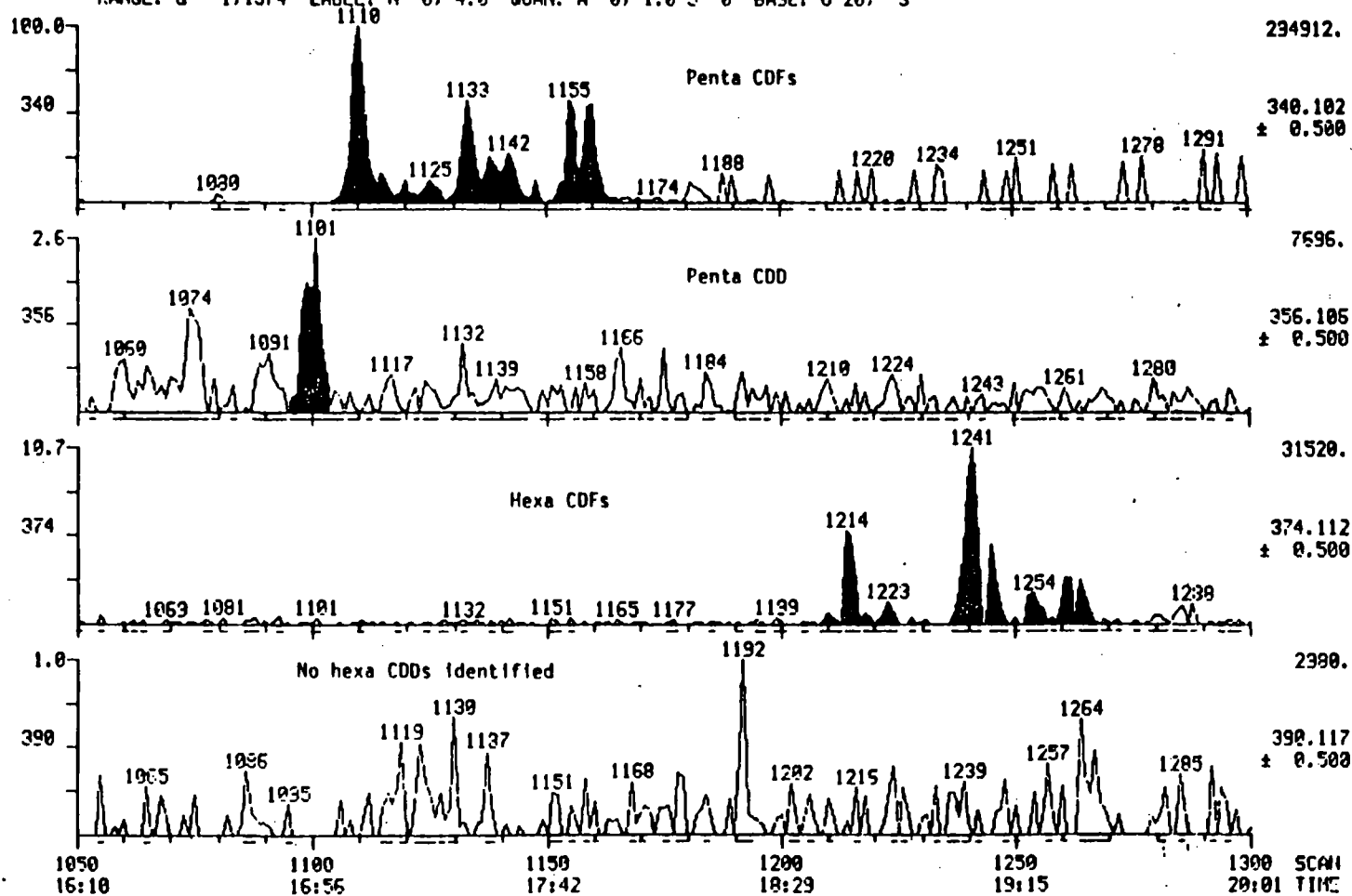


Figure 12. Penta and hexa CDFs and CDDs in sample 8-22-52-S500.

*Silicone oil @ 500 mg/kg PCB*

MID MASS CHROMATOGRAMS DATA: 8201115R5 #1 SCANS 850 TO 1040  
 09/15/84 16:34:00 CALI: MID250H21 #2  
 SAMPLE: 8201A23 RUN 8-30-62 ASKL 1/100 OIL IUL INJ  
 CONDS.: -2200EMV 70EV IMA DB5-3PM 100-1H-320-10/  
 RANGE: G 1.1300 LABEL: H 0.4.0 QUAN: A 0.1.0 J 0 BASE: U 20, 3

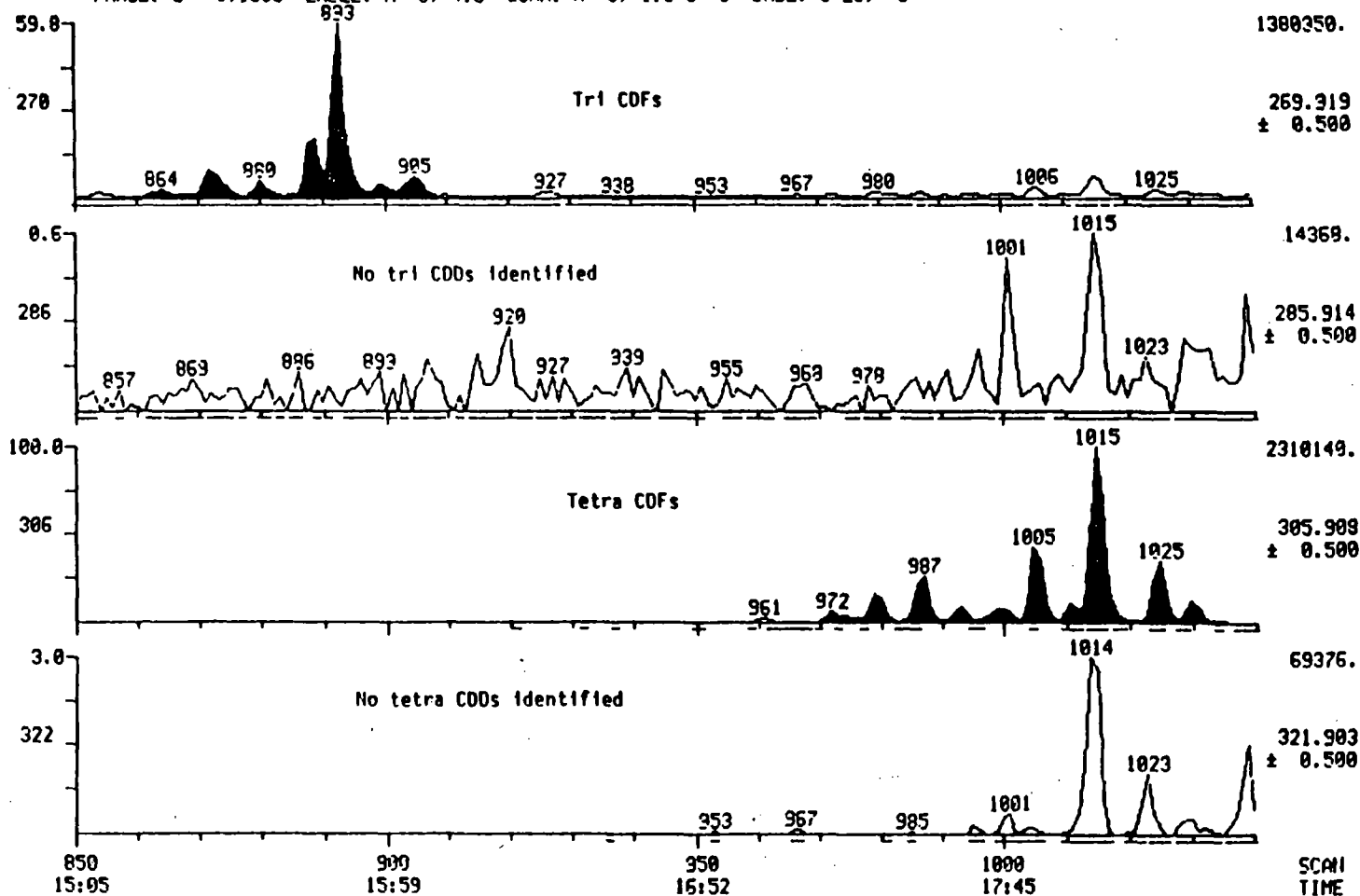


Figure 15. Tri and tetra CDFs and CDDs in sample 8-30-62-ASKL.

*Ascarall sample*



MID MASS CHROMATOGRAMS DATA: 8201115R1 #1 SCANS 1200 TO 1500  
 09/15/84 8:35:00 CALI: MID315114R1 #3  
 SAMPLE: 8-30-62-ASKL 1/100DIL PCDD/F/BPN IUL CL5-6  
 COMDS.: -2200EMU 7PEV 1MA DB5-30M 100-1H-320-10/  
 RANGE: G 1.1600 LABEL: N 0. 4.0 QUAN: A 0. 1.0 J 0 BASE: U 20. 3

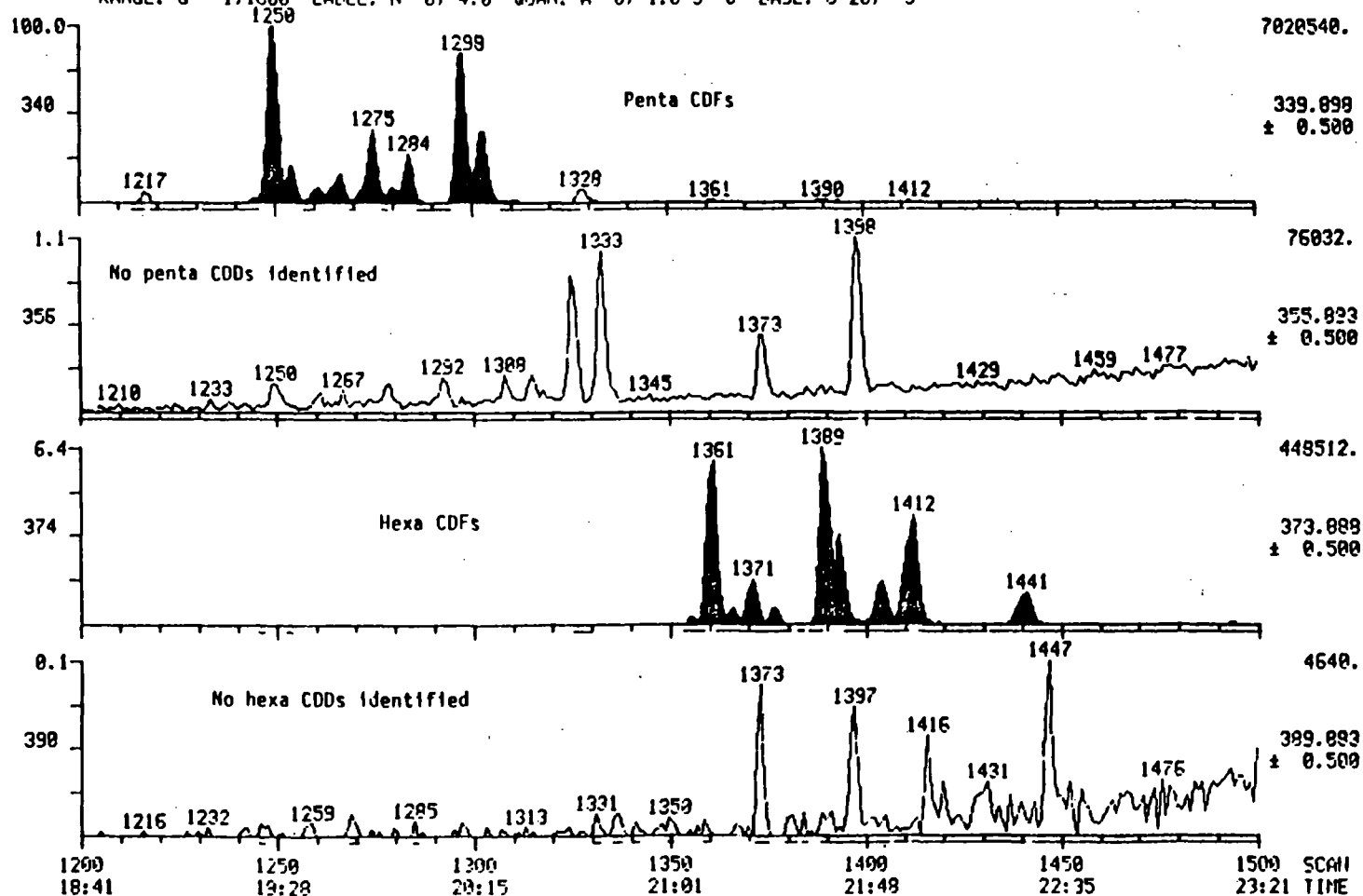


Figure 16. Penta and hexa CDDs and CDFs in sample 8-30-62-ASKL.

*Askerel Sample*

MID MASS CHROMATOGRAMS DATA: 8201111R12 #1 SCANS 580 TO 850  
 03/11/84 16:50:00 CALI: MID390111 #2  
 SAMPLE: 8-30-62-ASKL (D/F) IUL PCDD/F/BPN CL7-8  
 COND.: -2000EMU 7REV 1MA DB5-30M-200-2H-320-10/  
 RANGE: G 1, 900 LABEL: H 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

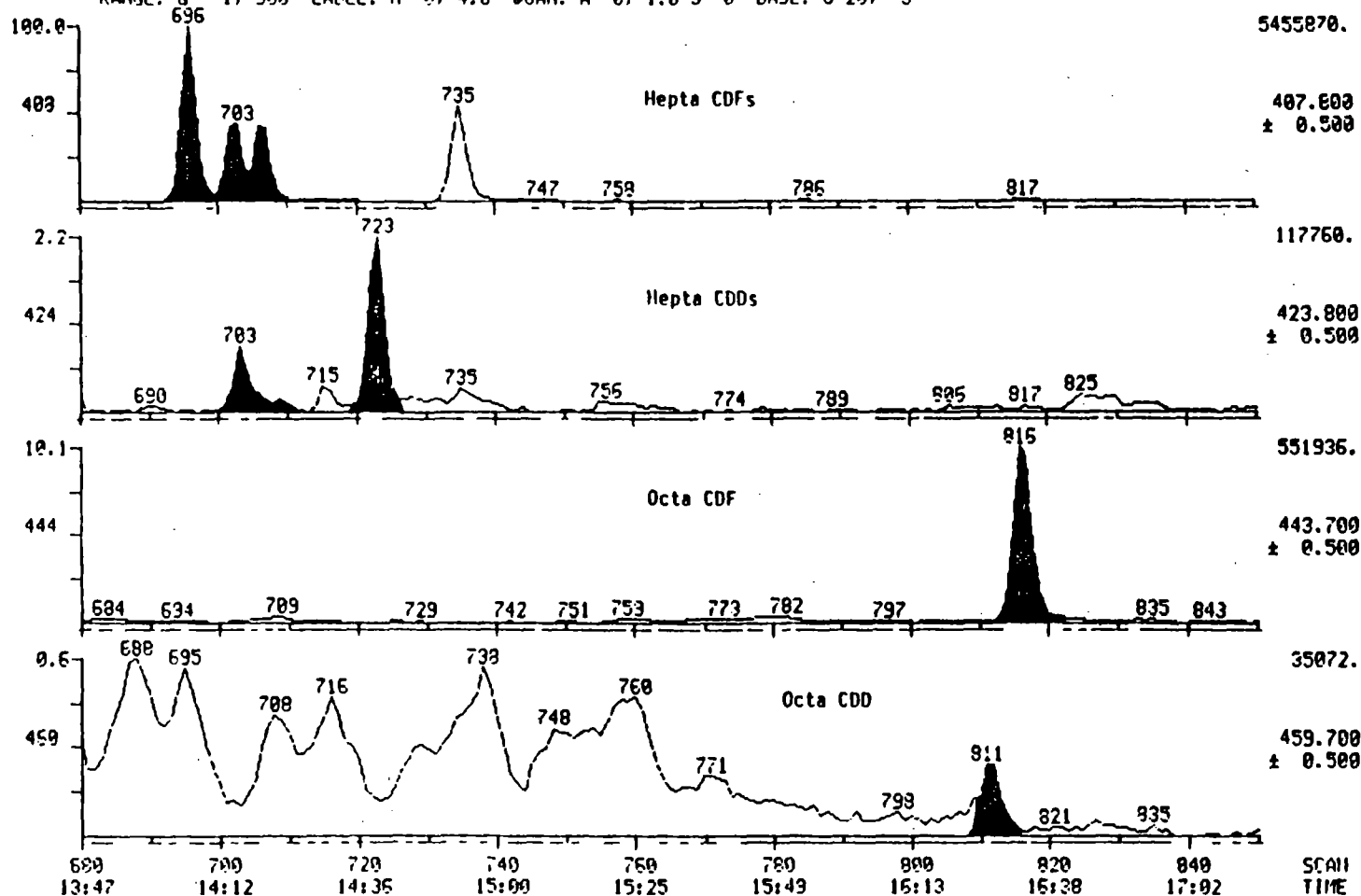


Figure 17. Hepta and octa CDFs and CDDs in sample 8-30-62-ASKL.

*Askael Sample*

MID MASS CHROMATOGRAMS

03/11/84 11:22:00

SAMPLE: 8201A23 RUN 8-15-43-M5 10L INJ

CONDS.: MG=3.5 70EV BC=3 DB5-15M 100-1H-325-10/

RANGE: G 1.1200 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

DATA: 8201111X5 #1  
CALI: MID250110X1 #3

SCANS 780 TO 1020

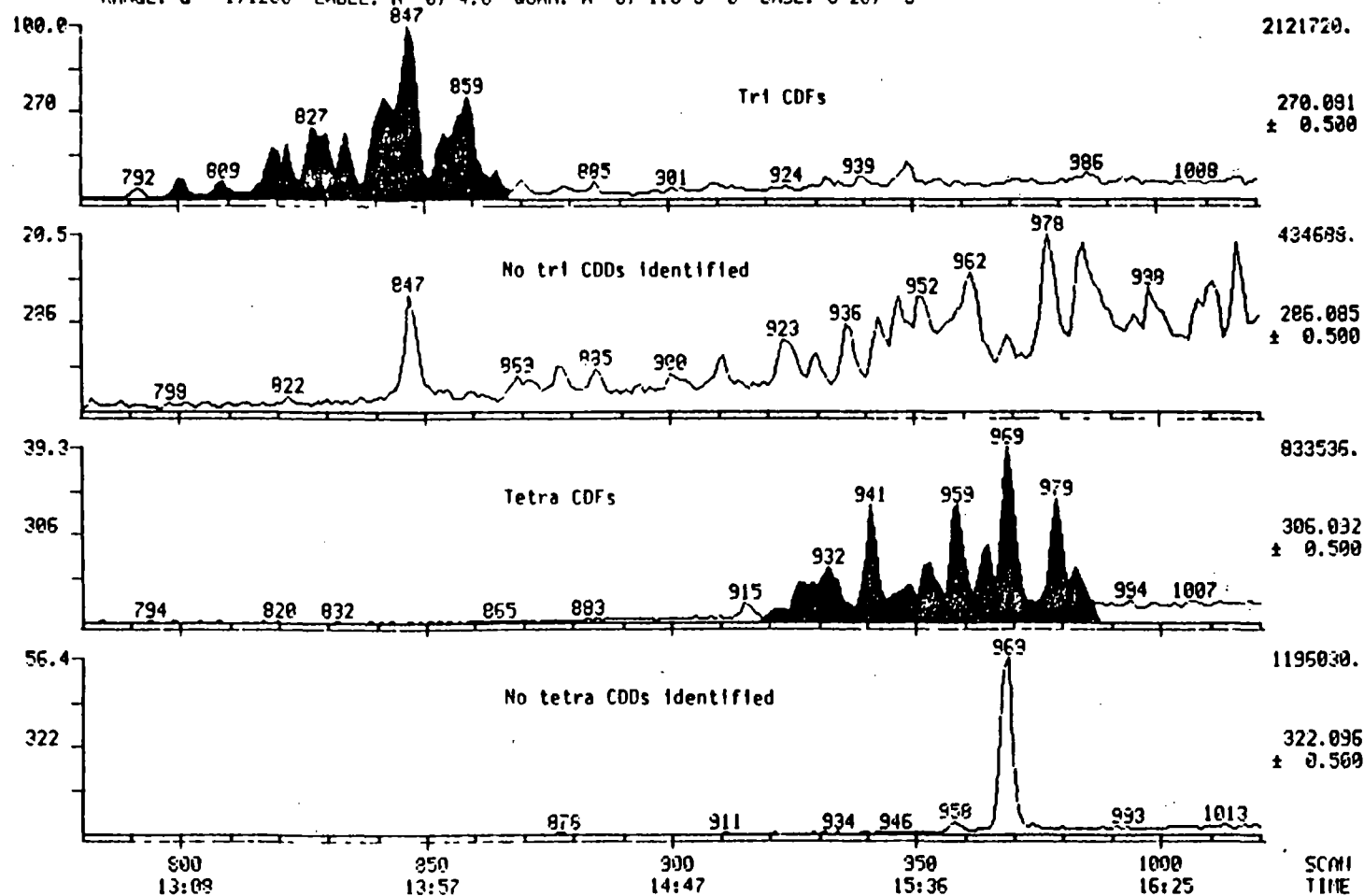


Figure 18. Tri and tetra CDFs and CDDs in sample 8-15-43-M5.  
*mineral oil at 5ug/1g PCB*

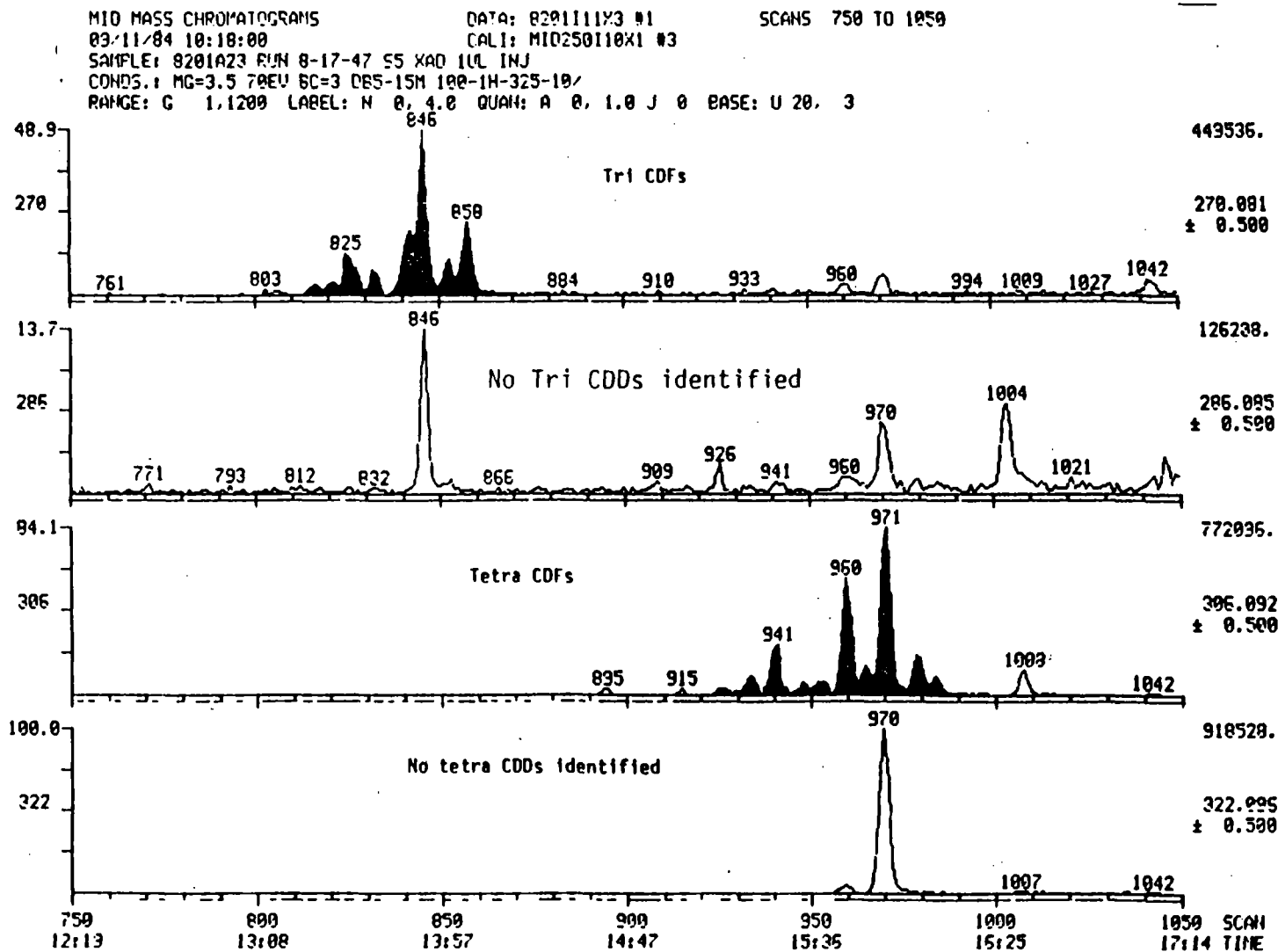


Figure 19. Tri and tetra CDFs and CDDs in sample 8-17-47-S5.

*Silicon oil at Sup/7 RB*

MID MASS CHROMATOGRAMS

DATA: 820110X2 #1

SCANS 800 TO 1050

03/10/84 15:45:00

CALI: MID250110X1 #3

SAMPLE: 8201A23 RUN 8-28-57 1UL INJ

CONDS.: MG=3.5 70EV BC=3 DBS-15M 100-1H-325-10/

RANGE: G 1.1250 LABEL: N 0. 4.0 QUAN: A 0. 1.0 J 0 BASE: U 20. 3

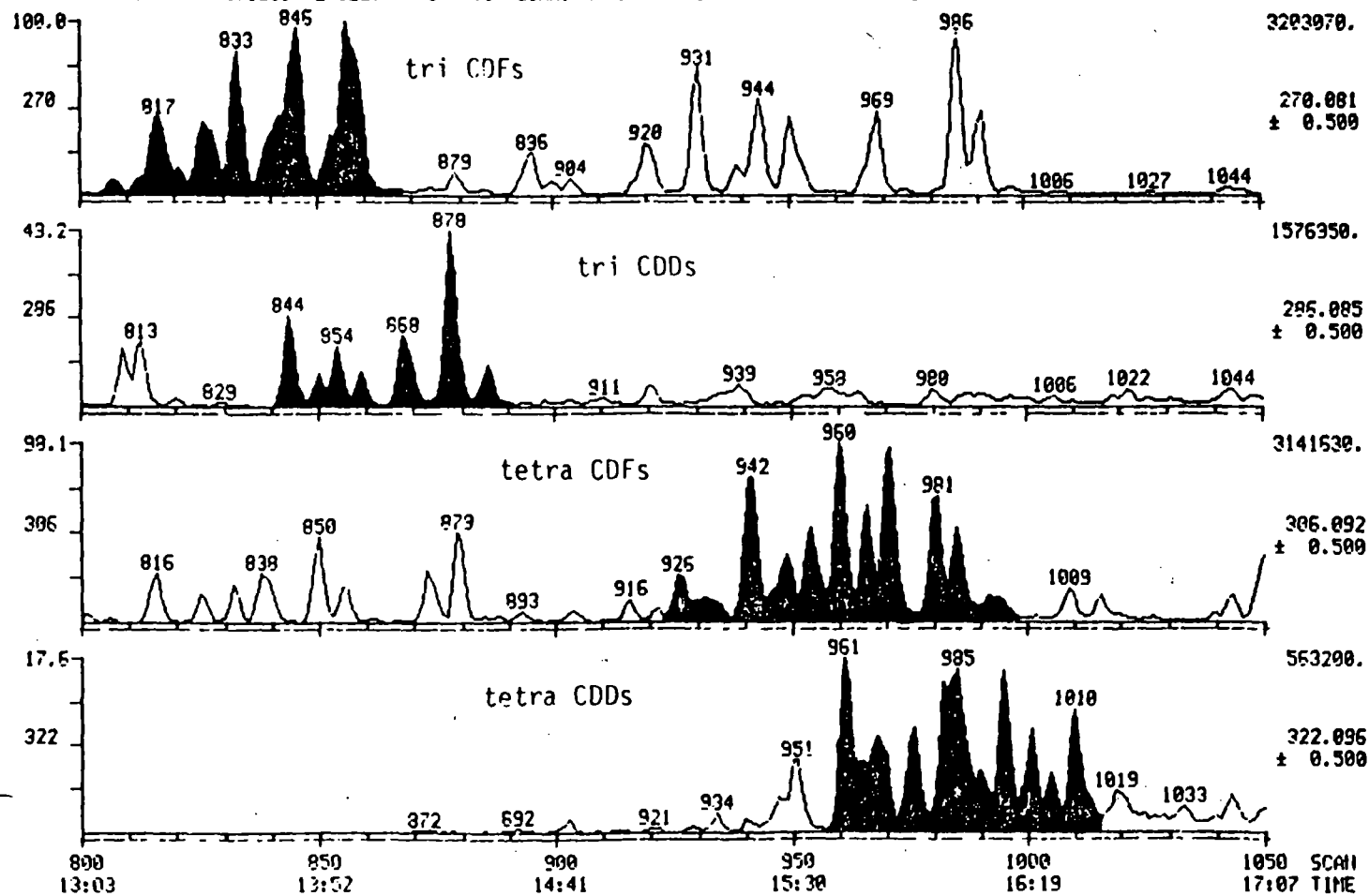


Figure 20. Tri and tetra CDFs and CDDs in sample 8-28-57-CLBZ.

*Chlorobenzene Sample.*

**ATTACHMENT 3**

**PCDD/PCDF Residue Levels  
in  
Phenoxy Herbicides**

Table 1. A Summary of the Reported Analytical Limits of Detection for Polychlorodibenzo-p-dioxins (PCDDs) and Polychlorodibenzofurans (PCDFs) in Phenoxyalkanoic Acid Herbicides<sup>a</sup>

Reference	Applicability	Compounds detected	Limit of detection (mg kg <sup>-1</sup> of sample)
Storherr et al.	2,4,5-T	TCDD	0.05
Brenner et al. (1972)	2,4-D, dichlorprop, mecoprop and 2,4,5-T	TCDD	0.01
Vogel (1976)	2,4,5-T	TCDD	0.03
Ramstad et al. (1977)	Butoxypropyl esters of 2,4,5-T and of fenoprop	TCDD	0.005
Elvidge (1971)	2,4,5-T, ethylhexyl ester of 2,4,5-T and formulations containing the ethylhexyl ester of 2,4,5-T	TCDD	0.05 (on 2,4,5-T content)
Woolson et al. (1972)	2,4-D, 2,4-DB, dichlorprop, 2,4,5-T, fenoprop and dicamba	Di-, tri-, tetra-, penta-, hexa-, hepta, and octa-CDDs	0.5 (for any one of the compounds detected)
Edmunds et al. (1973)	2,4,5-T, the butyl and octyl esters of 2,4,5-T and formulations of the butyl ester of 2,4,5-T (50% in mineral oil)	TCDD	0.05
Crummett and Stahl (1973)	Esters of 2,4,5-T and of fenoprop, Herbicide Orange	TCDD	0.05
Buser et al. (1974)	2,4,5-T, formulations of esters of 2,4,5-T and of amine salts of 2,4,5-T	TCDD	0.001 (on a standard solution)
Huckins et al. (1978)	Herbicide Orange	TCDD and penta-CDD	0.02
Rappe et al. (1978)	Formulations of esters of 2,4,5-T, Herbicide Orange	Di-, tri-, tetra-, penta- and hexa-CDDs, di-, tri-, tetra-, penta- and hexa-CDFs	0.01-0.05
Polzhofer (1979)	2,4,5-T, alkyl esters of 2,4,5-T and formulations	TCDD	0.005 (on 2,4,5-T content)
Carello et al.	Fenoprop	TCDD	0.01

<sup>a</sup>Baker PG, Hoodless RH, Tyler JC. 1981. Pestic Sci 12:297-304.

DRAFT

Raymark Industries, Inc.  
 RCRA Section 3013 Order  
 Docket No. 87-1057  
 ELI #91-545-10

AROCLOL 1268 VS. PCDD/PCDF TEF PHASE II A DATA TABLE			
LAB ID	FIELD ID	PCB - 1268	TEF
92-1747	18 00.10	190 ppm *	7.22
92-1748	18 24.34	ND < 0.1 ppm	0.0113
92-1749	18 14.20	BDL < 0.1 ppm	ND
92-1753	27 02.12	120 ppm *	0.956
92-1755	27 34.38	ND < 0.1 ppm	ND
92-1759	28 02.115	40 ppm *	0.339
92-1760	28 27.37	ND < 0.1 ppm	0.00013
92-1761	28 40.50	ND < 0.1 ppm	ND
92-1768	17 02.12	4 ppm *	0.0732
92-1769	17 15.24	0.2 ppm *	0.0003
92-1775	31 0.10	50 ppm *	0.585
92-1776	31 14.24	0.6 ppm *	0.0004
92-1797	35 00.10	190 ppm *	1.681
92-1798	35 34.42	ND < 0.1 ppm	0.00037
92-1802	46 00.08	ND < 0.1 ppm	0.00237
92-1803	46 22.32	ND < 0.1 ppm	0.00001
92-1855	66 00.08	30 ppm *	0.121
92-1856	66 12.22	ND < 0.1 ppm	0.00091
92-1857	66 26.36	ND < 0.1 ppm	ND
92-1861	03 07.15	1 ppm *	0.00035
92-1862	03 15.185	3 ppm *	0.0005
92-1863	03 31.43	ND < 0.1 ppm	0.00013



<b>AROCOR 1268 VS. PCDD/PCDF TEF</b> <b>PHASE II A DATA TABLE</b>			
<b>LAB ID</b>	<b>FIELD ID</b>	<b>PCB - 1268</b>	<b>TEF</b>
92-1864	03 55.63	ND < 0.1 ppm	0.0002
92-1870	EQBLK66	ND < 1.0 ppb	ND
92-1871	EQBLK03	ND < 1.0 ppb	ND
92-1897	51 00.10	BDL < 1.0 ppm	0.00442
92-1898	51 10.18	BDL < 1.0 ppm	0.1228
92-1899	51 36.46	ND < 0.1 ppm	ND
92-1900	51 46.545	ND < 0.1 ppm	ND
92-1911	11 00.06	90 ppm *	5.99
92-1912	11 06.11	150 ppm *	1.62
92-1913	11 40.45	ND < 0.1 ppm	0.00014
92-1914	11 32.38	ND < 0.1 ppm	0.00016
92-1949	02 00.08	30 ppm *	4.4
92-1950	02 08.11	30 ppm *	0.94
92-1951	02 38.48	ND < 1.0 ppm	ND
92-1952	02 92.102	ND < 1.0 ppm	0.00042
92-1953	EQBLK51	ND < 1.0 ppb	ND
92-1954	EQBLK02	ND < 1.0 ppb	ND
92-1955	62 00.10	ND < 1.0 ppm	ND
92-1956	62 12.22	2 ppm *	0.00097
92-1957	62 28.36	ND < 1.0 ppm	0.00037
92-1962	22 09.15	BDL < 0.1 ppm	0.00019
92-1963	22 41.49	ND < 0.1 ppm	0.00049
92-1984	37 05.11	1 ppm *	ND
92-1985	37 19.27	ND < 0.1 ppm	0.00008
92-2130	Hydrant	ND < 1.0 ppb	ND

<b>AROCLOR 1268 VS. PCDD/PCDF TEF PHASE II A DATA TABLE</b>			
<b>LAB ID</b>	<b>FIELD ID</b>	<b>PCB - 1268</b>	<b>TEF</b>
92-2677	F4 05.02	18.9 ppm *	0.431
92-2678	F4 56.64	ND < 0.1 ppm	ND
92-2707	A4 00.04	ND < 0.1 ppm	0.000073
92-2708	A4 22.27	ND < 0.1 ppm	ND
92-2777	E5 52.62	ND < 0.1 ppm	ND
92-2778	E5 38.44	ND < 0.1 ppm	ND
92-2779	E5 10.16	69.5 ppm *	0.442
92-2780	E5 02.08	48.6 ppm *	1.093
92-2795	EQBLKE5	ND < 1.0 ppb	<del>2.175</del> 0.002
92-3176	GWE1-1	ND < 1.0 ppb	ND
92-3177	GWE4-1	ND < 1.0 ppb	ND
92-3178	FdBLK	ND < 1.0 ppb	
92-3195	GWE3	ND < 1.0 ppb	ND
92-3196	FdBLK	ND < 1.0 ppb	
92-3228	GWE5	ND < 1.0 ppb	ND
92-3232	GWE2	ND < 1.0 ppb	ND
92-3234	FdBLK	ND < 1.0 ppb	
92-3242	GWA3	ND < 1.0 ppb	ND
92-3244	FdBLK	ND < 1.0 ppb	
92-3250	GWA5	ND < 1.0 ppb	ND
92-3252	GWA2	ND < 1.0 ppb	ND
92-3254	FdBLK	ND < 1.0 ppb	
92-3303	GWFI-1	3.5 ppb	ND
92-3305	GWE6	ND < 1.0 ppb	ND
92-3307		ND < 1.0 ppb	

<b>AROCLOR 1268 VS. PCDD/PCDF TEF PHASE II A DATA TABLE</b>			
<b>LAB ID</b>	<b>FIELD ID</b>	<b>PCB - 1268</b>	<b>TEF</b>
92-3325	GWF2	ND < 1.0 ppb	ND
92-3329	GWF3	ND < 1.0 ppb	ND
92-3332	GWF4	ND < 1.0 ppb	ND
92-3335	FdBLK	ND < 1.0 ppb	
92-3343	EQBLK	ND < 1.0 ppb	
92-3491	BD1/DP22 #8	ND ppm	0.065493
92-3502	BD7/DP43 #5	4.06 ppm	ND
92-3504	BD29/DP3/9 #4a	27.7 ppm	0.002634
93-0042	BD16/DP21 #4b	62.8 ppm	ND
93-0049	DB1/DP2 #2	ND ppm	0.000651
93-0051	BD1/DP22 #3a	ND ppm	ND
93-0117	BD6/DP6 #1	ND ppm	0.000628
93-0119	BD6/DP2 #7	ND ppm	ND
93-0233	BD1/DP22 #3b	ND ppm	ND
93-0234	BD4/DP42 #6	ND ppm	ND
93-0235	BD5a/DP5 #9	ND ppm	ND

\* Approximate concentration

RJD/jd  
036RM

## Test Analysis 8280

Boring No.  
Sample No.  
FIELD I.D.

2  
92-1949  
0200.08  
Soil

2  
92-1950  
0208.11  
Soil

Chemical Name	TOXICITY EQUIVALENCE FACTOR	Conc. (ppb)	2378-TCDD EQUI. CONCENTRATION	Conc. (ppb)	2378-TCDD EQUI. CONCENTRATION
2378-TCDD	1	ND	ND	ND	ND
12378-PeCDD	0.5	ND	ND	ND	ND
123478-HxCDD	0.1	ND	ND	ND	ND
123678-HxCDD	0.1	0.16	0.016	ND	ND
123789-HxCDD	0.1	0.18	0.018	ND	ND
1234678-HpCDD	0.01	2.30	0.023	0.29	0.029
OCDD	0.001	4.60	0.0046	0.96	0.00096
2378-TCDF	0.1	2.40	0.24	0.25	0.025
12378-PeCDF	0.05	1.50	0.075	0.25	0.0125
23478-PeCDF	0.5	5.10	2.55	1.10	0.55
123478-HxCDF	0.1	7.40	0.74	1.70	0.17
123678-HxCDF	0.1	1.50	0.15	0.27	0.027
234678-HxCDF	0.1	4.50	0.45	1.10	0.11
123789-HxCDF	0.1	ND	ND	ND	ND
1234678-HxCDF	0.01	13.10	0.131	4.20	0.042
1234789-HpCDF	0.01	ND	ND	ND	ND
OCDF	0.001	0.73	0.00073	0.23	0.00023
2378-TCDD EQUIVALENT CONCENTRATION			4.4		0.97

\*All Other Isomers' TEF = 0

2  
92-1951  
0238.48  
Soil

2  
92-1952  
0292.102  
soil

2  
92-1954  
002  
Water

3  
92-1861  
0307.15  
Soil

Conc.  
(ppb) 2378-TCDD  
EQUI.  
CONCEN-  
TRATION

Conc.  
(ppb) 2378-TCDD  
EQUI.  
CONCEN-  
TRATION

Conc.  
(ppb) 2378-TCDD  
EQUI.  
CONCEN-  
TRATION

Conc.  
(ppb) 2378-TCDD  
EQUI.  
CONCEN-  
TRATION

ND  
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0.42 0.00042

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0.35 0.00035

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ND

0.00035

3  
92-1862  
0315.18.5  
Soil

3  
92-1863  
0331.43  
Soil

3  
92-1864  
0355.63  
Soil

3  
92-1871  
003  
Water

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

ND  
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0.50 0.0005

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0.20 0.0002

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ND

0.0005

0.00013

0.0002

ND

11  
92-1911  
1100.06  
Soil

11  
92-1912  
1106.11  
Soil

11  
92-1913  
1140.46  
Soil

11  
92-1914  
1132.38  
Soil

Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION	Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION	Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION	Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION
ND		ND		ND		ND	
ND		ND		ND		ND	
ND		ND		ND		ND	
ND		ND		ND		ND	
ND		ND		ND		ND	
1.70	0.017	0.55	0.0055	ND		ND	
3.50	0.0035	1.10	0.0011	0.14	0.0014	0.16	0.0016
3.20	0.32	0.36	0.036	ND		ND	
1.60	0.08	0.43	0.0215	ND		ND	
7.40	3.7	1.30	0.065	ND		ND	
9.60	0.96	4.10	0.41	ND		ND	
1.60	0.16	0.78	0.078	ND		ND	
6.00	0.6	2.50	0.25	ND		ND	
ND	ND	ND	ND	ND		ND	
14.80	0.148	16.20	0.162	ND		ND	
ND	ND	ND	ND	ND		ND	
0.62	0.00062	0.92	0.00092	ND		ND	
	5.99		1.03		0.0014		0.0016

17  
92-1768  
1702.12  
Soil

17  
92-1769  
1715.24  
Soil

18  
92-1747  
1800.10  
Soil

18  
92-1748  
1824.34  
Soil

Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION	Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION	Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION	Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION
ND		ND		ND		ND	
ND		ND		ND		ND	
ND		ND		ND		ND	
ND		ND		ND		ND	
ND		ND		ND		ND	
ND		ND		1.30	0.013	ND	
0.10	0.001	ND		4.10	0.0041	ND	
0.01	0.001	ND		3.40	0.34	ND	
ND		ND		2.40	0.12	ND	
ND		ND		7.80	3.9	0.02	0.01
0.36	0.036	ND		14.40	1.44	ND	
0.04	0.004	ND		1.90	0.19	ND	
0.18	0.018	ND		8.30	0.83	ND	
ND	ND	ND		ND	ND	ND	
1.40	0.14	0.03	0.0003	37.60	0.376	0.13	
ND	ND	ND		0.19	0.0019	ND	
0.05	0.00005	ND		1.20	0.0012	ND	
	0.2		0.0003		7.22		0.01



18  
92-1749  
1814.20  
Soil

22  
92-1962  
2209.15  
Soil

22  
92-1963  
2241.49  
Soil

27  
92-1753  
2702.12  
Soil

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

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0.09 0.00009

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0.49

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0.83 0.0083  
2.20 0.0022

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0.01 0.0001  
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0.41 0.041  
0.27 0.0135  
1.20 0.6  
1.50 0.15  
0.22 0.022  
0.82 0.082  
ND ND  
3.60 0.036  
ND ND  
1.10 0.0011

ND

0.00019

0.956

27  
92-1755  
2734.38  
Soil

28  
92-1759  
2802.11.5  
Soil

28  
92-1760  
2827.37  
Soil

28  
92-1761  
2840.50  
Soil

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

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0.37 0.0037  
0.79 0.00079

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0.13 0.00013

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ND  
0.09 0.0045  
0.38 0.19  
0.65 0.065  
0.10 0.01  
0.41 0.041  
ND  
2.40 0.024  
ND  
0.14 0.00014

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ND

0.339

0.00013

ND

31  
92-1775  
3100.10  
Soil

31  
92-1776  
3114.24  
Soil

35  
92-1797  
3500.10  
Soil

35  
92-1798  
3534.42  
Soil

Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION	Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION	Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION	Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION
ND		ND		ND		ND	
ND		ND		0.11	0.055	ND	
ND		ND		ND		ND	
ND		ND		ND		ND	
ND		ND		ND		ND	
0.20	0.002	ND		ND		ND	
3.90	0.0039	ND		0.32	0.00032	0.07	0.00007
0.16	0.016	ND		0.26	0.026	ND	
0.12	0.006	ND		0.25	0.0125	ND	
0.47	0.235	ND		1.00	0.5	ND	
1.40	0.14	ND		5.10	0.51	ND	
0.22	0.022	ND		0.85	0.085	ND	
0.87	0.087	ND		3.30	0.33	ND	
ND	ND	ND		ND		ND	
7.20	0.072	0.04	0.0004	16.10	0.161	0.03	0.0003
ND	ND	ND		ND		ND	
0.61	0.00061	ND		1.20	0.0012	ND	
	0.585		0.0004		1.681		0.00037

37  
92-1984  
1005.11  
Soil

37  
92-1985  
1019.27  
Soil

46  
92-1802  
4600.08  
Soil

46  
92-1803  
4622.32  
Soil

Conc.  
(ppb) 2378-TCDD  
EQUI.  
CONCEN-  
TRATION

Conc.  
(ppb) 2378-TCDD  
EQUI.  
CONCEN-  
TRATION

Conc.  
(ppb) 2378-TCDD  
EQUI.  
CONCEN-  
TRATION

Conc.  
(ppb) 2378-TCDD  
EQUI.  
CONCEN-  
TRATION

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0.08 0.00008

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2.30 0.0023

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0.01 0.00001

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0.007 0.00007  
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ND

0.00001

51  
92-1897  
5100.10  
Soil

51  
92-1898  
5110.18  
Soil

51  
92-1899  
5136.46  
Soil

51  
92-1900  
5146.54.5  
Soil

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

ND  
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0.72 0.00072

ND  
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0.12 0.012  
ND  
5.50 0.055  
30.60 0.0306

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0.03 0.003  
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0.07 0.0007  
ND  
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0.07 0.007  
ND  
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ND  
1.30 0.013  
0.04 0.0004  
4.80 0.0048

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0.00442

0.1228

ND

ND

51  
92-1953  
051  
Water

62  
92-1955  
6200.10  
Soil

62  
92-1956  
6212.22  
Soil

62  
92-1957  
6228.36  
Soil

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

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0.97 0.00097

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0.37 0.00037

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0.00097

0.00037

66  
92-1855  
6600.08  
Soil

66  
92-1856  
6612.22  
Soil

66  
92-1857  
6626.36  
Soil

66  
92-1870  
066  
Water

Conc.  
(ppb) 2378-TCDD  
EQUI.  
CONCEN-  
TRATION

Conc.  
(ppb) 2378-TCDD  
EQUI.  
CONCEN-  
TRATION

Conc.  
(ppb) 2378-TCDD  
EQUI.  
CONCEN-  
TRATION

Conc.  
(ppb) 2378-TCDD  
EQUI.  
CONCEN-  
TRATION

ND  
ND  
ND  
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ND  
0.30 0.0003  
  
0.04 0.004  
ND  
ND  
0.62 0.062  
0.07 0.007  
0.32 0.032  
ND  
1.60 0.016  
ND  
0.19 0.00019

ND  
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0.91 0.00091  
  
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0.121

0.00091

ND

ND

# WELL SOIL

Hydrant  
92-2130

A4  
92-2707  
A40004  
W. SOIL

A4  
92-2708  
A42227  
W. SOIL

E5  
92-2777  
E55262  
W. SOIL

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

Conc. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

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0.073 0.000073

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ND

0.000073

ND

ND



E5  
92-2795  
W Soil

Cons. 2378-TCDD  
(ppb) EQUI.  
CONCEN-  
TRATION

ND	
ND	
ND	
ND	
ND	
1.400	0.014
11.000	0.011

[illegible]

1.093

$$\frac{2.175}{\cancel{100}} \approx 0.0022$$

F4  
92-2677  
F40502  
W. SOIL

F4  
92-2678  
F45664  
W. SOIL

Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION	Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION
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ND		ND	
ND		ND	
ND		ND	
ND		ND	
ND		ND	
0.059	0.00059	ND	
0.660	0.00066	ND	
0.120	0.012	ND	
0.067	0.00335	ND	
0.400	0.2	ND	
0.990	0.099	ND	
0.140	0.014	ND	
0.720	0.072	ND	
ND		ND	
2.900	0.029	ND	
ND		ND	
0.310	0.00031	ND	

0.431

ND

93-0117/18

93-0049/50

93-0051/52

93-0233

Solid

Solid

Solid

Solid

Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION	Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION	Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION	Conc. (ppb)	2378-TCDD EQUI. CONCEN- TRATION
ND	ND	ND	ND	ND	ND	ND	ND
ND	ND	ND	ND	ND	ND	ND	ND
ND	ND	ND	ND	ND	ND	ND	ND
ND	ND	ND	ND	ND	ND	ND	ND
ND	ND	ND	ND	ND	ND	ND	ND
ND	ND	ND	ND	ND	ND	ND	ND
ND	ND	0.651	0.000651	ND	ND	ND	ND
ND	ND	ND	ND	ND	ND	ND	ND
ND	ND	ND	ND	ND	ND	ND	ND
ND	ND	ND	ND	ND	ND	ND	ND
ND	ND	ND	ND	ND	ND	ND	ND
ND	ND	ND	ND	ND	ND	ND	ND
ND	ND	ND	ND	ND	ND	ND	ND
ND	ND	ND	ND	ND	ND	ND	ND
ND	ND	ND	ND	ND	ND	ND	ND
ND	ND	ND	ND	ND	ND	ND	ND
0.628	0.000628	ND	ND	ND	ND	ND	ND
	0.000628		0.000651		ND		ND

3/17/93

92-3504

93-0042/43

92-3502

93-0234

Solid

Solid

Solid

Solid

Conc.  
(ppb)2378-TCDD  
EQUI.  
CONCEN-  
TRATIONConc.  
(ppb)2378-TCDD  
EQUI.  
CONCEN-  
TRATIONConc.  
(ppb)2378-TCDD  
EQUI.  
CONCEN-  
TRATIONConc.  
(ppb)2378-TCDD  
EQUI.  
CONCEN-  
TRATIONND  
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**93-0235**

## Liquid

**Solid**

**Solid**

Conc.  
(ppb)

2378-TCDD  
EQUI.  
CONCEN-  
TRATION

**Conc.  
(ppb)**

2378-TCDD  
EQUI.  
CONCEN-  
TRATION

**Conc.  
(ppb)**

2378-TCDD  
EQUI.  
CONCEN-  
TRATION

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