ORGANIC CHEMICALS DIVISION RESEARCH Report

70-4
70-5
70-6
70-7
70-8
70-11
70-12
70-13
70-17
70-18
70-21

REPORT NO. - 3688 JOB NO. - 2-02-760.01-89013

FINAL REPORT
ON
ANALYTICAL CHEMISTRY INVESTIGATIONS - 1970
SPECIAL STUDIES

DATE - January 1971 WRITTEN BY - E. S. Tucker, et al.

Monsanto

ST. LOUIS, MO.

MONS 038127

AD-442

Analytical Chemistry
Special Study 70-4
Job No. 1630346

STUDY OF AROCLOR CHEMICAL CLEAN-UP PROCEDURES FOR USE WITH ELECTRON CAPTURE GAS CHROMATOGRAPHY METHODS

INTRODUCTION

The effects of three chemical clean-up procedures on the Aroclor 1200 series have been investigated. The chemical procedures in question: nitration, saponification, and treatment with sulfuric acid - were originally proposed by pesticide analysts for the clean-up and identification of pesticides in residue extracts. Later when polychlorinated biphenyls (PCB's) were identified as environmental contaminants these procedures were extended to the clean-up and identification of PCB's in residue extracts.

SUMMARY

1. The quantitative recovery and EC response of Aroclor materials are not affected by saponification or treatment with sulfuric acid.

2. Nitration can have a significant deleterious effect on the quantitative recovery and EC response of Aroclor 1242.

3. The extent of the effect of the nitration treatment is dependent on the temperature and exposure time employed as well as the specific Aroclor treated.

4. Analysts employing nitration procedures prior to EC analysis may or may not, depending on the conditions used, see an EC response for Aroclor 1242 if present.

DISCUSSION

We have found that saponification and treatment with sulfuric acid does not affect the quantitative recovery nor the electron capture (EC) response of the Aroclor materials. However, the nitration procedures have significant deleterious effects on both the quantitative recovery and EC response of the lower chlorinated biphenyls such as Aroclor 1242. The effect of this treatment on the Aroclor series is shown graphically in Figures 1-7.

The fact that the lower chlorinated biphenyls are nitrated under these conditions has been verified by GC/Mass identification of the

products as nitrochlorobiphenyls.

The nitration procedure employed was referenced by Risebrough* [Erro, F., Bevenue, A., and Beckman, H., Bull. Environ. Cont. Toxicol., 372 (1967)] in his Nature article on PCB residues in the environment.

Additionally, we have investigated a similar nitration procedure used by Widmark and co-workers (private communication via R. E. Keller). This treatment differs from Risebrough's in that a shorter exposure time and a lower temperature are employed. The Widmark method greatly dimishes the effects observed with Risebrough's referenced method.

RESULTS

Verification of the conclusion drawn are shown in Figures 1-7. Extraction and measurement were performed as described in Analytical Method No. 69-13.

* A quote from Risebrough's article Nature, 220, 1098 (1968), "The DDT compounds are destroyed by nitration and p,p'-DDT, DDD, and toxaphene are dehydrochlorinated by saponification with alcoholic KOH. PCB is not degraded by either procedure."

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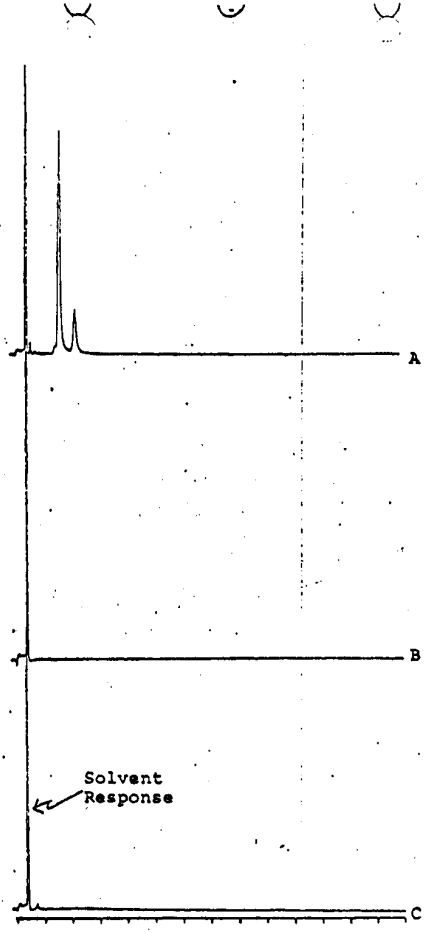

FIGURE 1. Electron capture gas chromatographic trace of (A) biphenyl treated with 1:1 mixture of nitric and sulfuric acids and extracted into hexane, (B) biphenyl treated with distilled water and extracted into hexane, and (C) untreated biphenyl standard in hexane.

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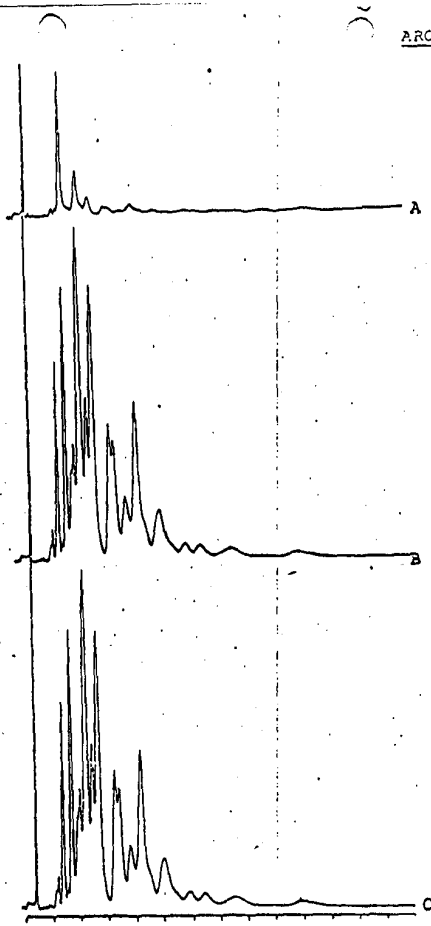

FIGURE 2. Electron capture gas chromatographic trace of (A) Aroclor 1242 and treated with 1:1 mixture of nitric and sulfuric acids and extracted into hexane, (B) Aroclor 1242 treated with distilled water and extracted into hexane, and (C) untreated Aroclor 1242 standard in hexane.

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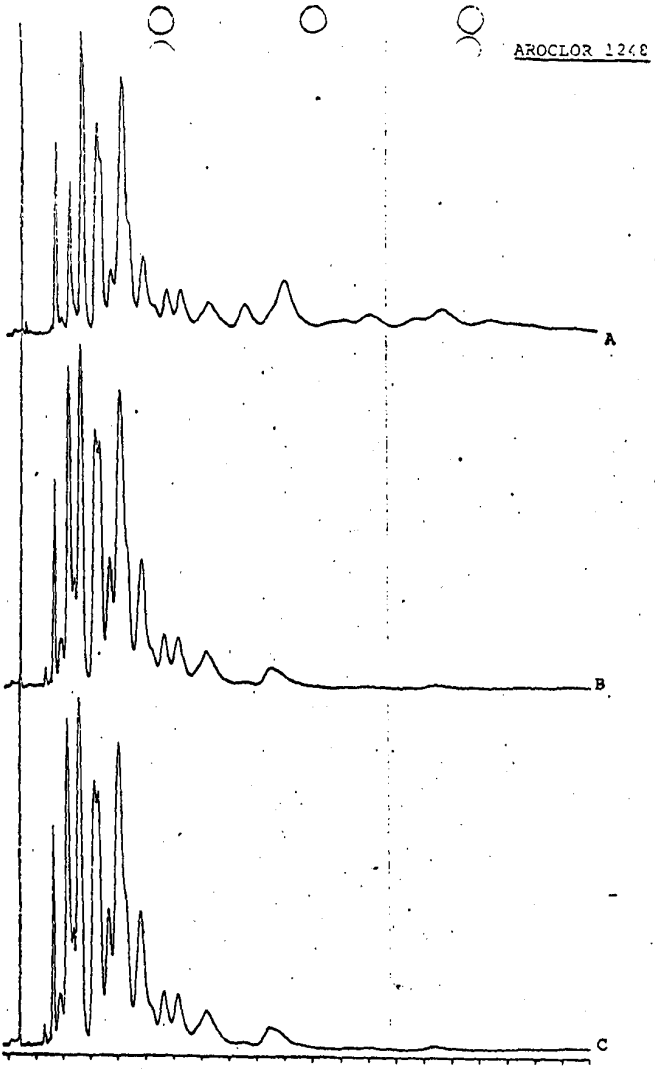

FIGURE 3. Electron capture gas chromatographic trace of (A) Aroclor 1248 treated with 1:1 mixture of nitric and sulfuric acids and extracted into hexane, (B) Aroclor 1248 treated with distilled water and extracted into hexane, and (C) untreated Aroclor 1248 standard in hexane.

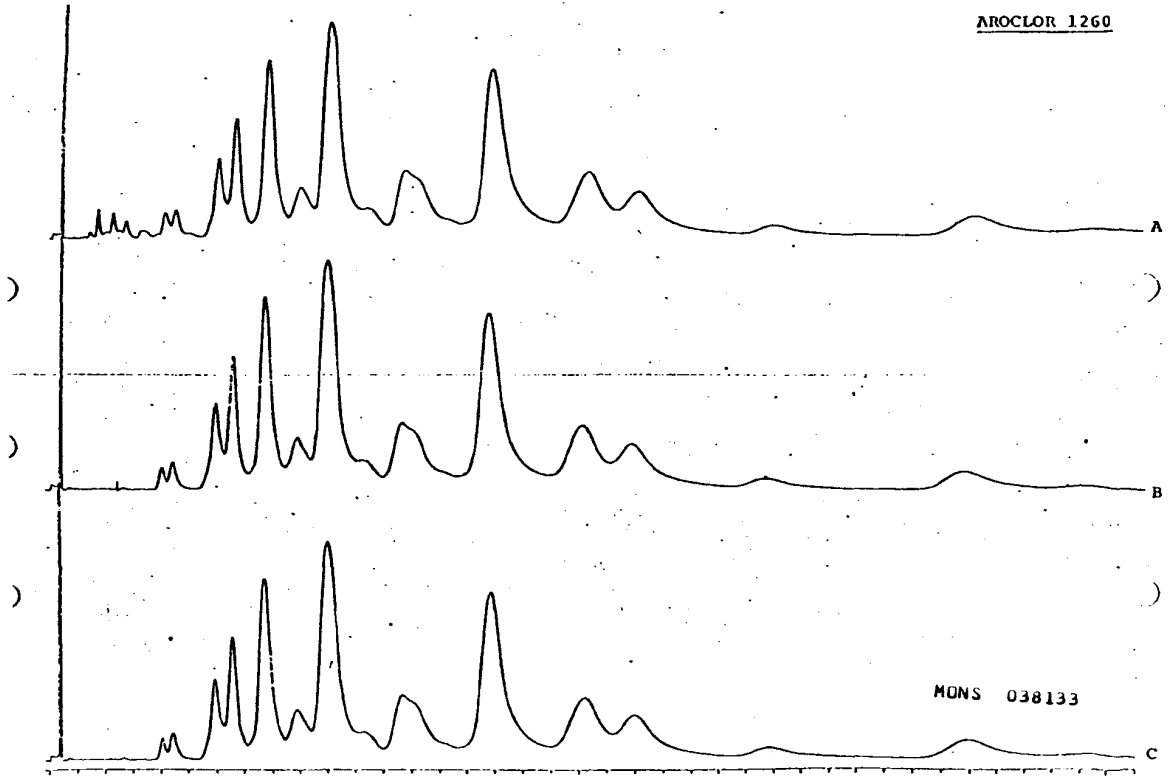

FIGURE 5. Electron capture gas chromatographic trace of (A) Aroclor 1260 and treated with 1:1 mixture of nitric and sulfuric acids and extracted into hexane, (B) Aroclor 1260 treated with distilled water and extracted into hexane, and (C) untreated

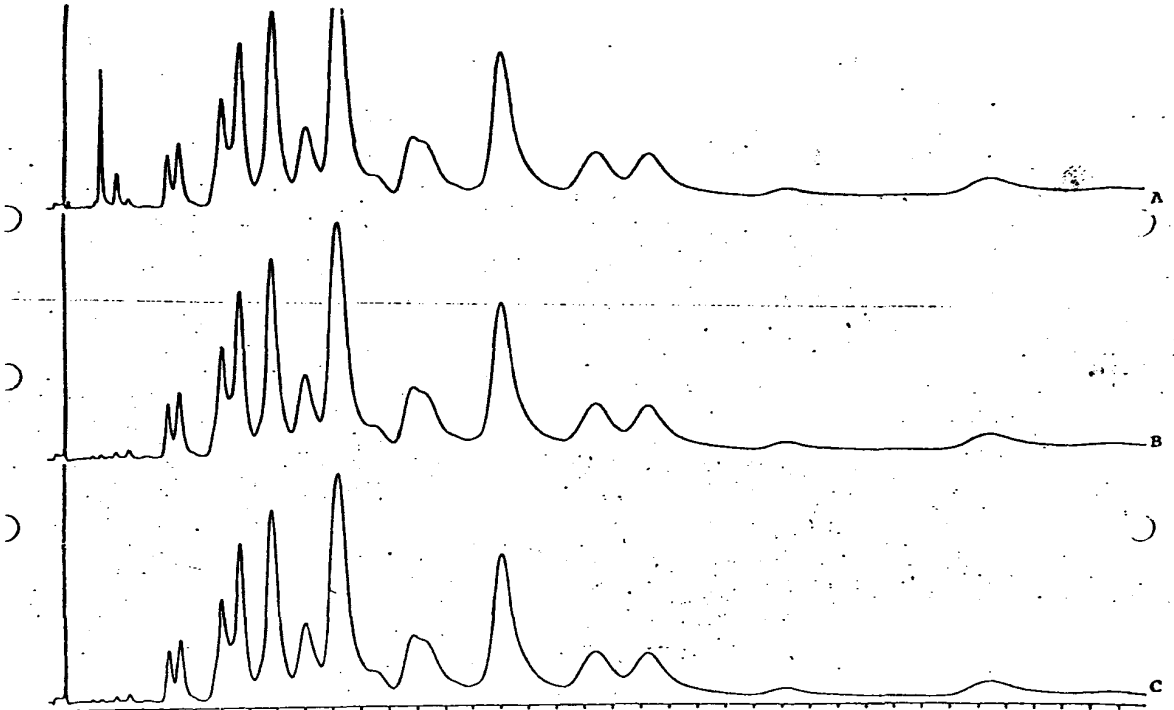

FIGURE 6. Electron capture gas chromatographic trace of (A) Aroclor 1262 and treated with 1:1 mixture of nitric and sulfuric acids and extracted into hexane, (B) Aroclor 1262 treated with distilled water and extracted into hexane, and (C) untreated Aroclor 1262 standard in hexane.

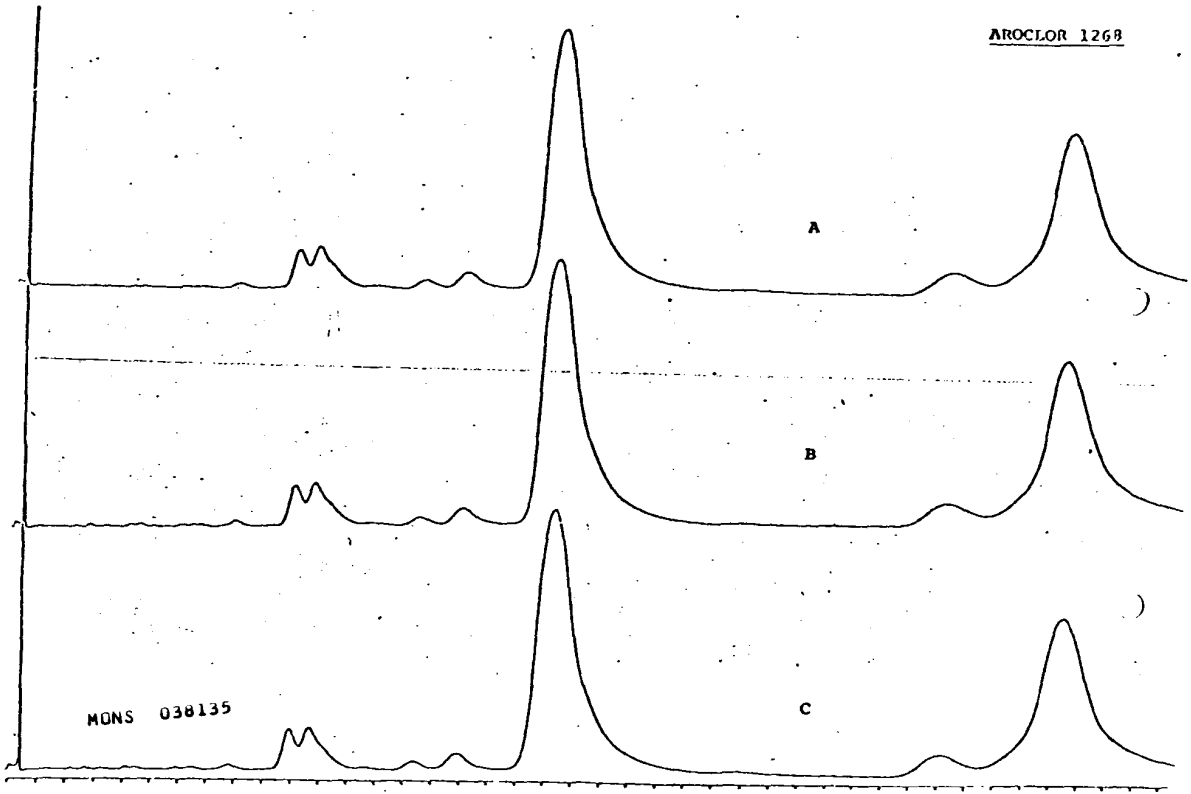

FIGURE 7. Electron capture gas chromatographic trace of (A) Aroclor 1268 and treated with 1:1 mixture of nitric and sulfuric acids and extracted into hexane, (B) Aroclor 1268 treated with distilled water and [illegible]

Analytical Chemistry
Special Study 70-5
Job No. 1630346

SOLUBILITY OF AROCLOR 1242, 1248 AND 1254 IN WATER

INTRODUCTION

An analytical procedure (Analytical Method No. 69-13) for extracting, concentrating and measuring Aroclor at the sub ppm level in water has been developed and verified with Aroclor "spiked" tap water samples (Table I).

SUMMARY

This procedure was employed to determine the tap water solubility of Aroclor 1242, 1248 and 1254 (Table II). The solubility was found to range from 50 ppb for Aroclor 1254 to a maximum of 375 ppb for Aroclor 1242 depending on the conditions used.

DISCUSSION

The Aroclor saturated tap water samples used in this study were prepared in the following manner: two 60-70g portions of each Aroclor fluid were placed in 32 oz. narrow neck screw top glass bottles and 500 ml of tap water (South Second Street) added to each. The bottles were then placed on a roller mill, rolled for 24 hours and allowed to stand undisturbed at room temperature (25°C) until analyzed. The period of time between preparation and analysis was approximately 4-5 months.

Inspection of EG/GC traces and the data in Table II indicates that the solubility of Aroclor is a function of both the amount of chlorination and the position. This anticipated behavior makes absolute quantification difficult since a standard with the same isomer distribution of the water solubilized material is impossible to prepare. Therefore, the "Aroclor found" was calculated in this manner: the original Aroclor in question was used as a standard and a calibration curve prepared by plotting the peak height of the major isomers versus ul Aroclor. The peak height response of the samples were then totaled, corrected for a tap water blank response and the amount of "Aroclor found" determined from the calibration curve. A rough idea of the error inherent in this procedure can be obtained by using 1242 as a standard for 1248, etc. A deviation on the order of ± 10% was experienced.

The data in Table II also seems to indicate that a stable Aroclor-water emulsion is formed at approximately twice their solubility levels.

RESULTS

The results are shown in Table II.

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TABLE I

RECOVERY OF AROCLOR FROM SPIKED TAP WATER SAMPLES**
USING EC/GC

Sample Number	ppb Aroclor Added	Sample Volume (ml)	ppb Aroclor Found	Per Cent Recovery	Comments
R 88932 #1	1980	250	1960	99.0	Aroclor 1242. Samples con-
R 88932 #2	1980	250	-----*	----	centrated wi vac. rot. ev Results ob-
					tained 9-6-6
R 88945 #1	270	225	266	98.5	Aroclor 1254. Samples con-
R 88945 #2	270	225	266	98.5	centrated c Kundera-Dani evaporative
R 88945 #3	540	225	577	107.0	concentrator Results ob-
R 88945 #4	540	225	577	107.0	tained 12-13-

The sample preparation and work up outlined for water samples was employed. The results were quantified by preparing a calibration curve using a hexane solution of the Aroclor 1242 and 1254 used to spike the water samples. The major isomer peak area was used to construct the curve. The water samples were spiked by injecting µl amounts of a concentrated acetone***-Aroclor solution.

* Sample contaminated - results discarded.

** Data used to confirm proposed water sample work up procedure and concentration techniques.

*** Spiked water samples contained 40-60 ppm Acetone.

TABLE II

SOLUBILITY OF AROCLOR IN TAP WATER

Sample Ident. Number	Grams Aroclor Equilibrated	Sample Vol. (ml)	ppb Aroclor Found	Average
1242				
OR 88936-1+	67.5	225	193	
-2+	67.5	225	213	203 ± 10
-1*	55.1	250	372	
-2*	55.1	250	378	375 ± 3
1248				
OR 88936-1+	70.5	250	92	
-2+	70.5	250	120	106 ± 14
-1*	70.6	224	196	
-2*	70.6	225	222	209 ± 13
1254				
OR 88936-1+	73.2	250	51	
-2+	73.2	250	48	50 ± 2
-1*	69.7	224	134	
-2*	69.7	225	143	139 ± 5

+ Aqueous sample filtered through pre-wetted Whatman #2 filter paper

* Aqueous sample carefully siphoned from bottle.

Analytical Chemistry
Special Study 70-6
Job No. 1630346

STUDY OF THE OPEN AIR COMBUSTION OF PAPER CONTAINING AROCLOR 1242

INTRODUCTION

It has been proposed from the observed distribution of polychlorinated biphenyls in tissue samples taken from marine organisms that global eco-distribution of these materials occurs via aerial transport (wind currents, airborne particulate matter, etc.)

A qualitative experiment has been performed with the objective of determining if Aroclor in N.C.R. paper is destroyed or volatilized when the paper is disposed of by open burning.

SUMMARY

Under the conditions of this experiment:

1. Aroclor 1242 is easily volatilized from N.C.R. paper containing Aroclor under open air combustion conditions using a Bunsen burner.

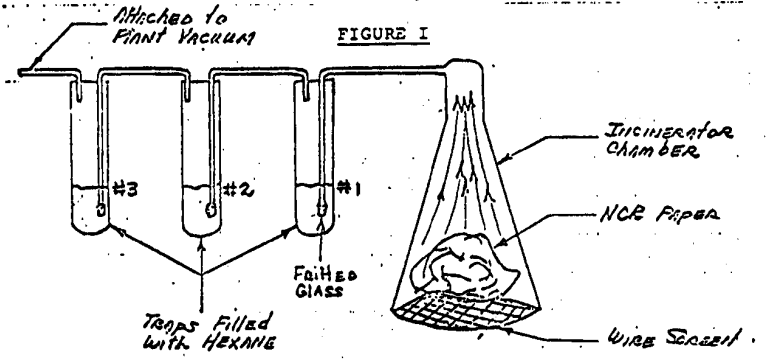
2. Aroclor 1242 undergoes little thermal decomposition at Bunsen burner temperatures.

3. It appears that air pollution could occur via open burning of N.C.R. paper or other Aroclor containing materials.

DISCUSSION

The experiment was carried out in the following manner. Approximately 3g (1 sheet) of paper (CB 15# white, 8½ x 11, long grain, F-3468-4-24-64, The N.C.R. Company) was placed in the incinerator chamber of the apparatus shown in Figure I.

FIGURE I



At this point the vacuum was turned on and the paper ignited using a Bunsen burner. After the paper had completely burned, the hexane in each trap, the hexane extracts of the ash and the walls of the incinerator chamber were analyzed for Aroclor by EC/GC (Analytical Method No. 69-13). Hexane samples from trap #1 were also analyzed after extraction with base and acid to remove any oxidized materials.

RESULTS

The qualitative data supporting the conclusions is shown in Figures 2, 3 and 4.

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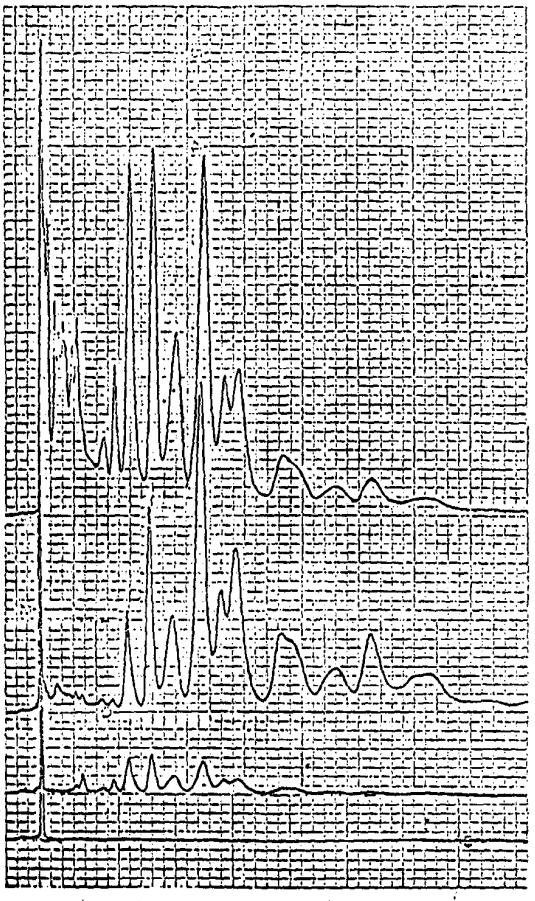
Material Trapped in Hexane
Aroclor 1242 Standard
Material Extracted From Residue
Blank

FIG. 2

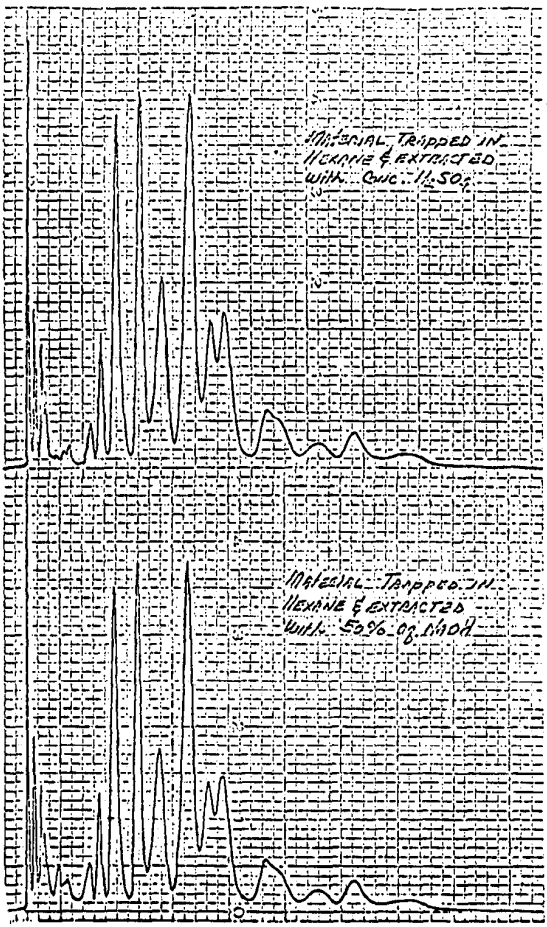

Fig 3

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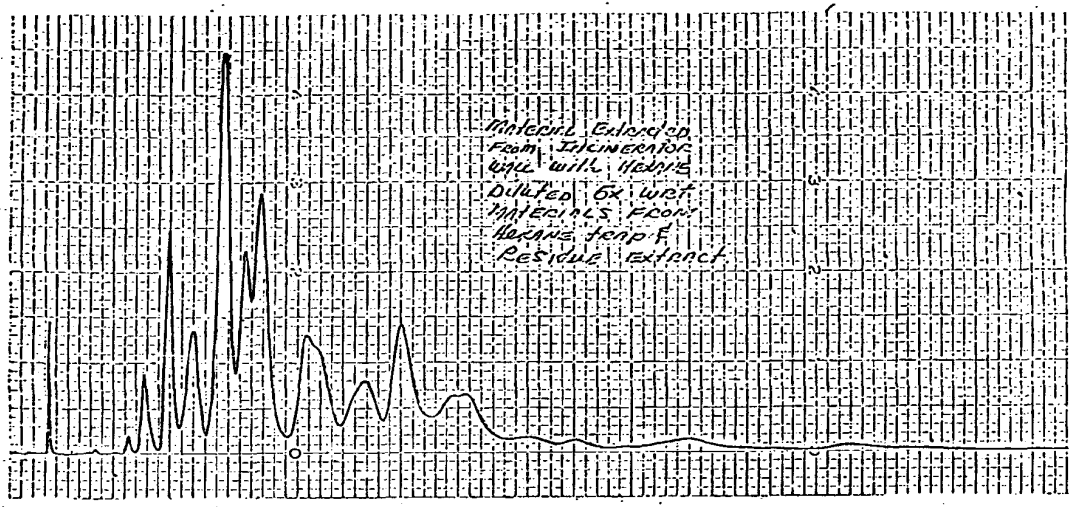
Material Extracted
From Incinerator
Ash with Hexane
Diluted 6x with
Materials From
Hexane Leaf &
Residue Extract

Fig. 4

Analytical Chemistry
Special Study 70-7
Job No. 1630346

THE PARTITION OF POLYCHLORINATED BIPHENYLS BETWEEN HEXANE AND VARIOUS ORGANIC POLAR SOLVENTS

INTRODUCTION

One of the current major interests of residue analysis is the development of practical analytical procedures for the isolation of polychlorinated biphenyls from high fat containing materials such as aquatic, avian and animal tissues. Accordingly, as part of a more general study on methodology the partition characteristics of the polychlorinated biphenyls between hexane and several polar solvents were investigated.

SUMMARY

The partition characteristics of three polychlorinated biphenyls between hexane and four polar solvents have been investigated. The results establish dimethyl formamide-hexane as the most efficient partition solvent system for incorporation into general analytical methods used to analyze high fat containing materials for polychlorinated biphenyls.

DISCUSSION

The criteria employed in evaluating these solvent systems were the quantitative recovery of the polychlorinated biphenyls and the amount of fat retained by the final hexane extract. The initial experiments involved a macro comparison of the recovery of 54% polychlorinated biphenyl with equal volumes of each partition solvent pair. The results of this preliminary study are summarized in Table I. These data establish that the most efficient polar solvent is dimethyl formamide.

The effects of the degree of chlorination and of the dimethyl formamide-hexane ratio were also investigated. The respective results shown in Tables II and III demonstrate that macro amounts of the polychlorinated biphenyls can be quantitatively partitioned using a 5:1 dimethyl formamide-hexane solvent ratio with two extractions.

In order to establish the efficiency of the dimethyl formamide partition clean up method a sample of chicken fat was extracted with hexane and partitioned. The original hexane solution contained 3.2g of chicken fat; after partition the final hexane phase contained 0.9g of fat indicating that 71% of the fat was removed by this procedure.

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A series of recovery experiments were conducted with hexane solutions of chicken fat to which microgram amounts of 54% chlorinated biphenyl had been added. Two successive extractions of the initial hexane solution with 5 times its volume of hexane saturated dimethyl formamide were used and the combined dimethyl formamide phases treated with twice their volume of water. The hexane which layers out is then collected and the aqueous dimethyl formamide extracted twice with hexane. The combined hexane extracts are then washed with an equal amount of water, isolated, and concentrated to a suitable volume. The recoveries were determined by electron capture GC to be quantitative.

RESULTS

The results are shown in Table I, II and III.

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TABLE I PARTITION OF 54% CHLORINATION BIPHENYL BETWEEN HEXANE AND SUCCESSIVE POLAR SOLVENT EXTRACTS.

PERCENTAGE RECOVERED IN EACH EXTRACT				PERCENT RECOVERY	PERCENT REMAINING IN HEXANE	PERCENT OVERALL RECOVERY
1ST	2ND	3RD	4TH			
33.5	25.2	15.3	9.2	80.2	17.8	98.0
13.5	18.1	3.3	0.2	35.1	2.5	93.6
50.3	19.7	6.2	1.1	77.3	23.0	101.0
27.2	29.5	14.6	10.2	72.5	27.5	100.0

TABLE II PARTITION OF SOME CHLORINATED BIPHENYLS BETWEEN HEXANE AND SUCCESSIVE DIMETHYLFORMIDE EXTRACTS. EFFECT OF PERCENT CHLORINATION

PERCENTAGE RECOVERED IN EACH DIMETHYL FORAMIDE EXTRACT				PERCENT TOTAL RECOVERY	PERCENT REMAINING IN HEXANE	PERCENT OVERALL RECOVERY
1ST	2ND	3RD	4TH			
61.9	16.8	12.6	1.2	92.5	0.2	92.7
72.0	19.8	4.9	1.6	98.5	0.5	99.0
67.8	21.6	6.4	2.3	98.1	0.9	99.0

TABLE III PARTITION OF 54% CHLORINATED BIPHENYL BETWEEN HEXANE AND DIMETHYL FORMIDE SUCCESSIVE EXTRACTS. EFFECT OF POLAR/NONPOLAR SOLVENT RATIO.

PERCENTAGE RECOVERED IN EACH DIMETHYL FORMIDE EXTRACT				PERCENT TOTAL RECOVERY	PERCENT REMAINING IN HEXANE	PERCENT OVERALL RECOVERY
1ST	2ND	3RD	4TH			
72.0	19.8	4.9	1.6	98.3	0.5	98.8
86.5	10.9	0.4	—	97.8	0.1	97.9
92.9	6.1	0.1	0.1	99.2	—	99.2

MONS 038147

Analytical Chemistry
Special Study 70-8
Job No. 1630346

EXTRACTION OF AROCLOR 1254 FROM PAINTED SURFACES BY WATER

INTRODUCTION

The following brief study was performed to indicate if Aroclor 1254 can be extracted by water from metallic surfaced painted with paint mixtures containing Aroclor.

SUMMARY

Detectable amounts (22 to 68 ppb) of Aroclor 1254 were found in water exposed to tin plates coated with a paint mixtures containing Aroclor 1254.

DISCUSSION

Tin plate sheets 1.5" x 4.5" were coated by dipping in a mixture containing:

34.7% Parlon S-10
13.7% Aroclor 1254
8.3% Aroclor 5460
3.3% Dioctyl Sebacata
40.0% Titanox AGG

in an equal weight of xylene. After drying the coated plates were placed in 16 oz. wide mouth jars, covered with water, and sealed. The jars were then placed in an oven at 50°C. The weight of paint on the sheet was not determined. After exposure for predetermined periods, the water from each jar was analyzed by GC using Analytical Chemistry Method No. 69-13.

RESULTS

Sample No.	Exposure Period	ppb Aroclor 1254 found
1	2 weeks	28
2	3 weeks	23
3	4 weeks	28
4	5 weeks	68
5	6 weeks	22

The EC/GC fingerprint was altered with respect to the material in the paint mixture. It appeared that some of the lower chlorinated

isomers were solubilized to a larger extent than the more heavily chlorinated ones. This type of alteration is indicative of Aroclor which has been solubilized in water and in part establishes the validity of the experimental results.

It is difficult to determine why twice as much Aroclor 1254 was found in Sample No. 4. A partial explanation could like in the fact that all the tin plates developed pin holes and rusted.

It is also interesting to observe that the amount of Aroclor 1254 found was not a function of exposure time. Unfortunately, a zero exposure time sample was not analyzed.

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Analytical Chemistry
Special Study 70-11
Job No. 1630346

DETERMINATION OF PCB RESIDUES IN FISH FROM LAKE MICHIGAN

INTRODUCTION

Upon ammendment of the WARF Institue Lake Michigan pesticide investigation to include analysis of selected samples for apparent polychlorinated biphenyls (PCB's), Monsanto as an industrial sponsor agreed to carry out collaborative analysis of a limited number of selected samples for PCB's. The results of our analysis of sample extracts supplied by WARF are reported.

SUMMARY

Four fish extracts and one human biopsy extract were analyzed by TLC/GC and GC/mass techniques. The presence of all chlorinated hydrocarbons as reported by WARF in two of the samples were confirmed with the exception of BHC which was not analyzed for. Additionally a sample (#9070652) contained lower levels of two unidentified chlorinated compounds observed by GC/mass. PCB's typical of Aroclor 1254 were found in all of the above samples.

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WARF Sample No.	Sample Type & Location	Sample Wt (g)	PPM DDE WARF	PPM DDE Hemp	PPM DDD WARF	PPM DDD Mon	PPM DDT WARF	PPM DDT Mon	PPM Dieldrin WARF	PPM Dieldrin Mon	PPM EPC WARF	PPM EPC Mon	PPM FCE Estimated WARF	PPM FCE Estimated Mon	PCB [illegible] GC [illegible]
9070876	Lake Herring, Fish Extracts Manistee R., Mich., Sample Point No. 52	12.0	2.90	2.54	1.17	—*	1.31	—*	0.13	0.03	0.034	—**	4.37	2.31	?
9070770	Alewife, Perch Extracts Burns Ditch, L. Mich., Sample Point No. 37	12.0	1.56	1.28	1.64	—*	0.13	—*	0.14	0.16	0.030	—**	2.33	2.73	+
9070652	Chub Extracts Manitowoc R., Wis, Sample Point No. 18	12.0	2.34	3.46	0.52	—*	1.31	—*	0.17	0.34	0.021	—**	0.87	1.56	+
9070639	Chub Extracts Kewaunee R., Sample Point No. 15	12.0	2.08	3.05	0.35	—*	1.07	—*	0.12	0.17	0.021	—**	1.35	0.75	?
9120924	Human biopsy Fat Extracts	0.13	2.93	5.48	<0.01	—*	0.21	—*	0.21	—***	0.31	—**	10.1	7.69	?

* Observed by EC/GC Not Analyzed For
** Not Analyzed For
*** 2nd Elution Not Sent

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Aroclor 248	Aroclor 1254	Aroclor 1260	# 9070 876 1st Elution	# 9070 876 2nd Elution	9070 770	# 9070 770 2nd Elution	9070 652 1st Elution	#9070 652 2nd Elution	9070 639 1st Elution	#9070 639 2nd Elution	9120 924
.21	0.43	0.69	0.27	~~0.79~~ 1.21*	0.27	1.21*	0.27	0.79	0.27	0.78	0.26
.27	0.52	0.75	0.35		0.35		0.35	1.21*	0.35	0.98	0.34
.31	0.54	1.00	0.43		0.43		0.43		0.43	1.06	0.42
.32	0.60	1.12	0.53		0.53		0.53		0.53	1.21*	0.49
.35	0.66	1.29	0.54		0.55		0.54		0.56		0.52
.40	0.70	1.51	0.60		0.61		0.62		0.62		0.55
.41	0.79	1.66	0.68		0.68		0.68		0.69		0.62
.52	0.85	1.72	0.72		0.71		0.71		0.72		0.67
.54	0.94	1.93	0.82		0.81		0.81		0.82		0.71
.60	1.03	2.13	0.88		0.89		0.88		0.88		0.82
.68	1.19	2.22	1.00		1.00		1.00		1.00		1.00
.72	1.41	2.43	1.12		1.14		1.12		1.13		1.12
.82	1.60	2.66	1.22		1.24		1.24		1.24		1.14
.87	2.03	3.00	1.31		1.48		1.32		1.34		1.23
.94	2.18	3.54	1.45		1.58		1.46		1.47		1.45
1.04	2.51	4.31	1.50		1.64		1.65		1.66		1.66
1.21	3.34	5.50	1.63		2.24		2.26		2.26		2.26
1.59			2.25		2.48		2.46		2.47		2.65
			2.46								

MONS 038152

*Dieldrin

FM-402 Biomedical G.C./Ni^{63} ECD
6mm x 6' Glass Column 4% XE-60 on
80/100 mesh Chromosorb W, HP, AW-DMCS
[illegible] – 205°C Column – 195°C Detector – [illegible]

II GC/MASS Results

Sample No 9070376 — Sample lost due to punctured septum.

Sample No. 9070770 — tetra- and penta-chlorobiphenyl homologs detected.

Sample No. 9070652 — DDE, tri-, tetra, penta, and hexa-, chlorobiphenyl homo detected.
Two un-identified chlorinat hydrocarbons with molecu weights of 370 and 404 contain respectively 7 and 8 chlori per molecule detected.

Sample No. 9070639 — Ion beam of during GC/mass spectrom de-focused by the large amou of extraneous material pre in the sample extracts.

Sample No. 9120924 — amount of chlorinated hydrog below the present detection capabilities of our GC/M system.

Analytical Chemistry
Special Study 70-12
Job No. 1630346

STUDY OF THE COMPLETENESS OF THERMAL OXIDATION OF POLYCHLORINATED BIPHENYLS AT JOHN ZINK POLLUTION RESEARCH

INTRODUCTION

An integral part of the Aroclor-Environment Program is the development of an effective means of disposing of waste PCB's. One such proposed technique, thermal oxidation, has been tested on three PCB containing materials, Aroclor 1242, Intereen PPO, and Aroclor 1260. These tests were carried out by John Zink Pollution Research, a division of the John Zink Co., at Tulsa, Okla. This special study reports the results of PCB, HCl and Cl_2 analyses of stack gas samples collected during the incineration tests by Wilson-Wright Petroleum Laboratories.

SUMMARY

PCB analysis of all stack gas samples indicates that thermal oxidation at 1600°F to 2000°F can be an effective means of destroying waste PCB's. The data is presented in Table I.

DISCUSSION

Each of the three test materials were incinerated by John Zink personnel at three temperatures - 1600°F, 1800°F and 2000°F. The equipment and conditions employed were reported to Monsanto by the John Zink Co. A copy of the data table is attached.

Stack gas samples were collected at three points by the Wilson-Wright Petroleum laboratory personnel at each temperature with each material. The details of the sampling procedures employed appear in the Wilson-Wright report of 3-10-70, file number 1142; a copy of this report is also attached.

The samples (aqueous caustic and toluene) were prepared for analysis in the following manner. After obtaining a total weight for each caustic sample, 5 and 10 ml aliquots were taken for chloride and chlorine analysis. Total chloride was determined according to Analytical Chemistry Method #68-11 and chlorine as sodium hypochlorite.

The remaining caustic sample was extracted with toluene to remove any entrained PCB's, the extract combined with the toluene sample, and then evaporated to less than 5 ml. The concentrated toluene was chromatographed on Alumina, concentrated, and the PCB content estimated by electron capture gas chromatography (EC/GC).

The EC chromatograms were quantitated by integrating the EC envelope over the region that the corresponding PCB standard material eluted and the amount present estimated from a calibration plot of integrated area versus concentration.

Calculation in this manner eliminates EC active materials that elude earlier than the standard (chlorobenzenes, etc.).

The amount of total organic chlorine present was determined in nine selected samples according to Analytical Method #66-30 for comparison with the estimated amounts of PCB's.

Monsanto Company
Organic Chemicals Division
Applied Sciences Section
St. Louis, Missouri

4/70 - E. S. Tucker, J. Litschgi, R. E. Keller

Temperature °F	Sampling Point	Resonance time (sec.)	Sample Size m^3	g/m^3 HCL	mg/m^3 Cl_2	Estimated mg/m^3 PCB	mg/m^3 Total Organic Chlorine
1600	1	0.67	0.027	27.4	11.1	NC	—
	2	1.71	0.026	51.5	34.2	0.08	—
	3	2.61	0.023	62.6	7.8	0.19	—
1800	1	0.57	0.028	40.4	13.9	0.06	—
	2	1.52	0.025	62.8	10.8	0.11	—
	3	2.52	0.023	61.7	17.4	0.11	—
2000	1	0.67	0.027	48.9	34.8	0.16	1.36
	2	1.78	0.035	50.0	24.6	0.10	1.81
	3	2.71	0.031	35.2	5.2	1.47	6.02
1600	1	0.58	0.025	60.8	24.4	1.13	—
	2	1.56	0.026	84.6	73.1	0.32	—
	3	2.37	0.026	47.3	11.5	4.94	—
1800	1	0.56	0.025	53.6	42.8	0.09	—
	2	1.48	0.026	101.9	71.2	0.07	—
	3	2.26	0.025	67.6	16.0	5.52	—
2000	1	0.76	0.025	86.8	72.0	0.06	>0.43
	2	2.04	0.025	100.0	11.2	0.08	12.93
	3	3.10	0.025	85.6	36.4	2.80	13.67
1600	1	0.52	0.025	43.6	7.6	2.20	—
	2	1.40	0.026	75.4	30.4	7.06	—
	3	2.13	0.025	52.8	61.6	5.20	—
1800	1	0.51	0.026	72.3	60.8	0.14	—
	2	1.35	0.025	100.0	76.0	0.08	—
	3	2.06	0.025	58.7	27.0	2.40	—
2000	1	0.51	0.020	88.5	105.0	0.22	0.92
	2	1.36	0.026	80.0	99.6	0.22	5.83
	3	2.07	0.025	68.8	67.6	7.60	11.34

foot note
not calculated
interferences

MONS 038156

WILSON-WRIGHT, INC. PETROLEUM LABORATORIES

BOX 2526 TULSA, OKLA. 74101 918 LU 4-6060 7 W. HASKELL

March 16, 1970
File 1142

John Zink Company
P. O. Box 7388
Tulsa, Oklahoma 74105

Attention: Mr. Gene McGill

Gentlemen:

The following pages will report our sampling during the 9 oxidation tests using 3 Monsanto materials, on March 10, 1970.

The sampling was done following instructions given in our discussion with Mr. Art Pier and Mr. Jeff Waldbeser on March 9, 1970, and with Mr. Scott Tucker and Mr. Bill Papageorge during the test oxidations on March 10, 1970.

The liquid samples obtained were packaged in oil absorbent material and styrene beeds. The boxes were crated and sent to Mr. E. S. Tucker, Monsanto Company, St. Louis, Mo. on Friday March 13, 1970 by motor freight.

Six of the gas samples were analyzed. The remaining gas samples will be destroyed unless you request otherwise.

We enjoy serving you.

Yours truly,

Clair C. Wilson

Clair C. Wilson

CCW:jw

[redacted]ELSON-WRIGHT, INC. PETROLEUM LABORATORIES

[redacted]2526 TULSA, OKLA. 74101 918 LU 4-6060 7 W. HASKELL

JOHN ZINK COMPANY

THERMO-OXIDIZER

File 1142
March 10, 1970

EQUIPMENT

[redacted] quartz sampling tubes, 3/4 inches outside diameter by 6 [redacted] long were made and turned down 4 inches into a 28 x 15 [redacted] ground Glass ball joint. This jointed onto the inlet tube of [redacted] pyrex gas scrubbing absorber. This absorbing bottle was kept in a water bath.

The gas was drawn through tygon tubing into the second gas absorption tube; through a gas sampling tube and metered with a dry test meter. Laboratory vacuum pumps were used to draw the gas through the sampling train.

PROCEDURE

[redacted] of the quartz sampling tubes were inserted to the centerline of the thermo oxidizer. The third tube was inserted until its tip was even with the centerline of the exhaust stack. The sampling tube was connected to the primary absorber which had been filled with 175 ml of 2 N Sodium Hydroxide solution. This was then connected to the second scrubber which had been filled with sufficient AR Grade Toluene to cover the sintered glass tube. The gas sample tube was connected in and the vacuum pump started.

Approximately 1 cubic foot of gas sample was drawn through the train in [redacted] minutes. The gas sample tube and train disassembled.

The caustic solution in the primary absorber was poured into a glass bottle and the absorber rinsed three times with distilled water into the glass bottle. The absorber was then rinsed with toluene and this rinse put into a second glass bottle. The toluene solution in the second absorber was poured into the second-glass bottle. The absorber was rinsed twice with toluene into this second bottle.

The quartz sampling tube was rinsed with water and poured into the caustic sample. The quartz tube was next rinsed with toluene and this poured into the toluene sample.

MONS 038158

ILSON-WRIGHT, INC. PETROLEUM LABORATORIES

2526 TULSA, OKLA. 74101 918 LU 4-6050 7 W. HASKELL

JOHN ZINK COMPANY

THERMO-OXIDIZER

Monsanto File No. 1142

Aroclor 1242 Date: March 10, 1970

Barometer in. Hg. 29.38

Temp. °F. 2000 Ambient Temp. °F. 46.

RUN NO. 1

Sampling Point	1	2*	3
Inches from center line	0**	0**	8
Inches from stack center plane			0
Impinger solution, milliliters			
First: AR Sodium Hydroxide 1 N.	175	175	175
Second: AR Toluene Malenkrodt	50	135	50
Meter Number	3	1	4
Calibration Factor	0.965	0.995	1.013
Initial Reading Cu. Ft.	0.000	0.000	0.000
Final Reading Cu. Ft.	1.052	1.294	1.365
Temperature °F.	51	64	49
Vacuum millimeters Hg.	30	64	125
Absolute Pressure mm.Hg.	716	726	621
Gas Metered, Gram Mols	1.123	1.481	1.294

*Made about 10 minutes after other samples were taken
**Using a stainless steel probe
Deformed by heat

MONS 038159

WILSON-WRIGHT, INC. PETROLEUM LABORATORIES

BOX 2526 · TULSA, OKLA. 74101 · 918 LU 4-6060 · 7 W. HASKELL

JOHN ZINK COMPANY

THERMO-OXIDIZER

Monsanto Company

File 1142
March 10, 1970

On the first run, the quartz tube could not be put in the second sampling point. A stainless steel tube 1/2 inch outside diameter was used, and this sample taken after the other two samples had been taken. The stainless steel tube was deformed by the heat and was approximately 8 inches below the centerline. The quartz tube at the first point also was deformed by the heat. It was broken by its forced removal through the sampling port. On succeeding runs, the quartz sampling tubes were inserted until the tips were 24 inches from the thermo oxidizer center line.

A stronger cuastic, 2 N was used after the first two runs. The washing of the sample tube was done after the third run. The caustic scrubbers were washed with toluene after the fourth run.

Three materials were oxidized at three different temperatures. Gas in the oxidizer was sampled at three different points. Twenty-seven (27) samples of each; caustic, toluene, and gas were taken, a total of 81.

The toluene samples were put in a polyethylene bag. This was packed into an oil absorbent material. The caustic samples were put in a polyethylene bag and packed in styrene beeds.

Three of the gas samples, runs 2, 5 and 8, at sampling point 2 were analyzed and are reported. These are only indicative of conditions as the water vapor and some of the carbon dioxide have been deleted from the sample.

The chloride content of three other gas samples were measured to check the efficacy of the scrubbing train. No chlorides (less than 10 ppm.) were detected in these samples.

The metered gas volumes were corrected for temperature and pressure to standard cubic feet at 60°F. and 14.7 psia. This was multiplied by 1.196 and reported as gram mols of gas.

WILSON-WRIGHT, INC. PETROLEUM LABORATORIES

BOX 2526 TULSA, OKLA. 74101 918 LU 4-6060 7 W. HASKELL

JOHN ZINK COMPANY

THERMO-OXIDIZER

Company: Monsanto

Material: Aroclor 1242

Burn Temp. °F. 1800

File No. 1142

Date: March 10, 1970

Barometer in. Hg. 29.38

Ambient Temp. °F. 46.

RUN NO. 2

Sampling Point	1	2	3
Inches from center line	24	24	8
Inches from stack center plane			0
Scrubber solution, milliliters			
First: AR Sodium Hydroxide 1 N.	175	175	175
Second: AR Toluene Malenkrodt	50	135	50
Meter Number	3	1	4
Calibration Factor	0.965	0.995	1.013
Initial Reading Cu. Ft.	0.000	0.010	0.000
Final Reading Cu. Ft.	1.093	1.002	1.102
Temperature °F.	53	62	53
Vacuum millimeters Hg.	30	52	130
Absolute Pressure mm.Hg.	716	694	616
Gas Metered, Gram Mols	1.171	1.069	0.968

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JOHN ZINK COMPANY

THERMO-OXIDIZER

Company: Monsanto File No. 1142

Material: Aroclor 1242 Date: March 10, 1970

Barometer in. Hg. 29.34

Furn. Temp. °F. 1600 Ambient Temp. °F. 50

RUN NO. 3

Testing Point	1	2	3
Inches from center line	24	24	8
Inches from stack center plane			0
Absorber solution, milliliters			
First: AR Sodium Hydroxide 2 N.	175	175	175
Second: AR Toluene Malenkrodt	50	135	50
Meter Number	3	1	4
Meter Calibration Factor	0.965	0.995	1.013
Meter Initial Reading Cu. Ft.	0.000	0.000	0.000
Meter Final Reading Cu. Ft.	1.100	1.015	1.099
Meter Temperature °F.	52	58	52
Meter Vacuum millimeters Hg.	52	52	135
Meter Absolute Pressure mm.Hg.	693	693	610
Gas Metered, Gram Mols	1.139	1.096	0.957

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JOHN ZINK COMPANY

THERMO-OXIDIZER

Company: Monsanto

Material: Inerteen PPO

Burn Temp. °F. 2000

File No. 1142

Date: March 10, 1970

Barometer in. Hg. 29.34

Ambient Temp. °F. 48.

RUN NO. 4

Sampling Point	1	2	3
Inches from center line	24	24	8
Inches from stack center plane			0
Scrubber solution, milliliters			
First: AR Sodium Hydroxide 2 N.	175	175	175
Second: AR Toluene Malenkrodt	50	135	50
Meter Number	3	1	4
Calibration Factor	0.965	0.995	1.013
Initial Reading Cu. Ft.	0.000	0.000	0.000
Final Reading Cu. Ft.	0.998	0.991	1.092
Temperature °F.	53	62	53
Vacuum millimeters Hg.	42	62	138
Absolute Pressure mm. Hg.	703	683	607
Gas Metered, Gram Mols	1.050	1.063	1.042

MONS 038163

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[illegible]26 ... TULSA, OKLA. 74101 918 LU 4-6060 7 W. HASKELL

JOHN ZINK COMPANY

THERMO-OXIDIZER

[illegible]Monsanto File No. 1142

[illegible]Inerteen PPO Date: March 10, 1970

Barometer in. Hg. 29.36

[illegible]°F. 1800 Ambient Temp. °F. 46.

RUN NO. 5

[illegible]oint	1	2	3
[illegible]from center line	24	24	8
[illegible]from stack center plane			0
[illegible]olution, milliliters			
[illegible]AR Sodium Hydroxide 2 N.	175	175	175
[illegible]AR Toluene Malenkrodt	50	135	50
[illegible]ber	3	1	4
[illegible]tion Factor	0.965	0.995	1.013
[illegible]Reading Cu. Ft.	0.000	0.000	0.000
[illegible]eading Cu. Ft.	1.000	1.005	1.084
[illegible]ture °F.	51	61	53
[illegible]millimeters Hg.	42	51	138
[illegible]te Pressure mm. Hg.	704	695	608
[illegible]tered, Gram Mols	1.050	1.095	1.036

MONS 038164

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JOHN ZINK COMPANY

THERMO-OXIDIZER

Company: Monsanto

Material: Inerteen PPO

Burn Temp. °F. 1600

File No. 1142

Date: March 10, 1970

Barometer in. Hg. 29.36

Ambient Temp. °F. 46

RUN NO. 6

Sampling Point	1	2	3
Inches from center line	24	24	8
Inches from stack center plane			0
Scrubber solution, milliliters			
First: AR Sodium Hydroxide 2 N.	175	175	175
Second: AR Toluene Malenkrodt	50	135	50
Meter Number	3	1	4
Calibration Factor	0.965	0.995	1.013
Initial Reading Cu. Ft.	0.000	0.000	0.000
Final Reading Cu. Ft.	1.006	0.992	1.085
Temperature °F.	50	60	53
Vacuum millimeters Hg.	35	48	140
Absolute Pressure mm. Hg.	711	698	606
Gas Metered, Gram Mols	1.064	1.083	1.033

MONS 038165

[redacted]-WRIGHT, INC. PETROLEUM LABORATORIES
[redacted] TULSA, OKLA. 74101 918 LU 4-6060 7 W. HASKELL

JOHN ZINK COMPANY

THERMO-OXIDIZER

[redacted] Monsanto — File No. 1142
[redacted] Aroclor 1260 — Date: March 10, 1970
[redacted] — Barometer in. Hg. 29.36
[redacted] F. 2000 — Ambient Temp. °G. 46

RUN NO. 7

[redacted] Point	1	2	3
[redacted] from Center line	24	24	8
[redacted] from stack center plane			0
[redacted] olution, milliliters			
[redacted] AR Sodium Hydroxide 2 N.	175	175	175
[redacted] AR Toluene Malenkrodt	50	135	50
[redacted] er	3	1	4
[redacted] bration Factor	0.965	0.995	1.013
[redacted] T Reading Cu. Ft.	0.000	0.000	0.000
[redacted] Reading Cu. Ft.	0.792	0.992	1.112
[redacted] ature °F.	48	59	52
[redacted] millimeters Hg.	33	52	142
[redacted] te Pressure mm. Hg.	713	694	604
[redacted] tered, Gram Mols	0.837	1.075	1.053

MONS 038166

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BOX 2526 TULSA, OKLA. 74101 918 LU 4-6060 7 W. HASKELL

JOHN ZINK COMPANY

THERMO-OXIDIZER

Company: Monsanto

Material: Aroclor 1260

Burn Temp. °F. 1800

File No. 1142

Date: March 10, 1970

Barometer in. Hg. 29.38

Ambient Temp. °F. 46

RUN NO. 8

Sampling Point	1	2	3
Inches from Center line	24	24	8
Inches from stack center plane			0
Scrubber solution, milliliters			
First: AR Sodium Hydroxide 2 N.	175	175	175
Second: AR Toluene Malenkrodt	50	135	50
Meter Number	3	1	4
Calibration Factor	0.965	0.995	1.013
Initial Reading Cu. Ft.	0.000	0.000	0.000
Final Reading Cu. Ft.	1.000	1.000	1.110
Temperature °F.	48	62	52
Vacuum millimeters Hg.	33	51	140
Absolute Pressure mm. Hg.	713	695	606
Gas Metered, Gram Mols	1.057	1.092	0.950

[redacted]GON-WRIGHT, INC. PETROLEUM LABORATORIES

[redacted] TULSA, OKLA. 74101 918 LU 4-6060 7 W. HASKELL

JOHN ZINK COMPANY

THERMO-OXIDIZER

[redacted] Monsanto File No. 1142

[redacted] Aroclor 1260 Date: March 10, 1970

Barometer in. Hg. 29.40

[redacted] °F. 1600 Ambient Temp. °F. 45

[redacted] RUN NO. 9

[redacted] Point	1	2	3
[redacted] from center line	24	24	8
[redacted] from stack center plane			0
[redacted] solution, milliliters			
[redacted]: AR Sodium Hydroxide 2N	175	175	175
[redacted]: AR Toluene Malenkrodt	50	135	50
[redacted] Number	3	1	4
[redacted]libration Factor	0.965	0.995	1.013
[redacted]itial Reading Cu. Ft.	0.000	0.000	0.000
[redacted] Reading Cu. Ft.	0.999	0.995	1.107
[redacted]perature °F.	48	59	52
[redacted] millimeters Hg.	35	51	144
[redacted]olute Pressure mm. Hg.	712	688	603
[redacted]metered, Gram Mols	1.054	1.069	1.046

MONS 038168

WILSON-WRIGHT, INC. PETROLEUM LABORATORIES

BOX 2526 TULSA, OKLA. 74101 918 LU 4-6060 7 W. HASKELL

JOHN ZINK COMPANY

THERMO-OXIDIZER

Monsanto Company

File 1142
March 10, 1970

GAS ANALYSIS

Component	Sampling Point 2 Run No. 2 Mol. %	Sampling Point 2 Run No. 5 Mol. %	Sampling Point Run No. 8 Mol. %
Hydrogen	0.00	0.00	0.00
Carbon Monoxide	0.00	0.00	0.00
Carbon Dioxide	5.79	6.91	4.45
Oxygen	4.22	4.36	3.56
Nitrogen	89.99	88.73	91.99
Totals	100.00	100.00	100.00

MONS 038169

John Zink Pollution Research
A Division of JOHN ZINK Company
4401 SOUTH PEORIA • TULSA, OKLAHOMA 74105

March 14, 1970

AIRMAIL

Monsanto Company
800 North Lindbergh Blvd.
St. Louis, Missouri 63166

Attention: Mr. Art Pier, Central Engineering

Gentlemen:

Enclosed you will find a table of the data that resulted from the tests that we ran for you on March 10. We managed to complete all nine of the tests that you asked for, finishing about 9 o'clock that night.

The Aroclor 1260 burned very nicely, but it did require a bit more fuel than the other two wastes in order to burn with a nice stable vortex. We also had no trouble at all pumping and handling the Aroclor 1260 once we had the pipes and gun parts hot.

The residence times we ended up with are in a very good range for comparison with analytical results. I feel these results will give us a good idea of what we need in the way of residence time in a commercial size unit in order to assure complete destruction of the waste material. I feel that the tests were quite sucessful and I hope you find the results useful.

If you have any questions about the tests or any future questions about the commercial size equipment involved, please feel free to call on us.

Yours truly,

JOHN ZINK COMPANY

A. Kim Reyburn

A. Kim Reyburn

AKR:slc
Enclosure

MONS 038170

Results of Thermal Oxidizer Tests Conducted for Monsanto on March 10, 1970

	APOCLOR 1242			INERTEEN PPO			APOCLOR 1260		
	1600° F	1800° F	2000° F	1600° F	1800° F	2000° F	1600°F	1800° F	2000°
Waste Rate (gpm)	0.94	1.00	0.89	1.03	1.03	0.84	0.85	0.85	0.92
Fired Fuel Rate (Btu/Hour)	0.51mm	0.62mm	0.97mm	1.95mm	2.15mm	2.4mm	3.4mm	4.0mm	4.3mm
Quench Water Rate (gpm)	2.3	1.8	___	2.5	1.8	0.15	2.6	1.9	___
Stack Gas									
Combustibles (vol. %)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Excess O_2 (vol. %)	3.8	4.0	4.0	4.2	4.2	4.5	4.0	2.5	3.0
Gun Type	R	R	R	R	R	R	EA865	EA865	EA865
Residence Time (sec.)									
First Point	0.64	0.57	0.67	0.58	0.56	0.76	0.52	0.51	0.51
Second Point	1.71	1.52	1.78	1.56	1.48	2.04	1.40	1.35	1.36
Third Point	2.61	2.32	2.71	2.37	2.26	3.10	2.13	2.06	2.07
Pressures (psig)									
Fuel	<1	<1	<1	<1	<1	<1	1.5	2.5	3.5
Waste	90	85	86	100	95	85	97	100	95
Steam	96	92	91	110	104	94	125	124	124
Pressure (in. w. c.)									
Combustion Air	8.5	8.7	9.2	8.7	8.9	8.9	11.3	11.3	11.3

Equipment: Horizontal Oxidizer with HI-10 Burner

Personnel: Monsanto — Art Pier, Central Engineering
Jeff Waldheser, Central Engineering
Scott Tucker, Organic Chemicals Research
Bill Papageorge, Manager, Environmental Control

John Zink Co. — A. Kim Reyburn, Pollution Control
Gene McGill, Pollution Control
Sam Napier, Test Furnace

MONS 038171

Analytical Chemistry
Special Study 70-13
Job No. 1630346

RETENTION TIMES AND ELECTRON CAPTURE RESPONSE FACTORS OF SOME POLYCHLORINATED BIPHENYL ISOMERS RELATIVE TO DDE

INTRODUCTION

Twenty-one polychlorinated biphenyl isomers were chromatographed to determine the relative electron affinities as a function of the number of chlorines per molecule and their position. DDE, a common pesticide residue, was employed as an internal standard and all calculations were made on a relative basis to eliminate instrumental variations. The information should be useful in estimating the accuracy of the quantitation techniques currently being employed.

SUMMARY

The relative electron affinities of the PCB isomers studied, mono-chloro-hexachloro, varied from 1 for 2-chlorobiphenyl to 58 for 4,4'-dichlorobiphenyl. The data in Table I establishes that the degree of chlorination does not dominate the electron affinity of the chlorinated biphenyl molecule.

EXPERIMENTAL

The polychlorinated biphenyl isomers used in this study were obtained from a variety of Monsanto sources (SRC, CRD, Ruabon, etc.) and were used as is and as labeled. The only criteria used to determine purity was one electron capture peak. All sample sizes were adjusted so that the response for the materials studied did not exceed the linear range of the detector.

The following chromatographic conditions were employed:

Instrument:	H&P Biomedical 402 equipped with a high temperature N_i^{63} electron capture detector.
Column:	6' X 6 mm, glass, 4% XE-60 on 80/100 mesh Chromosorb W, HP.
Injection Port:	~195°C.
Column Oven:	~160°C.*
Detector:	290°C.

*Column oven temperature adjusted to yield a retention time of 8.6 + .1 minutes for DDE.

Flow Rates

Carrier: ~60 ml/min, He
Purge: ~120 ml/min, AR/CH_5 (g/l).

db

Monsanto Company
Organic Chemicals Division
Applied Sciences Section
St. Louis, Missouri

4/70 - E. S. Tucker, R. E. Keller

TABLE I

PCB Isomer	Relative Retention Time (DDE = 1)	Relative Response Factor (DDE = 100)
monochloro		
2-	0.14	2
4-	0.15	1
dichloro		
2,3-	0.20	32
2,4-	0.16	20
2,5-	0.17	19
3,4-	0.28	12
3,5-	0.21	12
2,2'-	0.16	22
2,4'-	0.31	31
4,4'-	0.30	58
trichloro		
2,4,6-	0.20	34
2,3,2'-	0.30	25
2,3,4'-	0.41	45
2,5,2'-	0.19	27
2,5,4'-	0.34	31
3,4,2'-	0.37	27
3,4,4'-	0.61	18
tetrachloro		
2,3',4,4'-	0.42	51
2,2',4,6-	0.28	20
3,3',5,5'-	0.64	37
hexachloro		
2,2',4,4',6,6'	0.50	34

Analytical Chemistry
Special Study 70-17
Job No. 1348046

DETERMINATION OF POLYCHLORINATED BIPHENYL RESIDUES IN RATS FROM 30 DAY AROCLOR FEEDING STUDIES

INTRODUCTION

One of the more significant objectives of the Aroclor - Environment program has been to develop a more complete picture of what occurs to Aroclor products once they are released to the ecosystem. The importance of this information is two fold, first it can be used to guide the development of a more "degradable" Aroclor product and secondly, the general methods developed to study the current Aroclor products can be used to determine the degree of control that must be exercised over the replacement products to avoid similar problems with these materials in the future.

As a part of achieving this overall objective, Industrial Bio-Test Laboratories was requested to carry out a 30 day tissue collection study in albino rats with Aroclor 1242, Aroclor 1254 and Aroclor 1260. This report deals with the subsequent analysis of these tissues for residual PCB's.

The details of tissue collection study can be found in the Industrial Bio-Test report, IBT No. B7009. A copy of this report is attached.

SUMMARY

The results of this study demonstrate that the residual PCB levels in all tissues decreased significantly as the degree of chlorination and the dietary exposure level of the material fed decreased.

Additionally, it was observed that the amount of isomeric alteration increased as the degree of chlorination decreased resulting in the fact that the major PCB isomers retained by the rats fed Aroclor 1242 were those predominantly present in Aroclor 1254 and Aroclor 1260. There were no significant differences in the type of isomeric alteration from tissue to tissue or between sexes.

RESULTS

The experimental data supporting the conclusions are shown in Tables I - III, and in Figures I - VIII.

DISCUSSION

All tissue samples were analyzed as outlined in Analytical Chemistry Method No. 70-1 and the PCB levels estimated using the Aroclor fed as the standard. Upon completion of the electron capture (EC) work all tissue extracts, for each Aroclor fed, were combined and concentrated. The concentrates were then further purified via liquid chromatography on alumina and subjected to high resolution gas

chromatography on a support coated open tubular (S.C.O.T.) column in an attempt to identify as many of the remaining isomers and homologs as possible.

The EC chromatograms (Figures I-III) included in this report are representative of those obtained for each tissue (muscle, liver, kidney, and fat) and each Aroclor. For Aroclor 1242 the dominant PCB homologs retained are peak numbers, 8(tri); 10, 13, 14(tetra); 15, 16(penta); 20, 22(hexa); and 26(hepta). With Aroclor 1254 they are, 15(penta); 20, 22(hexa); and 26(hepta) and with Aroclor 1260 they are, 20, 22(hexa); 26, 27(hepta); and 28, 29, 30(octa). The fact that peaks 10, 13, 14, 15 and 16 are present in Aroclor 1242 extracts but not as dominant in Aroclor 1254 and Aroclor 1260 extracts seems to indicate that if the Aroclor 1242 dietary exposure level were reduced sufficiently these homologs would also be metabolized and/or excreted as they were with Aroclor 1254 and 1260. The very refractory homologs appear to begin with peak #20 in the penta-hexachlorobiphenyl region.

EC chromatograms of fractionated Aroclor 1130 and MCS 1016 (Figure IV) are included for comparison purposes. From these, it can be readily seen that these materials are a marked improvement over Aroclor 1242 in that they contain no observable amounts of the homologs that build up.

The S.C.O.T. column chromatograms (Figures VI-VIII) of the concentrated extracts are even more instructive in that the greater separation power of the column allows the identification of some of the PCB isomers that are not retained. For example, only traces of the following biphenyl isomers, dominantly present in the material fed remain: 2-chloro; 3-chloro; 4-chloro; 2,6-dichloro; 2,4-dichloro; 2,5-dichloro; 2,3'-dichloro; 2,4'-dichloro; 2,5,2'-trichloro; 3,3'-dichloro; 3,4-dichloro; 3,4'-dichloro; 4,4'-dichloro; 2,3,2'-trichloro; 3,4,2'-trichloro and 2,3,4'-trichloro.

The S.C.O.T. column chromatograms (Figure IX) of the proposed substitutes are again included for comparison purposes.

db

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MONS 038176

Test Compound	Sex	Dietary Level	FAT ppm Found		KIDNEY ppm Found		LIVER ppm Found		MUSCLE ppm Found	
			Wet Wt	Lipid Wt	Wet Wt	Lipid Wt	Wet Wt	Lipid Wt	Wet Wt	Lipid Wt
AROCLOR 1242	M	300	-	-	1.40	12.4	2.70	60.6	5.00	37.3
	F	300	276	395	2.30	28.1	4.60	220	12.2	58.3
	M	1000	377	816	5.00	276	10.5	753	16.9	736
	F	1000	412	722	2.30	146	17.4	793	29.0	855
AROCLOR 1254	M	300	695	1094	7.90	655	56.9	2370	55.8	1396
	F	300	660	1289	12.9	1895	53.7	3358	36.0	1274
	M	1000	4660	7208	46.6	3325	176	5318	271	7735
	F	1000	3430	7544	156	12972	227	6678	167	6202
AROCLOR 1260	M	300	2130	3061	28.8	2056	126	3808	81.6	2470
	F	300	2035	3430	16.0	2671	125	6226	136	4849
	M	1000	9190	13715	72.5	5181	200	6263	266	8879
	F	1000	11970	19088	91.0	7579	206	10300	555	24111

TABLE II
ELECTRON CAPTURE GAS CHROMATOGRAMS

Chromatographic Conditions

Instrument: Hewlett-Packard Model 402

Type Detector: Ni^{63} Electron Capture

Column: 6' X 4 mm glass U, 4% XE-60 on 80/100 Mesh Chromosorb W, HP

Column Temperature: 200°C

Detector Temperature: 300°C

Injection Port Temperature: 220°C

Carrier - He, 60 ml/min

Purge - Ar/CH_4 (g/1) 120 ml/min

Electron Capture Peak	Dominant Homolog as Identified by GC/MS		
	Aroclor 1242	Aroclor 1254	Aroclor 1260
1	1	-	-
2	1	-	-
3	2	-	-
4	2	-	-
5	3	-	-
6	3	-	-
7	3	-	-
8	3	-	-
9	3	-	-
10	4	4	-
11	4	4	
12	4	4	-
13	4	4	-
14	4	5	5
15	-	5	5
16	-	5	-
17	-	5	6
18	-	6	6
19	-	6	6
20	-	6	6
21	-	6	6
22	-	6	6
23	-	-	7
24	-	-	7
25	-	-	7
26	-	-	7
27	-	-	7
28	-	-	8
29	-	-	8
30	-	-	8
31	-	-	8

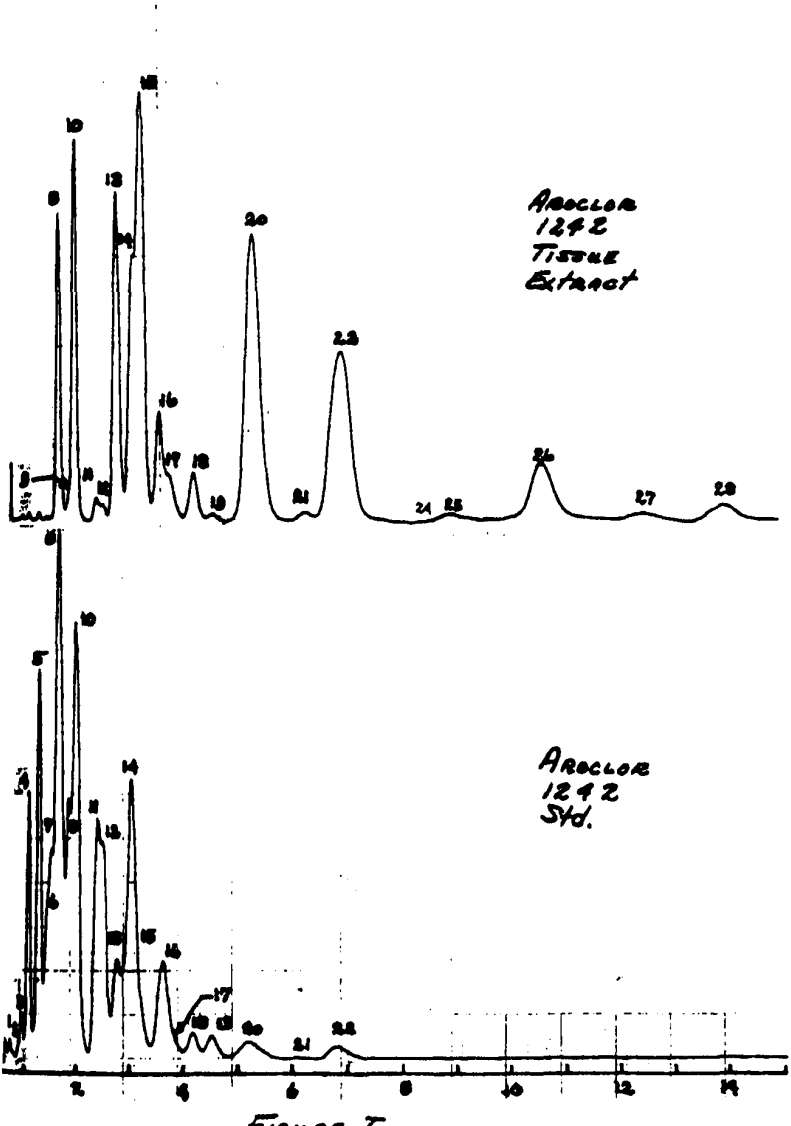


FIGURE I

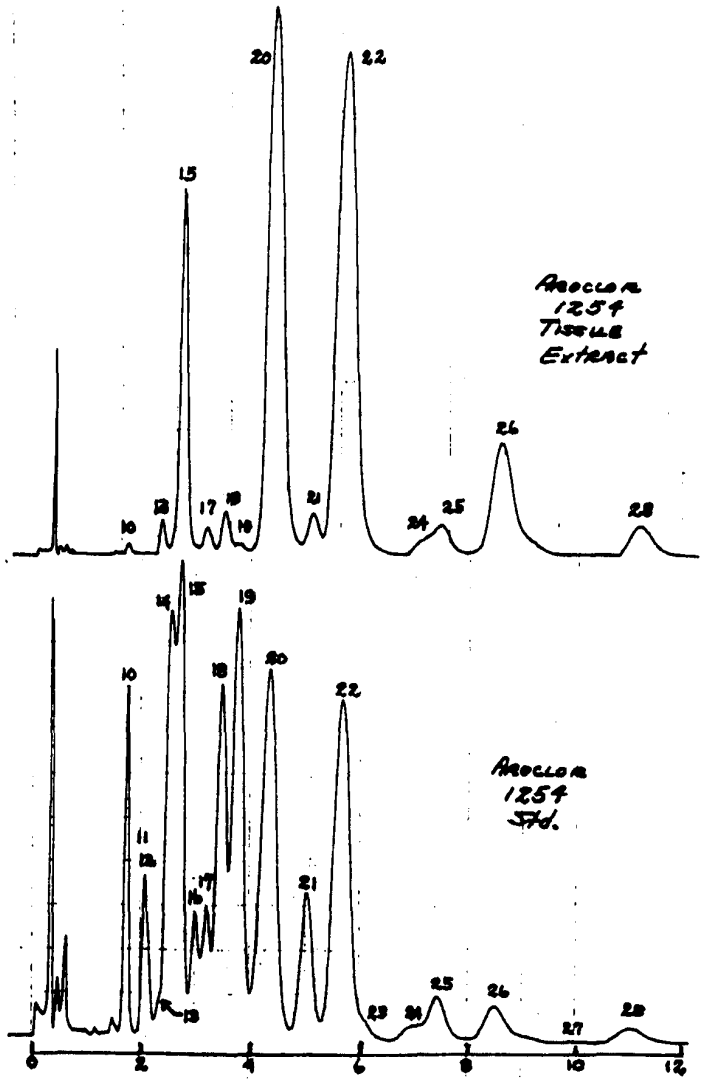


FIGURE II

MONS 038180

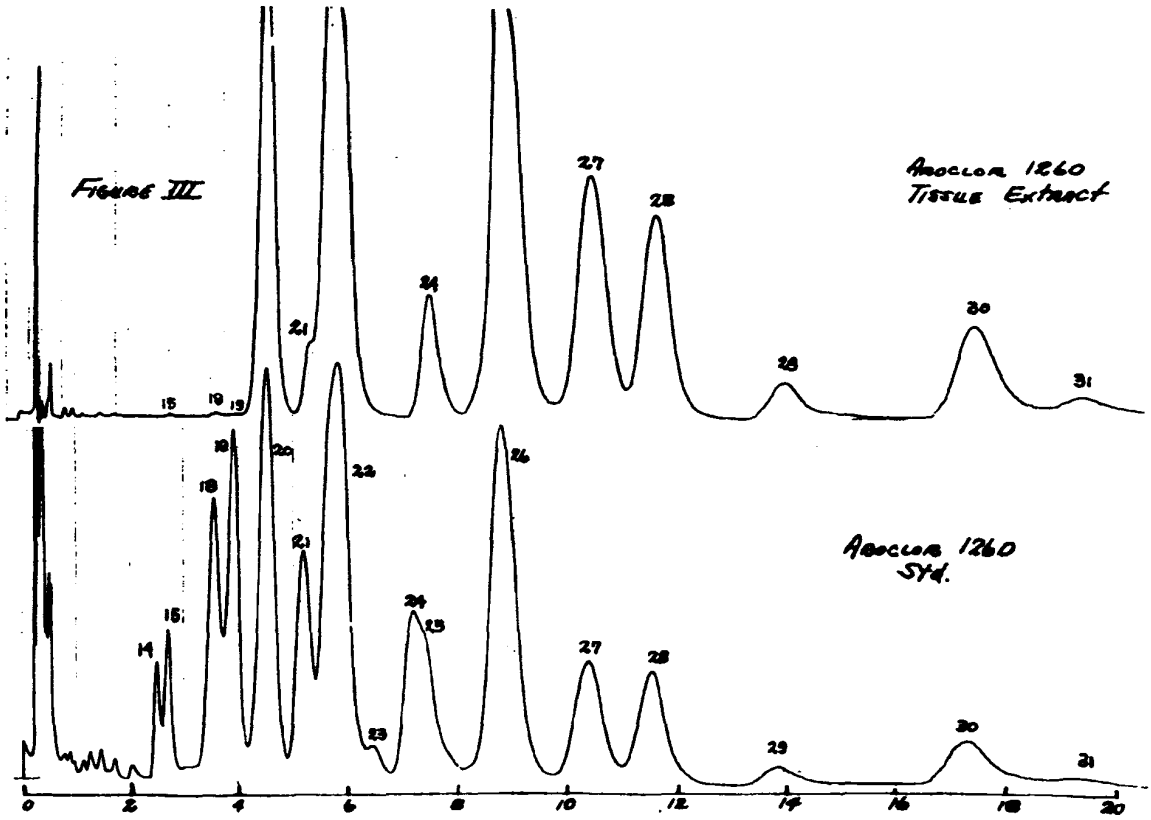


FIGURE III
AROCLOR 1260
TISSUE EXTRACT
AROCLOR 1260
STD.
14
15
16
18
19
20
21
22
23
24
25
26
27
28
29
30
31
0
2
4
6
8
10
12
14
16
18
20

MONS 038181

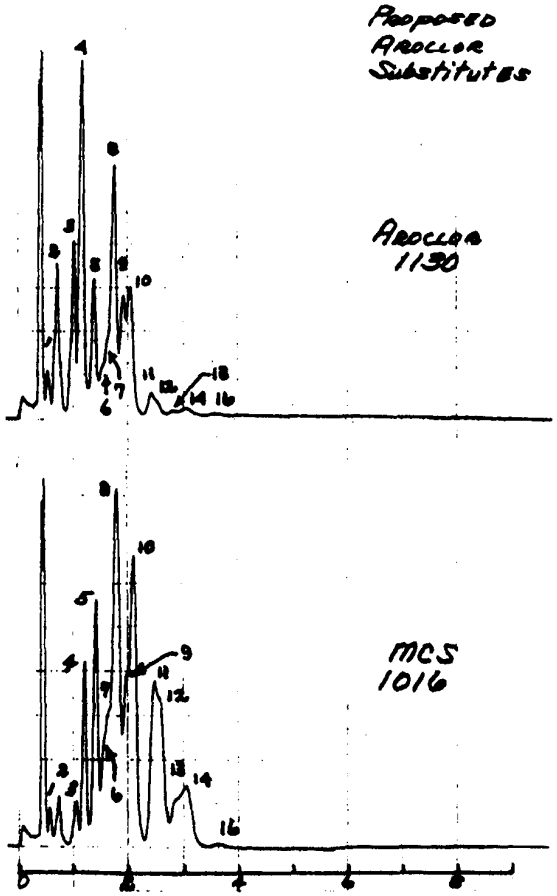


FIGURE IV

MONS 038182

TABLE III

FFAP S.C.O.T. COLUMN CHROMATOGRAMS

Chromatographic Conditions

Instrument: Perkin-Elmer Model 800 equipped with a 30/1 sample inlet splitter

Type Detector: Flame Ionization

Column: 100' X .02" FFAP S.C.O.T. Column

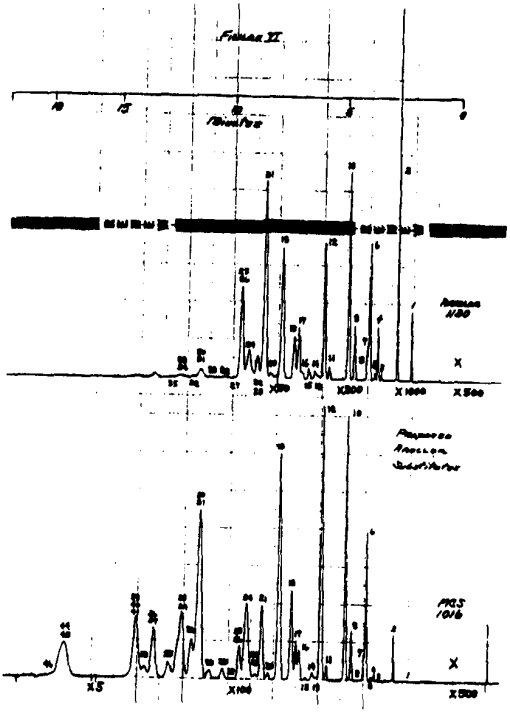
Column Temperature: 235°C

Detector Temperature: 280°C

Injection Port Temperature: 340°C

Carrier Gas - Helium @ 15#

FFAP S.C.O.T. Column Peak No.	Compound As Identified by Retention Time	Number of Chlorines
1	Biphenyl	0
2	2	1
3	3	1
4	4	1
5	2,6	2
6	2,2'	2
7	2,4	2
8	2,5	2
9	2,3'	2
10	2,4'	2
11	-	3
12	2,5,2'	3
13	3,3'	2
14	3,4	2
15	3,4'	2
16	-	3
17	4,4'	2
18	2,3,2'	3
19	2,5,4'	3
20	-	3
21	3,4,2'	3
22	-	3
23-26	-	3
27	2,3,4'	3
28-37	-	4
38	3,4,4'	3
39-42	-	4
43-49	-	5



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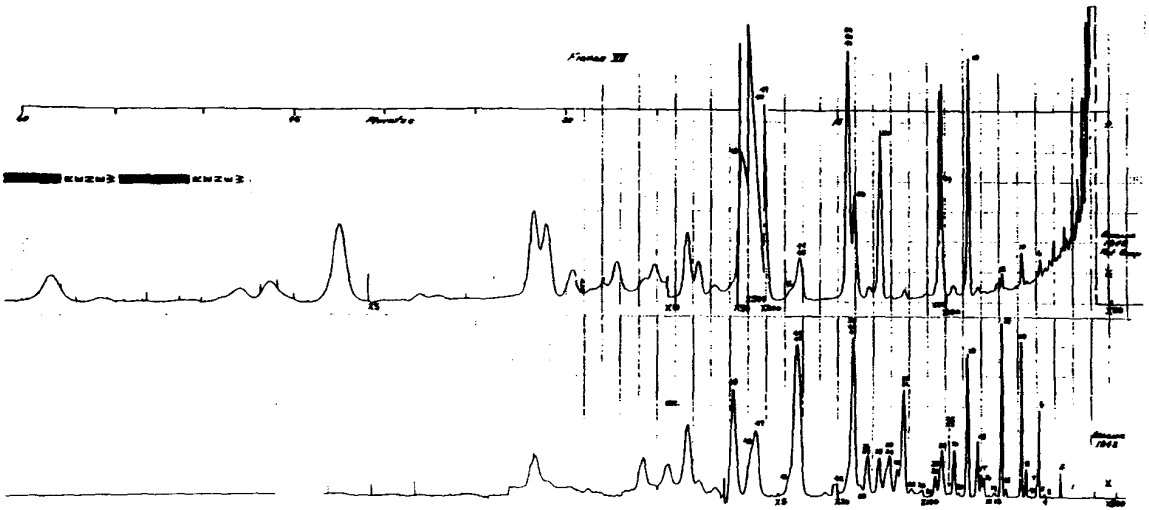

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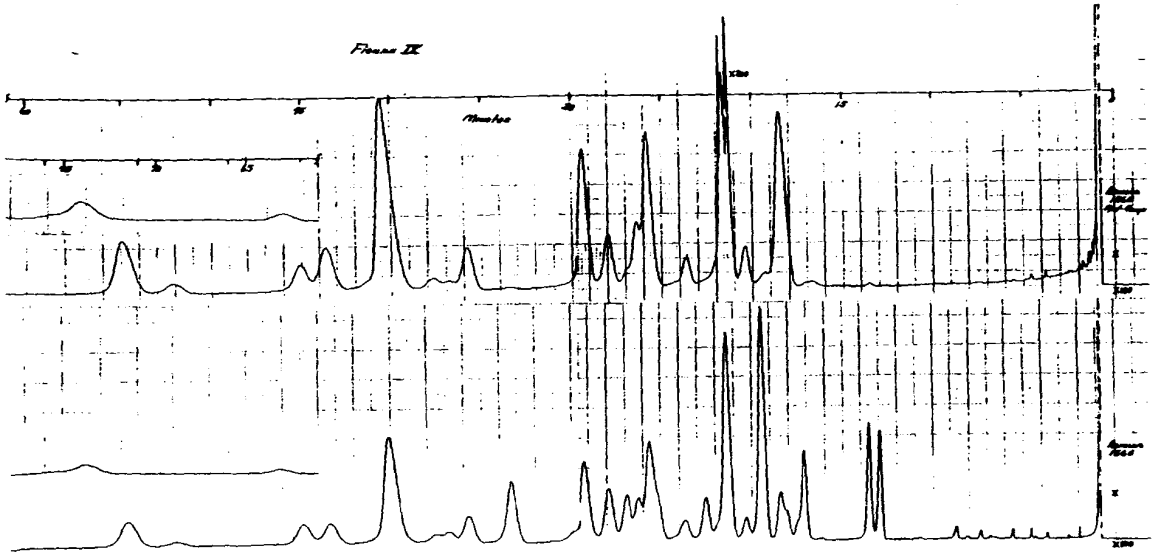

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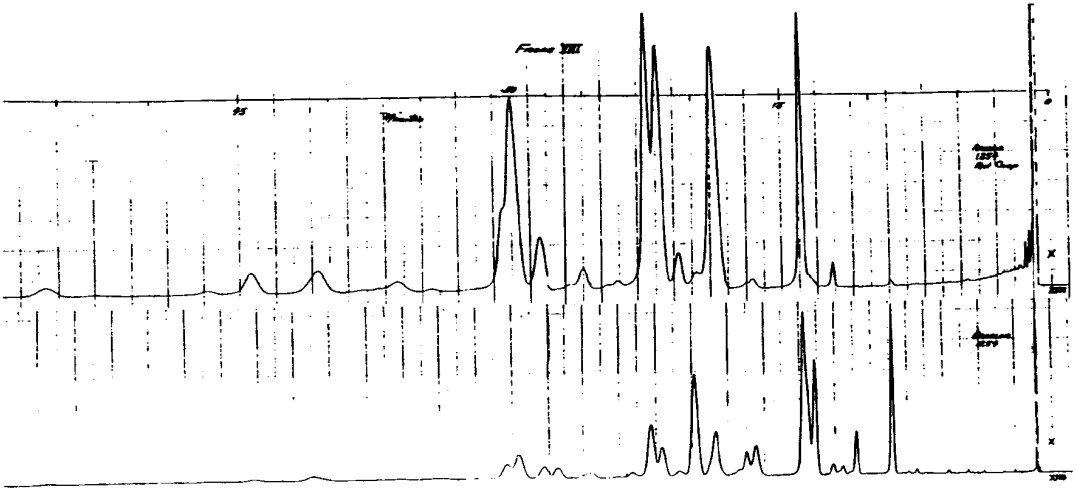

MONS 038187

Analytical Chemistry
Special Study 70-18
Job No. 1348046

CHLORINE DISTRIBUTION OF AROCLOR PRODUCTS BY PACKED COLUMN, ELECTRON CAPTURE, GAS CHROMATOGRAPHY/MASS SPECTROMETRY

NTRODUCTION

his report covers the determination of the number of chlorines per olecules for the peaks observed on the electron capture chromato- rams of our Aroclor product. Extracts of environmental samples are outinely analyzed using electron capture gas chromatography. requently, a mass spectrometer with a gas chromatograph attached to ne inlet is used to confirm the presence of chlorinated biphenyls n the extracts. The gas chromatograph on our mass spectrometer is quipped with a flame ionization detector. Since the electron apture and flame ionization detectors respond differently to the ame compound, a means of translating the mass data back to the lectron capture chromatogram was needed.

UMMARY

n instrument equipped with both an electron capture and a flame onisation detector was set up and the effluent from the column was plit between the two detectors. This set up allowed monitoring of he response of both detectors to the same compound simultaneously. n this manner, the number of chlorines per molecule for the peaks n the EC chromatograms of Aroclors 1242, 1248, 1254 and 1260 can be etermined by comparison of simultaneously recorded FID trace, as dentified by FID/MASS, to the EC trace.

XPERIMENTAL

C onditions used:

Instrument: Hewlett-Packard F&M 5750.

Column: 2M X 1/4" glass column packed with 4% OV-17 Silicone on 80 to 100 mesh, High Performance Chromosorb W.

Column Temp.: 190°C.

Injector Temp.: 225°C On column injection.

Detector Temperatures: Flame Ionization - 350°C.
Electron Capture - 290°C.

Gases: Helium - 75 ml./min.
Hydrogen - 55 ml./min.
Air - 400 ml./min.
10% CH_4/Argon - 120 ml./min.

A pulse interval of 150 μ sec. was used with the Ni-63 electron capture cell. The column effluent was split, 40 parts to the flame ionization detector and 1 part to the electron capture detector.

ANALYTICAL RESULTS

The attached chromatograms were obtained using an XE-60 silicone column which is the one used for the analysis of environmental samples. The more stable OV-17 silicone was chosen for this study to avoid problems with column bleed using the mass spectrometer.

The peaks on the chromatograms are composed of several isomers. Advancing down the series of Aroclors, larger total amounts of chlorine, the ratios of the isomers under any one peak change. The number of chlorines for the principal isomer under a peak is indicated below for Aroclors 1242, 1248, 1254 and 1260.

db

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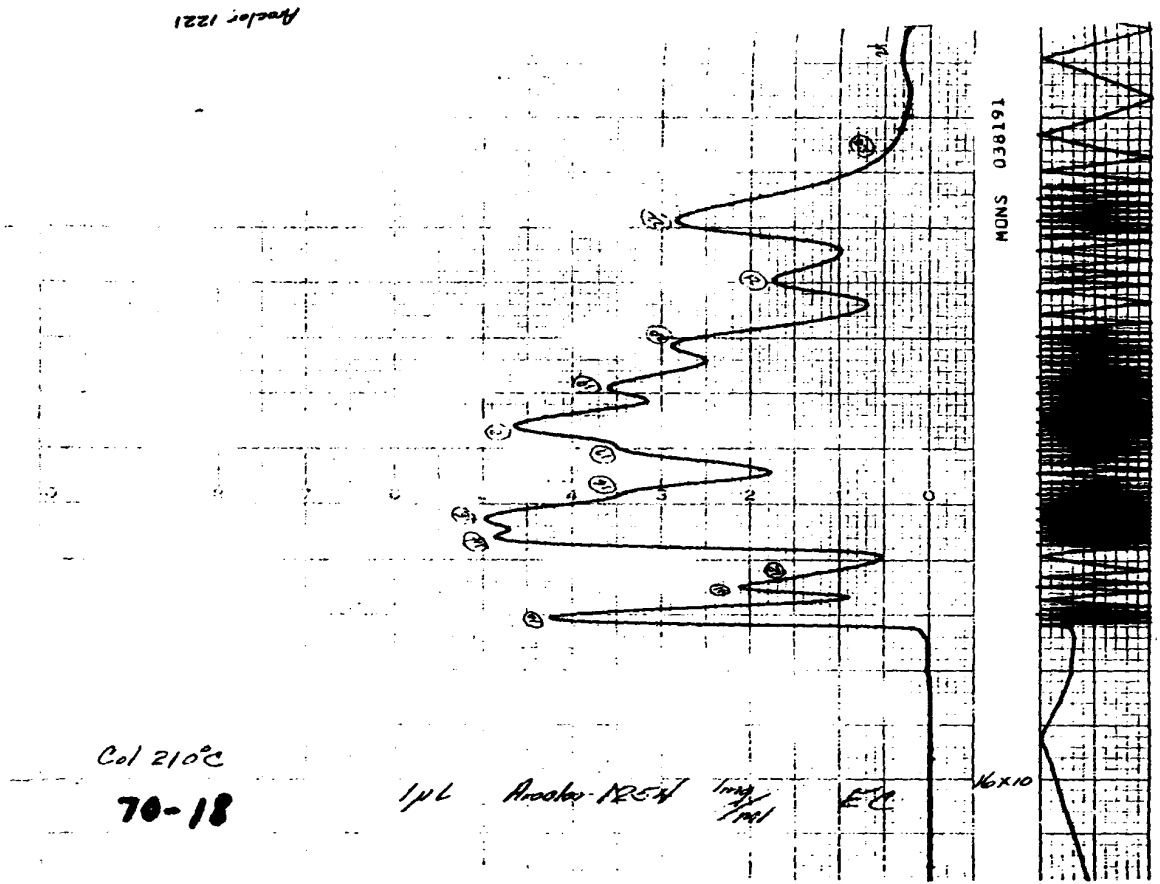
Organic Chemicals Division
Applied Sciences Section
St. Louis, Missouri

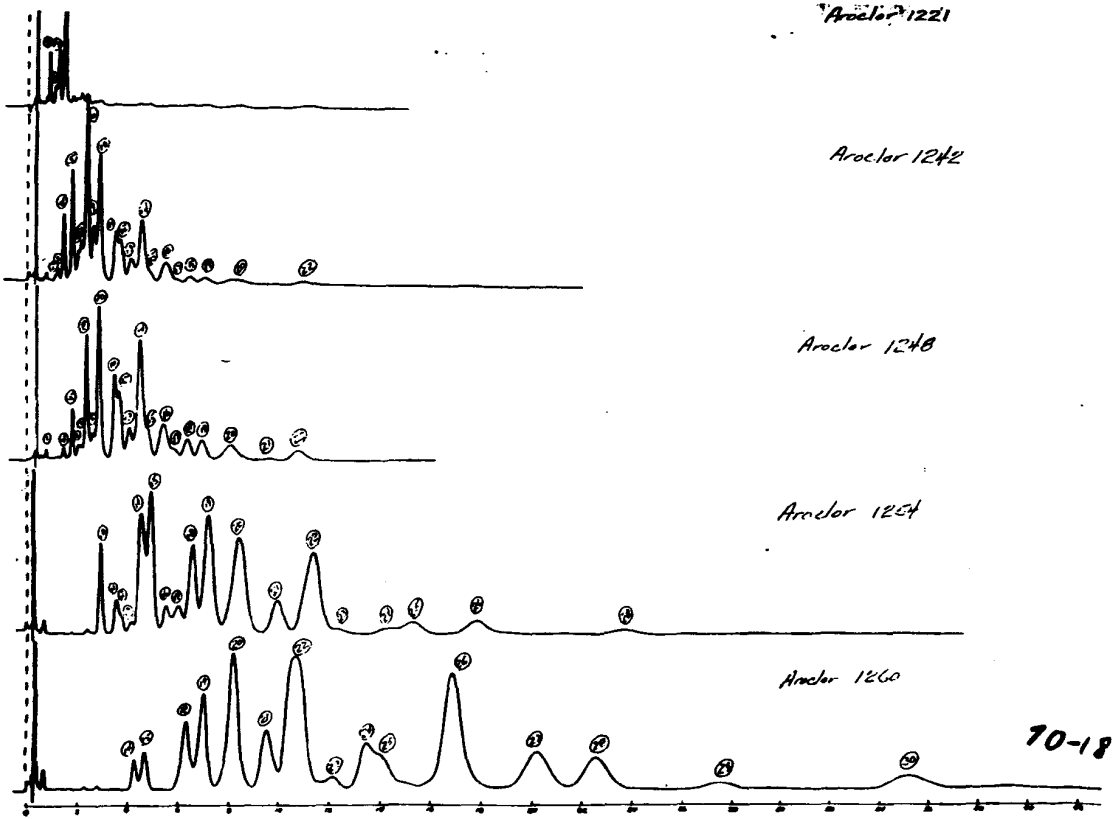
11/70 - W. M. Mees, E. S. Tucker

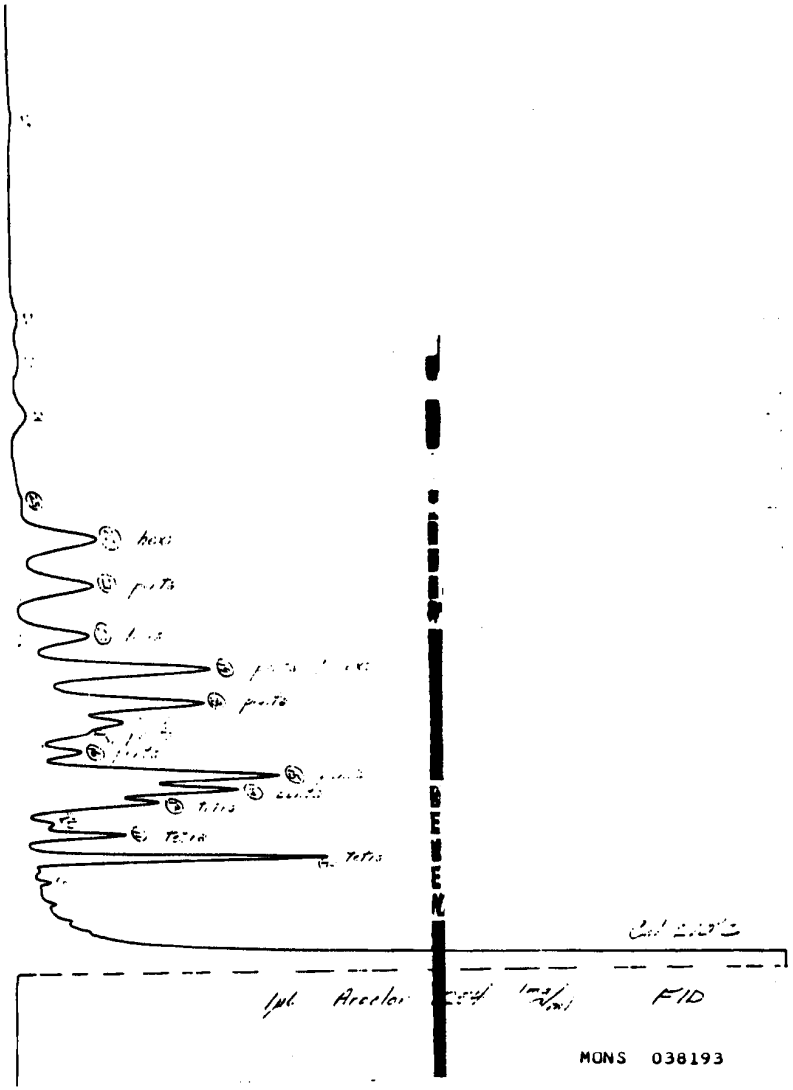
NUMBER OF CHLORINES FOR AROCLOR

Peak	1242		1248		1254		1260	
	#Cl	Area %*	#Cl	Area %*	#Cl	Area %*	#Cl	Area %*
1	1	1.0	-	-	-	-	-	-
2	1	0.7	-	-	-	-	-	-
3	2	4.1	2	1.1	-	-	-	-
4	2	10.7	2	2.1	-	-	-	-
5	3	20.5	3	7.9	-	-	-	-
6	3	6.6	3	4.0	-	-	-	-
7	3	1.6	3	1.0	-	-	-	-
8	3	8.4	3	11.1	-	-	-	-
9	3	8.2	3	6.0	-	-	-	-
10	4	11.5	4	20.4	4	9.5	4	0.8
11	4	8.4	4	11.1	4	3.8	-	-
12	4	3.7	4	6.0	4	1.2	-	-
13	4	2.5	4	5.1	4	6.8	-	-
14	4	8.0	4	15.3	5	10.1	5	3.8
15	-	-	-	-	5	14.8	5	4.0
16	5	4.1	5	8.9	5	3.5	-	-
17	-	-	-	-	5	3.0	-	-
18	-	-	-	-	6	12.3	6	8.6
19	-	-	-	-	6	14.0	6	8.8
20	-	-	-	-	6	5.7	6	10.2
21	-	-	-	-	6	7.4	6	8.4
22	-	-	-	-	6	7.9	6	10.0
23	-	-	-	-	-	-	7	8.8
24	-	-	-	-	-	-	7	6.9
25	-	-	-	-	-	-	7	5.2
26	-	-	-	-	-	-	7	11.9
27	-	-	-	-	-	-	7	-
28	-	-	-	-	-	-	8	12.6

*Uncorrected normalized area per cents


Aroclor 1221
MONS 038191
4
3
2
0
16x10
Col 210°C
70-18
1μL
Aroclor-1254
1mg/ml
EC


Aroclor 1221
Aroclor 1242
Aroclor 1248
Aroclor 1254
Aroclor 1260
70-18



MONS 038193

Analytical Chemistry
Special Study 70-21
Job No. 13480/16300

AROCLOR DEFENSE - 1970 PCB SUPPORT ANALYSIS

INTRODUCTION

In the past year, in addition to the analysis of the numerous samples generated by internal Organic Division research on Aroclor and Aroclor substitutes, several hundred assorted types of samples have been analyzed for PCB's. The results of these analyses have been used by both Monsanto and its customers to: (1) identify, locate, and assess the magnitude of Aroclor pollution problems, (2) help determine what needs to be done to control the pollution source(s), (3) verify that the proposed pollution control procedure(s) is (are) effective, and (4) develop methods of reclamation for recycle and/or safe destruction of Aroclor wastes.

The objective of this special study report is to consolidate this information for ease of reference as well as provide a general over view of the findings.

db

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(Copies to be distributed on request only - contact E. S. Tucker)