CONFIDENTIAL

EXPLORATORY AROCLOR CHEMISTRY. PART II.
DIPHENOLS BY HYDROLYSIS

RD-58-Int. Report No. 1182 (l) May 13, 1958

R&E RESEARCH Dayton, Ohio
By: J. Dazzi
Chemists: J. Dazzi and D. B. Sharp

Job Nos.: 9310; 9378; 9411

Confidential Information
This document contains confidential information which is the property of Monsanto Company, and which must not be disclosed to any duly authorized individual. Any unauthorized use of this document outside the company without the written consent of the recipient is accountable for any loss or damage to the owner.

DISTRIBUTION

Organic Chemicals Division		
(a)	J. H. Lum	St. Louis
(b)	M. C. Throdahl	St. Louis
(c)	F. B. Zienty	St. Louis
Research and Engineering Division		
(d)	E. W. Gluesenkamp/F. C. Meyer	Dayton
(e)	Milton Kosmin	Dayton
(f)	D. T. Mowry/J. K. Craver	St. Louis
(g)	H. K. Nason	St. Louis
(h)	D. B. Sharp	Dayton
(i)	Extra	Dayton
(j)	Extra	Dayton
(k)	Extra	Dayton
(l)	Research and Development Files	St. Louis

TABLE OF CONTENTS

		Page No.
I.	PURPOSE	1
II.	SUMMARY	2
III.	CONCLUSIONS	4
IV.	RECOMMENDATIONS	5
V.	DATA AND DISCUSSION	6
	Syntheses	6
	Evaluation Data	10
	Analytical Procedures	11
VI.	EXPERIMENTAL DETAILS	12
	Apparatus	12
	Raw Materials	12
	Experiments	12
VII.	PATENT STATUS	33
VIII.	REFERENCES	3.
IX.	COST ESTIMATES	36
X.	RECOMMENDED PROCESS	37
XI.	MATERIAL SPECIFICATIONS	38
XII.	ANALYTICAL PROCEDURES	39
XIII.	TOXICITY AND HAZARDS	-3
XIV.	ACKNOWLEDGMENT	-1
XV.	APPENDIX	-2

STLCOPCB4094613

PURPOSE

Monsanto's main market for Aroclors today is in dielectrics but the use patent for this application expired some years ago. It is apparent that studies of the preparation of new derivatives of Aroclors and other highly chlorinated aromatic materials would constitute a sound exploratory program for Monsanto.

After preparation of a few ethers by reaction of Aroclors with alcohols, attempts were made to obtain Aroclor ethers with ethylene glycol. Instead of etherification, hydrolysis occurred readily (R-15-16-17) and re-kindled interest in Monsanto-based polychlorinated diphenols as chemical intermediates. These can be used to prepare special types of epoxy resins, one of the fastest growing areas in the plastics industry. Currently, most of the epoxy resins are based on bisphenol A. This and other uses for the Aroclors and other halogenated aromatics as chemical intermediates has been suggested in several memos (R-1-5).

Experimental data reported herein concerns ethylene glycol hydrolysis of eight Aroclors, three polychlorobenzenes, and hexachlorodiphenyl oxide. Study of an Aroclor methanolic hydrolysis process is reported by J. R. LeBlanc (R-6).

Promising application leads were discovered and reported in R-7 for bis-glycidyl ethers of these diphenols and for hydrolyzed Aroclors as curing agents in polyepoxies, as stabilizers for PVC, and as wood preservatives.

The data reported herein represent part of our exploratory research on chlorinated aromatics carried out between April 1954 and February 1957.

Confidential Information

This document contains [illegible] which is the property of Monsanto [illegible] and which must not [illegible] than duly authorized [illegible]

[illegible] document remains [illegible] Company and must be [illegible]

II. SUMMARY

Alkali-hydrolyses in ethylene glycol of chlorinated compounds such as Aroclors 1271, 1270, 1268, 1262, 1254, 1248, 1168, 5460, hexachlorodiphenyl oxide, hexachlorobenzene, 1,2,4,5-tetrachlorobenzene, p-dichlorobenzene and octachloronaphthalene were investigated. The hydrolysis of Aroclors goes smoothly and rapidly to yield mixtures of isomers. However by this method dihydroxy polychlorobiphenyls are obtained only from Aroclors 1270 and 1271; neither of these Aroclors is now manufactured by Monsanto. The "lower" Aroclors give mixtures of di- and monophenols.

Pilot plant quantities of hydrolyzed Aroclor 1262 and 1268 were prepared using the ethylene glycol process by the Organic Chemicals Division in St. Louis, a direct scale-up of our laboratory procedure (R-8,-9,-10,-12). The hydrolyzed Aroclor 1262 was distilled in the pilot plant.

The failure of the ethylene glycol process to give di-hydrolyzed Aroclor 1268, led to development of a methanolic-alkali pressure process by J. R. LeBlanc (R-6). This process gives difunctional product with 1268.

Hydrolysis of Aroclor 5460 in ethylene glycol gives a mixture of di- and tri-hydrolyzed polychloroterphenyl.

Attempts to replace more than two chlorines by hydroxyl in Aroclor 1270 were unsuccessful.

Although not competitive with current production processes, ethylene glycol hydrolyses can be used for the preparation of two Monsanto products; pentachlorophenol (95%) from hexachlorobenzene, and 2,4,5-trichlorophenol (82%), the 2,4,5-T intermediate, from 1,2,4,5-tetrachlorobenzene (R-13).

Hexachlorodiphenyl oxide and p-dichlorobenzene also hydrolyze in alkali-ethylene glycol; the former gives a mixture of the mono- and dihydroxy products, and the latter gives a practically chlorine-free material of unknown composition.

Copper powder fails to catalyze or increase the extent of hydrolysis. Hydrolysis in isopropylbiphenyl or o-nitroethylbenzene as solvents is not promising; the latter decomposed violently and is unsafe for this use.

Qualitatively, superheated steam or fusion with caustic results in chlorine displacement in Aroclor 1268 at 250-300°C.

The chlorine atoms found to be most labile toward hydrolysis are those in octachloronaphthalene. More than four halogen atoms are replaced. However, due to the known toxicity of the starting material (skin

STLCOPCB4094615

irritant) this reaction has no practical value.

A model molecule, 4,4'-bis-(2,6-dichlorophenol), was prepared by chlorination of 4,4'-diphenol. Studies of this model were most helpful in understanding the chemistry of hydrolyzed Aroclors and their glycidyl ethers and for modifications and interpretation of analyses.

Manufacturing cost estimates for hydrolyzed Aroclor 1268 were reported by J. F. Adams of Organic Research, St. Louis (R-14). Of the two the methanolic process is more economic than the ethylene glycol process, and the former gives diphenol from Aroclor 1268. The latter gives diphenol only with Aroclors 1270, 1271, and 5460. Solvent losses in the ethylene glycol process are major cost factors.

The main market foreseen for a di-hydrolyzed Aroclor is as an intermediate for diglycidyl ethers, which, on curing, give epoxy resins (R-7). Dihydroxy Aroclors, although higher in cost than bisphenol A, form epoxy resins having some properties superior to those of the bisphenol A epoxies (R-7).

Development of di-hydrolyzed Aroclor 1268 (methanol process) is now in progress in the Organic Chemicals Division (R-24). Hydrolyzed Aroclors 1270 and 1268 are efficient curing agents for glycidyl ethers (R-7).

Hydrolyzed Aroclor 1270 was found to be somewhat less active than pentachlorophenol as a wood preservative in preliminary screening; tests are continuing. No significant leads resulted from other biological toxicant evaluations at Creve Coeur of seven hydrolyzed Aroclor samples (R-7).

Because of prior art (R-15,-16,17), only limited patent protection on hydrolyzed Aroclors 5460, 1268 and lower homologs appears likely.

DSW 620589

STLCOPCB4094616

III. CONCLUSIONS

Aroclors are raw materials for dihydroxypolychlorobiphenyls as promising Monsanto-based chemicals. Di-hydrolyzed Aroclors are intermediates for new epoxy resins (R-6,-7), for which hydroxyl di-functionality is critical. However, products that are less than difunctional can be employed as epoxy resin-drying oil fatty acid formulations, useful in surface coating applications (R-7).

Hydrolysis of Aroclors by the ethylene glycol process goes smoothly, but difunctionality is obtained only with Aroclors 1270 and 1271. Di-hydrolyzed 1268 is produced in the methanolic-alkali process (R-6). Lower-chlorinated biphenyls give mixtures containing mono- and diphenols, using the ethylene glycol process. Aroclor 5460 hydrolyzes to a mixture containing between 2-3 phenolic groups in the molecule. Attempts to replace more than 2 Cl atoms in Aroclor 1270 were unsuccessful.

Ethylene glycol hydrolysis of hexachlorobenzene to pentachlorophenol, and 1,2,4,5-tetrachlorobenzene to 2,4,5-trichlorophenol was demonstrated, but cannot compete with current Monsanto processes for these two materials.

The chlorine atoms most reactive toward hydrolysis are those in octachloronaphthalene; at least four halogen atoms are replaced. However, toxicity of the starting compound eliminates this from any further consideration.

STLCOPCB4094617

IV. RECOMMENDATIONS

A study of the successful di-hydrolysis of Aroclor 1268 by the methanolic process has been reported by J. R. LeBlanc (R-6). For hydroxyl di-functionality the ethylene glycol process is not recommended for Aroclor 1268 or lower analogs.

Samples of dihydrolyzed Aroclor 1270 and 5460 (when cleared by our patent department) should be prepared for development purposes and evaluated as curing agents for epoxy compounds.

Glycidyl ethers derived from a tri-hydroxy aryl compound might be well accepted by the trade and justify serious consideration by Monsanto. Hydrolyzed perchlorinated terphenyl is a possible candidate.

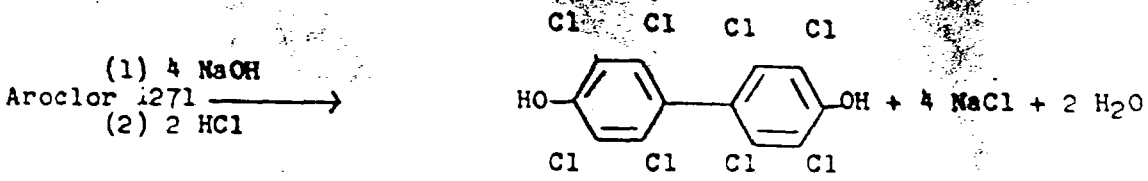
Recommendations for uses for hydrolyzed Aroclors are given in R-3. Octachloronaphthalene, because of its toxicity, should not be investigated further.

DSW 620591

STLCOPCB4094618

V. DISCUSSION

SYNTHESES Two chloro groups of decachlorobiphenyl (Aroclor 1271) hydrolyze with caustic in ethylene glycol solution at 160° to 200°C; Aroclor 1270 also gives diphenol.



This replacement reaction takes place with the lower chlorinated Aroclors 1268, 1168, 1262, 1254, and 1248, however, the yield of dihydroxypolychlorobiphenyl is directly proportional to chlorine content of the Aroclor used: these lower analogs give mixtures containing mono- and diphenols.

The caustic charged may react first with ethylene glycol to form the monosodium salt at 100-120°C. This may be indicated by the water separated by azeotropic distillation. Although not necessary for hydrolysis, removal of this water increases the boiling point of the reaction mixture from 155-160°C to 195-200°C, and the higher temperature reduces the reaction time substantially.

Hydrolysis in ethylene glycol always gives diethylene glycol as a by-product (Expt. 3a). A possible explanation for its formation may be via nucleophilic displacement of hydroxyl group of ethylene glycol as $OH^{\ominus}$ upon attack by a β-hydroxyethoxy anion, formed by NaOH removal of a proton.

$$HOCH_2CH_2O^{\ominus} + HOCH_2CH_2OH \longrightarrow HOCH_2CH_2OCH_2CH_2OH + OH^{\ominus}$$

For each chlorine replaced in hexachlorobenzene, 0.52 mole of diethylene glycol was isolated (Expt. 39); more may actually form since the total material recovery of the mono- and diethylene glycol was only 75.8%.

No difference was observed between NaOH and KOH as hydrolysis agents, and solid or 50% aqueous caustic are equivalent, removing the water azeotropically in the latter case (Expt. 11 and 12). Neither small amounts of specially prepared copper (R-18) nor the use of a copper stirring blade changed the rate of hydrolysis.

As expected, hydrolysis in ethylene-glycol proceeds slowly at relatively low temperature. Hexachlorobenzene gives pentachlorophenol in 13.2% conversion in 5.5 hr at 115°C (Expt. 40).

DSW 620592

STLCOPCB4094619

Hydrolyses of Aroclor 1270 and 1271 are quantitative. The caustic-insoluble material is less than 1% of the Aroclor charged and consists mainly of iron compounds, arising either from the chlorination catalyst or from steel equipment (Expt. 4).

Hydrolyzed Aroclor 1270 and 1271 are best precipitated hot by adding the diphenolate salt solution to dilute HCl at 80-90°C. This yields a product which filters and dries rapidly (Expts. 18-20); precipitation at ice- or room temperature occasionally gave products which filtered slowly and dried very slowly; formation of a hydrate is a possible explanation for this low-temperature behavior. In most of the cases, the isolated hydrolyzed Aroclor 1270 is free of NaCl (Expt. 4). For accurate conclusions based on Cl analysis, NaCl was removed by hot water leaching.

Hydrolyzed Aroclor 1268 and lower analogs melt below 100°C, therefore are best precipitated and washed hot with vigorous stirring; layer separation is fast and complete. The final product dries without foaming at 100-120°C/20 mm. Hydrolyzed Aroclor 1262 and lower homologs were flash-distilled easily at 1-2 mm and showed neither foaming nor thermal decomposition; distillation residues were always low (Expt. 32). The hydrolyzed product boils over a wide range, as expected for a mixture of isomers. The color of the distillate is light yellow.

Directions for the hydrolysis of Aroclor 1262 and 1268 were passed to the Organic Chemicals Division (R-8,-9). They duplicated the procedure in the pilot plant (R-10,-12). The 1262 product was distilled.

Hydrolyzed Aroclor 1268 and all lower analogs hydrolyzed by the ethylene glycol process are mixtures of mono- and dihydroxy Aroclors. Misleading analytical data were obtained with early products. The hydroxyl group determination used at Dayton did not work well, so the OH content was estimated from the C, H and Cl values. Since the Cl contents were all low due to implete peroxide fusion, the calculated OH content was too high, hence indicated diphenol for mixtures of mono- and diphenol. Application of St. Louis analytical procedures for OH group and neutral equivalent were finally used and showed that hydrolyzed Aroclor 1268 and the lower analogs were all mixtures of mono- and dihydroxy Aroclors. J. Herbig, Polymer Evaluation Group, also arrived at this conclusion as a result of comparative studies of curing properties of glycidyl ethers prepared from hydrolyzed Aroclor 1271 and the 1262 analog. Independently, S. E. Gebura at Springfield came to this same conclusion. Because of the inconsistencies between early analytical data and the evaluation data, it was necessary to clarify several points by studies of a model compound of unequivocal phenolic difunctionality. Accordingly, 4,4'-bi-(2,6-dichlorophenol) was prepared by chlorination of 4,4'-diphenol in glacial acetic acid solution. Halogenation was rapid at 40°C, the precipitated material was filtered off, and the filtrate, still containing biphenol, was re-chlorinated to the point where no more HCl was formed (Expt. 49-52). The combined

STLCOPCB4094620

solids were recrystallized from glacial acetic acid giving pure 4,4'-bi-(2,6-dichlorophenol) in 74.3% conversion (Expt. 52). This product gave very good analytical checks for chlorine, OH content, and N.E., using the revised analytical techniques. Concentration of the recrystallization mother liquors gave trichlorinated diphenols.

The original chlorination filtrate contained small amounts of what may be chlorinated quinones, analyzing for between 4 and less than 7 Cl atoms in the molecule (Expt. 49-52). Infrared spectra showed typical quinone absorption at 5.83 microns and a very weak-OH absorption. These minor by-products were not investigated further.

Studies of this model compound led to establishment of reliable analytical methods, and confirmed that a di-functional epoxy compound was required for optimum curing to resins. With this information, efforts were resumed to obtain di-hydrolyzed Aroclors, deemed necessary for the proposed applications. The ethylene glycol process was repeated successfully with Aroclors 1271 and 1270 in larger size batches (Expt. 4). Curing studies of the diglycidyl ethers prepared from these products confirmed their di-functional nature.

Since Aroclor 1270 is no longer manufactured by Monsanto, the decision was made to concentrate our efforts on Aroclor 1268, which is still in interim production. A few runs were made using more drastic conditions, such as higher temperature and longer reaction times (Expt. 28), but the nearest approach was 91% of theory di-functionality (Expt. 29). It was concluded from these experiments that even more drastic reaction conditions were needed to get di-functionality.

A hydrolysis under pressure in methanolic caustic at 190°C was made. Although most of the product was lost by accident, a small amount was recovered and was 95% di-functional (Expt. 37). J. R. LeBlanc subsequently developed a methanol-pressure process giving 100% diphenol from Aroclor 1268 (R-6).

Three runs (Expt. 21-24) on hydrolysis of Aroclor 1271 were made at 155-160°C, each for three-hr. In the first experiment, the water was removed at the rate of formation, while in the second and third experiment the water was not removed. The third experiment was under N_2. Only 92% of the charged Aroclor reacted to give a mixture of mono- and dihydroxy Aroclor 1271, shown later by OH content. Color of the final product was best for the sample hydrolyzed in the absence of water. Data obtained, however, were insufficient to circumvent prior art information in a Monsanto patent disclosing hydrolysis of Aroclors (R-17).

A reduction in process costs could be obtained if other Monsanto-manufactured products could be made economically by the ethylene glycol hydrolysis process in the same plant. Hydrolysis of 1,2,4,5-tetrachlorobenzene to 2,4,5-trichlorophenol, the 2,4,5-T intermediate, went in 92% conversion (Expt. 42). Data were passed to Nitro (Appendix, R-19), but a cost estimate made at Nitro (R-20) indicates that their

DSW 620594

STLCOPCB4094621

autoclave process is 4.2 ¢/lb lower than the glycol process. The latter process is protected for tetrachlorobenzene in a 1950 U.S. patent issued to Givaudan (R-13); Monsanto licenses this process.

Hydrolysis of hexachlorobenzene goes smoothly to yield pentachlorophenol in 96.5% conversion and very high purity (Expt. 39). The sample was sent to St. Louis for purity determination and found to be superior to their reference sample. Monsanto's Santophen-20 is a mixture of tetra- and pentachlorophenol, prepared by chlorination of chlorophenols. The mixture is claimed to be biologically superior to pentachlorophenol.

One of our earliest investigations was to react p-dichlorobenzene with ethylene glycol in the presence of NaOH. Since no reaction took place at 150°C, part of the dehydrated mixture was heated in a bomb at 250°C for 10 hrs. A brittle, heat stable material, very soluble in water, was isolated. Analyses slowed low carbon content (C, 33.55; H, 4.82) practically no chlorine (total 1.13%), and high sodium content (21.51%) The analytical values are most inconclusive (see Expt. 43-44), but no further studies were made.

Undistilled Aroclor 1168 behaves like 1268 on hydrolysis (Expt. 30). Hydrolyzed Aroclor 1268 is a dark brown material. For some applications it should be distilled. In such cases Aroclors 1168 should be used as starting material.

Ethylene glycol hydrolysis of chlorinated terphenyl (Aroclor 5460) gave a quantitative conversion to a mixture of di- and trihydroxypolychloroterphenyl (Expt. 35). In an (Expt. 36) attempt to increase the tri-functionality in the final product by using more caustic, results were inconclusive. Glycidyl ethers derived from hydrolyzed Aroclor 5460 were successfully prepared and cured. Hydrolysis of chlorinated terphenyl by the NaOH methanol pressure process is protected by a Monsanto patent to R. L. Jenkins (R-21).

Octachloronaphthalene and hexachlorodiphenyl oxide were hydrolyzed successfully by the ethylene glycol process. The chlorine atoms in the former compound were found to be the most reactive of any aromatic halogen compound studied to date (Expt. 41). More than four Cl atoms were replaced; the exact Cl content of the final material could not be determined since the product failed to dissolve in the reagent used for the H-8-57 Cl analytical method. Thiolysis and ammonolysis work also showed high chloro group reactivity. However, the toxicity of the octachloronaphthalene precludes further investigation of this as a raw material; it causes severe dermatitis. Hydrolyzed hexachlorodiphenyl oxide is a mixture of mono- and dihydrolyzed material (Expt. 45). The purpose of these two experiments was to broaden the scope of this hydrolysis method.

The ideal hydrolysis solvent from the standpoint of recovery should be water soluble. With this purpose in mind o-nitroethylbenzene, an excellent solvent for Aroclor 1268, was investigated. It is dangerous

STLCOPCB4094622

In this use [illegible] violently and exothermically with KOH at about 100°C [illegible]

Aroclor 1268 was dissolved in m(p)-isopropylbiphenyl, then NaOH was added along with less than molar amounts of ethylene glycol. Incomplete hydrolysis took place, and the product formed was much darker and messier than in all other runs (Expt. 46). This solvent is not recommended for further investigation.

Practically no reaction took place on heating Aroclor 1270 in pyridine and NaOH dissolved in ethylene glycol (Expt. 27).

Highly chlorinated biphenyls are soluble in very few solvents stable to NaOH. However, dimethyl sulfoxide is a good solvent for Aroclor 1270 and also dissolves KOH. None of these solvents, however, can compete price-wise with ethylene glycol which, in turn, is more expensive then methanol. It is conceivable that less ethylene glycol than given in Expt. 4 can be used, but the lower limits were not determined.

The recovery of the solvent can be simplified by distilling off part of the solvent after hydrolysis and before adding water to separate the NaCl formed.

Other possiblities exist for hydrolysis of Aroclors; for example, with super-heated steam or by fusion with NaOH. Both syntheses, based on commercial phenol processes, were demonstrated qualitatively in small size experiments made at 300°C using Aroclor 1268 and 1270 (Expts. 48 and 48-A).

EVALUATION DATA Converting hydrolyzed Aroclors to glycidyl ethers was the main objective of this program, and is the subject of a separate report (R-25).

Another potential use for dihydrolyzed Aroclor is as a curing agent for diglycidyl ethers, where it might replace the popular HET anhydride, hexachlorocyclopentadiene maleic anhydride 1 to 1 adduct. HET anhydride possesses flame-retarding properties, but a drawback is its thermal instability; it loses HCl. Hydrolyzed Aroclors are very heat stable and are recommended for further applications. Hydrolyzed Aroclor 1270 is a relatively strong acid, pH 3.5.

Biological toxicant properties of hydrolyzed Aroclors are given in R-7; no outstanding use is indicated.

Bisphenol A is patented for use as a stabilizer for PVC in U. S. 2,625,521. Samples of hydrolyzed Aroclor 1242, 1248, 1254, 1262, 1268 and 1270 were submitted for screening as stabilizers for PVC and "iso" alcohol esters. Evaluation data are pending on these samples, submitted as Lot Nos. L-971-976 on 9-29-55.

DSW 620596

STLCOPCB4094623

ANALYTICAL PROCEDURES [illegible] composition [illegible] hydrolyzed Aroclors, especially 1260, 1262 [illegible] was estimated from Cl content, carbon and hydrogen values [illegible] significantly between the Aroclor and its hydrolyzed product. Since Dayton's OH determination failed for our product, your early conclusions were based mainly on Cl content, which turned out later to be too low also; this led to high estimates of OH content from which we assumed difunctionality in cases where mixtures of mono- and diphenols existed.

The St. Louis reports on the hydrolysis of Aroclor 1262 and 1268 (R-10, -12) contain a more accurate method for OH determination. Using this method, and the chlorine analysis developed John J. Hawkins at Dayton for highly chlorinated compounds, prior discrepancies in product identity were eliminated.

It was also found that Aroclor 1270 contains 71% Cl; this increases the molecular weight based on Cl content from 480 (Aroclor 1270) to 496.54 and consequently changes the calculated OH and N.E. values for hydrolyzed product. The NE values are more accurate than the OH (R-6). Table I contains the C, H, Cl, OH and NE values for the whole Aroclor series. All calculations are based on Cl as the reference value. It is recommended that a given Aroclor be analyzed since the chlorine content varies from batch to batch. Small chlorine differences give large changes in NE value.

DSW 620597

STLCOPCB4094624

EXPERIMENTAL DETAILS

APPARATUS. All experiments were conducted in standard taper Pyrex glass equipment. Hydrolysis was carried out in a 3- or 4-necked, round-bottom flask equipped with a stirrer, condenser and thermometer. In cases where the water was removed, a 10" Vigreux column carrying a Dean and Stark trap was used. In some experiments a freshly polished copper blade was used to study the influence of copper metal and the rate of reaction.

All melting points were determined on the Fisher-Johns block.

RAW MATERIALS. All Aroclors used are Monsanto products. Aroclor 1271 was received from Anniston via St. Louis as Lot Number K-1583.

Aroclor 1270 represents material (600-700 lbs) from St. Louis sent to Everett and from there to Dayton; this is the only stock in the company. Chlorine content by H-8-57 procedure reveals it contains 71% halogen, consequently its real molecular weight is 496.5 as shown in Table I. Other raw materials used are listed below;

Hexachlorobenzene		Eastman
1,2,4,5-Tetrachlorobenzene		"
Hexachlorodiphenyl Oxide	Dow X 6	Dow
Octachloronaphthalene (Halowax 1051)		Bakelite
p-Dichlorobenzene		Monsanto
m,(p)-Isopropyldiphenyl		Monsanto
o-Ethylnitrobenzene		Monsanto
1,5-Pentanediol		Eastman

All other materials used are stock room items.

EXPERIMENTS

Hydrolysis of Aroclor 1271 and 1270

Expt. 1, 298301-2. A mixture consisting of Aroclor 1271 (0.5 mole, 249.5 g), ethylene glycol (4 moles, 248 g), 700 g of diethylene glycol diethyl ether, and 70 ml of toluene was stirred at 160°C; all dissolved. The mixture was cooled to 80°C, then 1.2 moles (50 g) of powdered sodium hydroxide was added and reheated. At 130°C water was formed, and 29.2 g of water-ethylene glycol mixture was collected, n_D^{25} 1.3681, during two hours. The pot temperature was increased in the meantime to 155°C.

The reaction mixture was stirred for 10 hr at 150-170°C. The cooled mixture was diluted with 300 ml of H_2O, and all solids (NaCl) went into

DSW 620598

STLCOPCB4094625