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EXPLORATORY ALCOHOLYSIS AND HYDROLYSIS OF CHLORINATED AROMATICS

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I. INTRODUCTION

This work, carried out during the summer of 1955, is chiefly concerned with a portion of a long range program of Dr. J. Dazzi dealing with organic chlorine chemistry (1). It also includes a few miscellaneous, unrelated preparations, carried out for various reasons (2).

II. SUMMARY AND CONCLUSIONS

A. Aroclors 1268, 1262, 1254 and 1248 were hydrolyzed to yield mixtures of polychloro di- and mono-hydroxybiphenyls in satisfactory yields. Aroclor 1242 gave a low yield of phenolics. The hydrolysis of Aroclors and other chlorinated aryl compounds proceeds according to the following equation (shown for Aroclor 1268).

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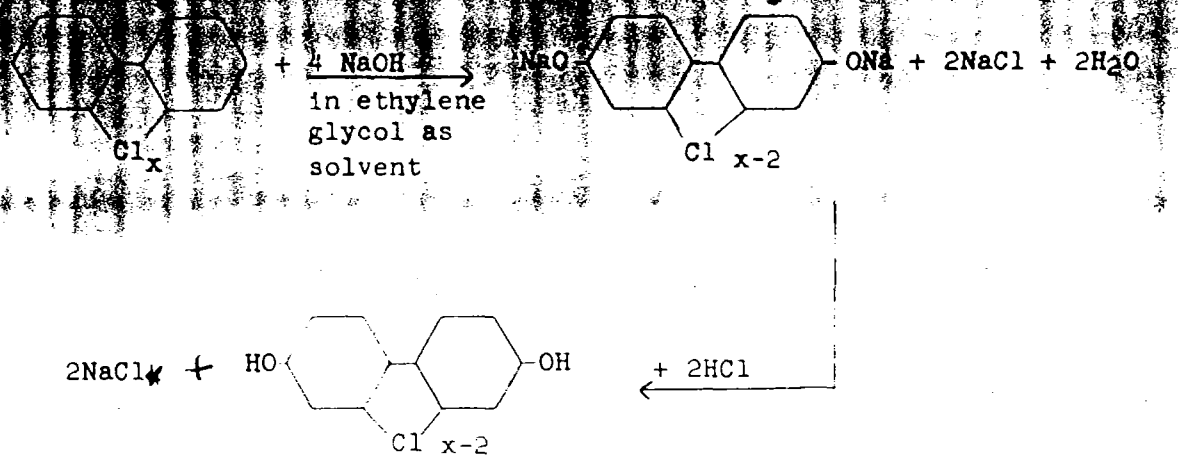
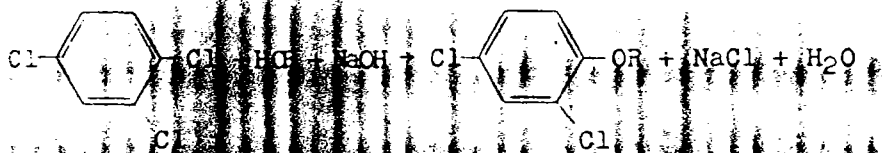


Sodium hydroxide was found to be a satisfactory hydrolyzing agent for Aroclors 1268 and 1262, but only potassium hydroxide was used with the lower Aroclors. The hydrolysis of Aroclor 1268 was carried out with good yield with sodium hydroxide at 190-200°C. for one hour. The other Aroclors were heated for longer periods of time.

B. o-Dichlorobenzene, 1,2,4,5-tetrachlorobenzene and α-chloronaphthalene were hydrolyzed to give 50%, 91%, and 53% yields, respectively, of the corresponding phenols. 1,2,4,5-Tetrachlorobenzene did not form any dichlorodihydroxybenzene at 190-210°C. for 12 hours.

C. Eight ethers were prepared from aryl chlorides containing two or more chlorine atoms, alcohols, and base. Most of these were obtained in fair yield and also some diethers, formed by replacement of a second chlorine atom by the alkoxy radical, were formed. One ether was prepared from α-chloropyridine in the same manner in 88.2% yield.

These ethers were prepared according to the general equation



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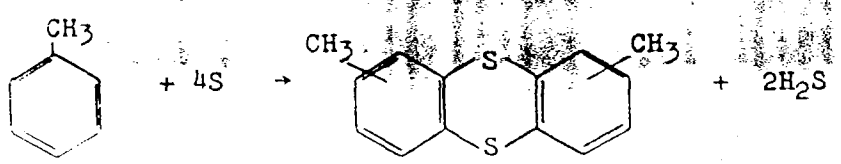
[illegible]

E. 2-Mercaptoethanol gave no thioether with 1,2,4-trichlorobenzene when kept at 160-175°C for 6 hours.

F. A mixture of dimethylthianthrenes was prepared from toluene, sulfur and $AlCl_3$ in 91% yield by the following equation:



G. Ditetrahydrofurfuryl carbonate was prepared by ester interchange in 65% yield.

III. RECOMMENDATIONS

No recommendations are made.

IV. REFERENCES

1. Memo on exploratory research to M. Kosmin by J. Dazzi, April 21, 1954.

2. Memo on program for J. Katon to M. Kosmin by J. Dazzi, June 15, 1955.

3. British Patent [illegible] 764 (to I. G. Farbenindustrie Aktiengesellschaft), June 6, 1935.

4. IRC memo "Low Cost Thianthrene Derivatives". J. Dazzi, June 14, 1955.

5. IRC memo "New Epoxy Compounds for Resins", J. Dazzi, November 16, 1954.

6. Monthly Progress Reports on exploratory work. J. Dazzi Job Nos. 4-9261, 4-9310, 9378-10-3.

7. [illegible] 49, 273 ([illegible])

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A. Hydrolysis of Aromatic Polychloro Compounds (5)

The hydrolyses were carried out by charging ethylene glycol (solvent) and base (10% excess) into a four-necked flask equipped with an efficient stirrer, thermometer and column having a head for removal of condensed vapors. When solution was complete (heat evolved and yellow to reddish-brown color formed), the material to be hydrolyzed was added and the mixture heated further with stirring and continuous removal of water (see Table A). If the hydrolysis was difficult, 3-4 grams of copper powder was added. The mixture was then cooled, diluted with an approximately equal amount of water, and filtered or separated in a separatory funnel to remove unreacted starting material. The aqueous solution was acidified with concentrated hydrochloric acid and the organic layer was separated, washed with water, and distilled. If the phenol was somewhat soluble in water, the water layer was extracted with ether and the ether extracts were distilled.

See Table B for products.

B. Preparation of Aryl Ethers (6)

These ethers were prepared by charging the alcohol, chloro-compound, 100 ml of toluene or xylene, and base (5-10% excess) into a four-necked flask equipped with an efficient stirrer, thermometer, and column having a head for removal of condensed vapors. The material was then heated with stirring and continuous removal of water (see Table C). At the end of the heating period the mixture was cooled and acidified with concentrated hydrochloric acid. The organic layer was then separated, washed with water and distilled.

See Table D for products.

C. Preparation of α-Pyridyl α-Tetrahydrofurfuryl Ether

This ether was prepared as in B above except that only the base and alcohol were charged and after the theoretical amount of water was removed, the α-chloropyridine was added dropwise (see Table C). The reaction product was not acidified after addition of water, but was immediately separated, washed with water, and distilled (see Table D).

321023 D. Attempted Preparation of 2-Hydroxyethyl 2,4-Dichlorophenyl Sulfide

Into a three-liter, four-necked flask equipped with a stirrer, thermometer, and column having a head for removal of condensed vapors, was [illegible] of 2-mer[illegible]

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[illegible] at 170-175° [illegible] additional two hours [illegible] to a gummy [illegible] unpleasant odor [illegible] cooling. The material was not worked [illegible] It is most likely [illegible] molecular weight polyethylene sulfide, previously prepared by [illegible] of 2-mercaptoethanol, see J.D.N.D.

[illegible] E. Water Evolution of Ethylene Glycol at High Temperatures in the Presence of Potassium Hydroxide

Into a three liter, four-necked flask equipped with a stirrer, thermometer, and column bearing a head for removal of condensed vapors, was placed 10 moles (620 g) of ethylene glycol and 3 moles (168 g) of potassium hydroxide. The material was kept at 170-200°C for three hours and 4.6 moles of water were collected. The mixture was cooled, diluted with 250 ml of water and acidified with concentrated hydrochloric acid. The mixture was then filtered to yield 102 g of a brownish solid. The material was not worked up further.

324227 F. Preparation of Dimethyl [illegible] (3), (4)

Into a five liter, three-necked flask equipped with a stirrer, thermometer, and reflux condenser, was placed 14.4 moles (1325 g) of toluene, 0.[illegible] moles (108 g) of aluminum chloride, and 13.75 moles (440 g) of sulfur. The mixture was heated to 50°C., and then heated slowly, the temperature being raised to [illegible]°C. during seven hours. The material was refluxed for an additional twelve hours, cooled to 50°C and the aluminum chloride decomposed with water ([illegible] ml). The material was stirred for [illegible] minutes, 1000ml of water added, and the layers separated. The organic layer was washed twice with 1000 ml portions of water and distilled (see Table E).

324250 G. Preparation of Ditetrahydrofurfuryl Carbonate

Into a three liter, three-necked flask equipped with a stirrer and a column bearing a head for removal of condensed vapors, was charged 4 moles ([illegible] g) of tetrahydrofurfuryl alcohol, 2.[illegible] moles (260 g) of diethyl carbonate, and [illegible] g of potassium carbonate. The mixture was heated with stirring and the ethanol removal as it was distilled. When distillation ceased the material was cooled to 70°C, filtered, and the residue washed with acetone. Distillation yielded 293 g (65%) of material, n_D^{25} 1.4624.

Analysis: Calcd for $C_{11}H_{18}O_5$: C, 57.38; H, 7.88.
Found: C, 58.69; H, 8.[illegible]

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[illegible]

To [illegible] g ([illegible] moles) of [illegible]ylaniline was added 364 g (2 moles) of 1,2,4-trichlorobenzene dropwise at 180-190°C. The material was kept at 180-[illegible] for an additional [illegible] hours, 3 g of copper powder added, and the temperature kept at 180-200°C for an additional 9 hours. The material was cooled and 1500 ml of water were added with stirring. The water gave a negative test with silver nitrate.

326104 To 362 g (1 1/2 moles) of di-2-ethylhexylamine was added 182 g (1 mole) of 1,2,4-trichlorobenzene at 200-220°C over a period of [illegible] hours. Copper cowder (3 g) was added and the temperature maintained at 200-220°C for an additional 35 hours. At the end of this time there was no change in the reaction mixture.

326115 I. Diels-Alder Reaction of Furfuryl 2,4-dichlorophenyl Ether and Maleic Anhydride

Into a 25[illegible] ml flask was placed .05 mole (12.1 g) of furfuryl 2,4-dichlorophenyl ether and about 5[illegible] ml of dry ether. Maleic anhydride (0.05 moles, [illegible] g) was added with shaking until solution was effected. The mixture was allowed to stand for two days, by which time a gummy solid had precipitated. The solid was triturated with ethanol and a white crystalline precipitate was obtained, melting point (crude) [illegible]-93°C. The material was not worked up further due to lack of time.

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Although nucleophilic substitution of aryl halides is well-known, it is generally considered to occur with aryl halides only when the halogen is activated by strong electron-attracting groups. Thus, hydrolysis of o- and p-nitrochlorobenzenes to the corresponding nitrophenols is known to occur quite readily.

There are several examples of the hydrolysis of simple aryl chlorides in the literature, particularly in the patent literature. However, these processes involve high pressures and temperature, and relatively little work has been done with these compounds at atmospheric pressure. The work carried out here has indicated that the reaction is quite general for aryl chlorides with more than one chlorine atom, and gives good yields at temperatures not exceeding 220°C and at atmospheric pressure. Furthermore, it has been found that other moderately strong nucleophilic agents will replace chlorine in aryl chlorides (6).

As would be expected, the ease of reaction, and consequently, the yield of product, under a given set of conditions, varies with both the number of chlorine atoms per benzene ring and the nucleophilic power of the substituting agent. In general, the presence of additional chlorine atoms on a given ring results in increased ease of reaction, as reflected by improved yields under the same reaction conditions. It would also be predicted that the reaction would take place more readily with substituting agents possessing a greater nucleophilic character, and this is borne out, to a large extent, by the results of this investigation.

The reagents used, in order of decreasing nucleophilicity, and thus predicted decreasing ease of reaction, is:

$$^{\ominus}CR > {}^{\ominus}OH > {}^{\ominus}OAr > R\text{-}NH_2 > ArNH_2$$

R = alkyl, Ar = aryl

The results of this investigation bear out this sequence quite well as can be seen from the tables. An example is o-dichlorobenzene, which may be hydrolyzed at 170-200°C, but may be etherified with an alkoxide, as is p-dichlorobenzene, at 140-160°C. It will not, however, form a diaryl ether with phenol at reflux temperatures.

It may be noted that no reaction was obtained with amines and trichlorobenzene. This is probably because the relative unreactivity of the amines necessitates a higher temperature than is accessible at atmospheric pressure. However, both of these used might be expected to [illegible]

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[illegible] mixture, then yield. A good deal of [illegible] the explanation for this lies in the formation of phenols from the [illegible] chloro- compounds. In some cases, the phenols were separated in crude form, but they were not purified. These by-products may also be formed by basic cleavage of the ethers under the reaction conditions. Both of these side reactions could be eliminated by adding the aryl chlorides to the anhydrous solutions of the potassium or sodium alkoxides. This precaution and correct temperature control would likely give better yields of the ethers. In some cases a small amount of copper powder seems to catalyze the etherification, but results indicate that it also catalyzes the hydrolysis of the aryl chloride.

Although this reaction proceeded quite satisfactorily with all monofunctional alcohols used, it failed with two difunctional alcohols. Ethylene glycol does not form an ether with 1,2,4-trichlorobenzene (6) and may be used as a solvent in the hydrolysis with no apparent ether formation. This failure to react was also exhibited by 2-mercaptoethanol. This lack of reactivity toward ether formation does not appear to be readily explicable. However, it may be due to the prevalence of a side reaction, namely, the dehydration of the glycol or thioglycol by the base.

The ethers prepared are to be evaluated as biologicals, functional fluids, dielectrics, and plasticizers with dielectric properties.

From Table B it may be seen that the hydrolyzed Aroclors have wide boiling ranges and a general lack of agreement in analytical data. The Aroclors themselves are a rather complex mixture of polychlorobiphenyls and the hydrolysis acts to increase the complexity of the mixture due to the formation of both the mono- and di-hydroxy compounds. It is likely that the more highly chlorinated Aroclors yield predominantly dihydroxy compounds, while the lower members of the series yield a higher percentage of monophenols. Some reaction condition studies were made with Aroclors 1268 and 1262 in an attempt to improve the reaction from the point of economics. Results have indicated that sodium hydroxide is roughly as effective as potassium hydroxide for these two hydrolyses. It has also been shown that the time of hydrolysis originally thought necessary for Aroclor 1268 may be drastically shortened. Later results have also indicated that the amount of ethylene glycol present as a solvent may be reduced with no adverse effects. At present these hydrolyzed compounds are undergoing evaluation for use as intermediates in epoxy resins.

It was noted that a good deal more than the theoretical amount of water was evolved during the hydrolyses. For this reason the "blank" [illegible] potassium hydroxide was carried

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were made toward identification.

1,2,4,5-Tetrachlorobenzene was hydrolyzed as a possible cheaper route to 2,4,5-trichlorophenol; o-dichlorobenzene to seek a possible utilization for orthene; and α-chloronaphthalene as a preliminary experiment to the preparation of β-naphthol from β-chloronaphthalene. The yield obtained in these reactions is only fair due to various difficulties which could doubtlessly be overcome. The two most important reasons for loss are probably volatility of reactants at the high temperatures involved and solubility of the phenol in water. The solid α-naphthol was found to be difficult to remove from the solid resulting from the ethylene glycol reaction.

The preparation of dimethylthianthrene yields a good many isomers. This is apparent from the very wide boiling range and the fact that some fractions are liquid while others are solid. The total yield, however, is quite good. A relatively small amount of lower boiling material was obtained which is a mixture of ditolyl sulfides.

Ditetrahydrofurfuryl carbonate was prepared in fairly good yield using a standard ester-interchange procedure that requires no further comment. The compound is a liquid over a rather exceptionally wide temperature range, boiling at about 235°C and freezing at about -50°C.

The Diels-Alder reaction of maleic anhydride and furfuryl 2,4-dichlorophenyl ether gave indications of proceeding, but lack of time prevented workup of the material and the product was not isolated in a pure state.

VII. PATENT STATUS

Patent disclosures have been submitted on the etherification and hydrolysis of Aroclors as new reactions (6).

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TABLE A

REACTION CONDITIONS FOR THE HYDROLYSIS OF POLYCHLORO AROMATIC COMPOUNDS

[illegible]	moles	Base	moles	Moles Glycol	time (hrs.)	Temp. (°C)	Product	Yield	[illegible]
268	1	KOH	4.4	24	8	150-160	(a)	111% (wet)	100%
268	1.5	KOH	6.6	30	2	170-180	(a)	130% (wet)	[illegible]
268	1.5	NaOH	6.6	30	1	180-190	(a)	106% (wet)	[illegible]
262	1.5	KOH	6.6	32	9	150-160	(a)	83% (crude)	[illegible]
262	1	NaOH	4.4	22	13	150-160	(a)	94%	[illegible]
254	2	KOH	8.8	30	1 2	150-170 170-200	(a)	80%	78%
248	2.5	KOH	11	30	1 4	150-170 170-200	(a)	81%	68%
242*	3	KOH	13.2	10	1	1[illegible]-200	(a)	50%	50%
[illegible]	3	KOH	6.6	15	3	140-160	o-chloro-phenol	trace	trace
[illegible]	3	KOH	6.6	5	4	150-170 170-200	o-chloro-phenol	50%	60%
[illegible]tra-[illegible]ene	2	KOH	4.4	15	3 7.5	140-170 170-200	2,4,5-tri-chlorophenol	81%	81%
[illegible]etra-[illegible]ene	2	KOH	8.8	15	12	190-210	2,4,5-tri-chlorophenol (b)	60%	97%
[illegible]	2.4	KOH	5.4	10	7	180-210	1-naphthol	53%	29%

[illegible]atalyst added.

[illegible]me of hydroxy and dihydroxy polychlorobiphenyls soluble in aq. NaOH.

(b) No dihydroxydichlorobenzene obtained.

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TABLE B

PRODUCTS FROM THE HYDROLYSES OF
POLYCHLORO AROMATIC COMPOUNDS

Compound	BO (°C)	Pressure (mm. of Hg)	n_D^{25}	NBP	Calculated % C	Calculated % H	Calculated % Cl	Number of OH Groups	Found % C
Hydrolysis product of	---	--	---	321012	34.56	0.80	56.97	2	
				321025	33.10	0.53	62.70	1	
				321028					
Hydrolysis product of	--	--	---	321014	41.20	1.51	48.14	2	
				321016	35.61	1.06	59.38	1	
Hydrolysis product of	140-195	0.1-0.3	1.6381	321031	49.77	2.44	36.74	2	47.71
					46.79	1.96	46.04	1	
Hydrolysis product of	120-165	0.2-0.4	1.6340	321035	57.35	3.25	26.67	2	
					50.43	2.51	35.86	1	
Hydrolysis product of	125-175	0.3-0.4	1.6195		64.30	4.01	17.41	2	
	175-190	0.3-0.4	1.6305		59.41	3.29	30.70	1	
Phenol	120-145	20-30	---	324205	36.4	1.52	53.90		
	75-100	0.2-0.3	---	324211					
	168-172	atmospheric	---	324219	56.05	3.95	27.58	1	
	150-175	20-25	---	324243	83.31	5.59	--	1	80.55

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Hydroxy Compound	Moles	Chloro-Compound	Moles	Base	Moles	Time (hrs)	Temp. (°C)	Product	Yield	Conversion (indicated by acidification)	NBP
BC	6.1	T	8	NaOH	6.25	3 12	170-200	n-Butoxyethyl 2,4-dichlorophenyl ether, 1,2-di-(n-butoxyethoxy)-4-chlorobenzene	52% 1%	93%	321001
EH	7.5	T	5	NaOH	6	11 7.5	180-190	2-Ethylhexyl 2,4-dichlorophenyl ether, 1,2-di-(2-ethylhexoxy)-4-chlorobenzene	65% 3%	60%	321006
EH	4.5	T	1	KOH	1.8	9	165-175	2-Ethylhexyl 2,4-dichlorophenyl ether, 1,2-di-(2-ethylhexoxy)-4-chlorobenzene	84% <1%	100%	321019
Phenol	7.5	orthene	5	NaOH	5	2 6.5	140-170 170-180	Phenyl 2-chlorophenyl ether	[illegible]	[illegible]	324201
Phenol *	5	o-dichloro-benzene	4	KOH	4.4	1.5 6	140-160 160-1[illegible]0	Phenyl 2-chlorophenyl ether	[illegible]	[illegible]	324207
Phenol *	[illegible]	T	5	KOH	5.5	3 6	1[illegible]0-180 195-205	Phenyl 2,4-dichlorophenyl ether 1,2-diphenoxy-4-chlorobenzene	[illegible] [illegible]	[illegible]	324215
BC	3	3,4-dichloro-toluene	2	KOH	3.3	8	160-170	Monoethers 1,2-di-(n-butoxyethoxy)-4-methyl-benzene	[illegible] <1%	[illegible]	324231
BC	3	2,4-dichloro-toluene	2	KOH	[illegible]	[illegible]	160-170	Monoethers 1,3-di(n-butoxyethoxy)-4-methyl-benzene	27% <1%	100%	324235
BC	3	p-dichloro-benzene	2	KOH	3.3	7.5	160-1[illegible]0	n-Butyoxyethyl-4-chlorophenyl ether, 1,4-di-(n-butoxyethoxy)-benzene	[illegible]	[illegible]	3242[illegible]
Tetrahydro-furfuryl alcohol	2	2-Chloro-pyridine	0.75	KOH	[illegible]	[illegible] [illegible] [illegible]	90-100 120-170 150-160	Tetrahydrofurfuryl 2-pyridyl ether	[illegible]	was not acidified	3261[illegible]
Furfuryl alcohol	9	T	6	KOH	9.3	8.5	150-160	furfuryl 2,4-dichlorophenyl ether (polymerized upon distillation)	[illegible]	[illegible]	324247
[illegible]	[illegible]	T	6	KOH	[illegible]	[illegible]	150-160	furfuryl 2,4-dichlorophenyl ether	[illegible]	was not acidified	3261[illegible]

BC = [illegible] cellosolve [illegible] chlorobenzene; EH = 2-ethylhexa[illegible]

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TABLE D

PRODUCTS FROM THE ETHER PREPARATION

Compound	NBP	BP (°C)	pressure (mm.of Hg)	n_D^{25}	Calculated % C	% H	% Cl	% C
[illegible]2,4-dichlorophenyl	321001	126-133	0.1-0.3	1.5158	54.77	6.13	26.95	[illegible]
[illegible]xy[illegible]oxy)-4-[illegible]ene	321001	164-173	0.1-0.3	1.4981	62.68	8.48	10.28	[illegible]
[illegible]2,4-dichlorophenyl	321006 321019	110-140 110-120	0.1-0.3 0.1-0.2	1.5124 1.5122	61.10	7.33	25.77	[illegible] [illegible]
[illegible]hylhexoxy)-4-[illegible]ene	321006 321019	180-189 160-170	0.1-0.3 0.1-0.3	1.4970 1.4975	71.61	10.11	9.64	[illegible] [illegible]
[illegible]chlorophenyl	324215	110-126	0.1-0.3	1.[illegible]	60.28	3.37	29.66	[illegible]
[illegible]-4-chlorobenzene	324215	170-185	0.1-0.3	1.[illegible]	72.85	4.41	11.95	[illegible]
[illegible]thoxy)-3-chloro-[illegible]/or 3-(n-butoxy-[illegible]chlorotoluene	324231	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	14.61	64.49
[illegible]toxyethoxy)-toluene	324231	200-24[illegible]	[illegible].3-0.6	1.[illegible]	70.33	9.94	--	[illegible]
[illegible]hoxy)-2-chloro-[illegible]/or 2-(n-butoxy-[illegible]chlorotoluene	324235	170-185	2[illegible]-25	1.5023	64.32	7.89	14.61	[illegible]
[illegible]toxyethoxy)-toluene	324235	22[illegible]-24[illegible]	[illegible].2-0.3	1.[illegible]	70.33	9.94	--	[illegible]
[illegible]4-chlorophenyl	324239	17[illegible]-1[illegible]	[illegible]	1.[illegible]	[illegible]	7.49	15.50	63.5[illegible]
[illegible]toxyethoxy)-benzene	324239	22[illegible]	[illegible]	1.[illegible]	[illegible]	9.74	--	80.0[illegible]
[illegible]furyl 2-pyridyl	326107	14[illegible]	[illegible]	1.[illegible]	[illegible]	7.31	%N-7.82	66.[illegible]
[illegible]dichlorophenyl	326111	8[illegible]	[illegible].1-[illegible]	1.[illegible]	54.12	3.72	29.05	[illegible]

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TABLE E

PRODUCTS FROM THE PREPARATION OF

DIMETHYLTHIANTHRENES

B.P (°C)	Pressure (mm. of Hg)	Wt. (g.)	n^{25}_D	Calculated % C	Calculated % H	Calculated % S	Found % C	Found % H
105-130	0.1-0.3	19	1.6173				77.47	6.4[illegible]
130-150	0.1-0.2	45	1.6342				74.49	5.95
150-170	0.1-0.2	616	[illegible]	[illegible].81	4.95	26.24		
							69.25	5.2[illegible]
170-207	0.1-[illegible]	[illegible]	[illegible]					
207-300	0.2-4.5	71	solid				69.00	[illegible]
residue	--	57	solid				68.57	[illegible]

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CONTENTS

(By Preparation)

Preparation	Numbers
n-Butoxyethyl 2,4-dichlorophenyl ether	321001-5
2-Ethylhexyl 2,4-dichlorophenyl ether	321006-11, 321019-22
Hydrolysis of Aroclor 1268	321012-3, 321025-30
" " " 1262	321014-8
" " " 1254	321031-4
" " " 1248	321035-8
" " " 1242	321046-50
Phenyl 2,4-dichlorophenyl ether	321215-9

CONTENTS

(By Number)

Number	Compounds
321 1-5	Products from the prep. of n-butoxyethyl-2,4-dichlorophenyl ether
321 6-11	Products from the prep. of 2-ethylhexyl-2,4-dichlorophenyl ether
321 12-3	Hydrolysis products of Aroclor 1268
321 14-8	Hydrolysis products of Aroclor 1262
321 19-22	Products from the prep. of 2-ethylhexyl-2,4-dichlorophenyl ether
321 25-30	Hydrolysis products of Aroclor 1268
321 31-4	Hydrolysis products of Aroclor 1254
321 35-8	Hydrolysis products of Aroclor 1248
321 46-50	Hydrolysis products of Aroclor 1242
321 215-9	Products from the prep. of phenyl 2,4-dichlorophenyl ether

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