[illegible]

[illegible] PART
[illegible] DERIVATIVES BY AMMONOLYSIS

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R&E RESEARCH Dayton, Ohio
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This is a continuation of exploratory work on utilization of Aroclors as starting materials for Monsanto. This report covers nitrogen-containing chloro compounds, by chloro-group displacement by ammonia and amines. These were to be tested, for example, as curing agents for epoxy resins. A goal of this project was to expand Monsanto's position on aromatic nitrogen compounds with a line of new diamines derived from a basic Monsanto product.

Interest in these derivatives is low, however, since they are related to benzidine, 4,4'-diaminobiphenyl, and 4-amino-biphenyl, all of which are known carcinogens.

This report covers part-time work from February to May 1957, as a part of a program on Aroclor chemistry, (R-1, R-2, Part III).

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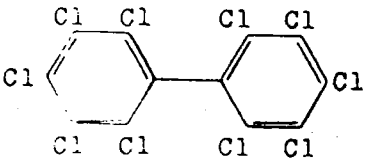
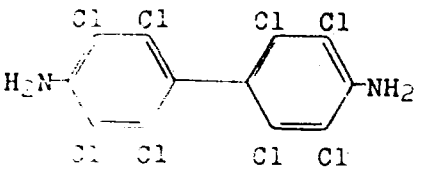
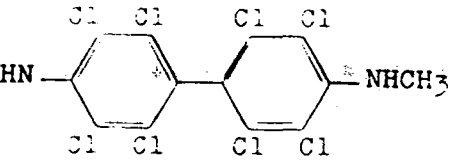
Single experiments were run covering the chemistry outlined below. Further work was not carried on when a stubborn dermatitis attacked the writer; this was probably due to octachloronaphthalene, a known irritant.

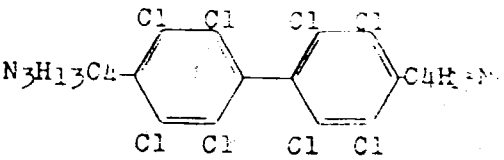
The equations below represent successful reactions of Aroclors and octachloronaphthalene with ammonia and various amines.

1. 

Aroclor 1270



2. Aroclor 1270 $\xrightarrow[\text{Excess}]{H_2NCH_3}$ 

3. Aroclor 1270 $\xrightarrow[\text{Excess}]{H_2NCH_2CH_2NHCH_2CH_2NH_2}$ 

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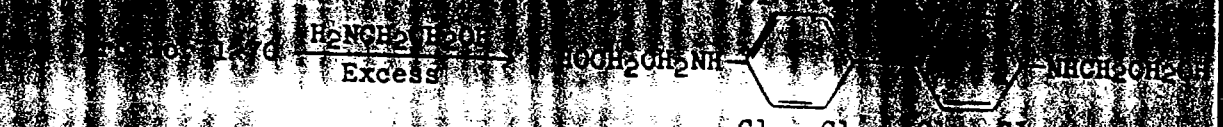
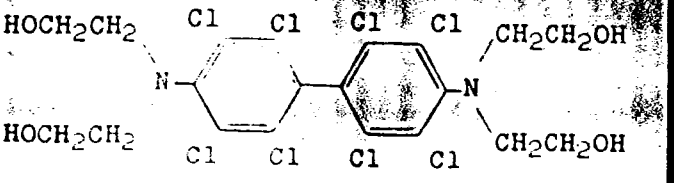
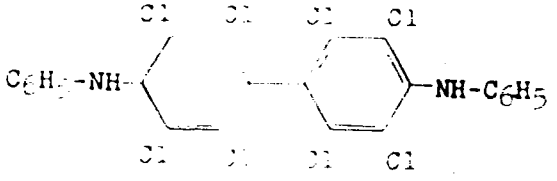


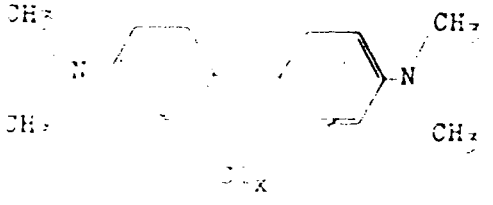
5. Aroclor 1270 $\xrightarrow[\text{Excess}]{HN(CH_2CH_2OH)_2}$



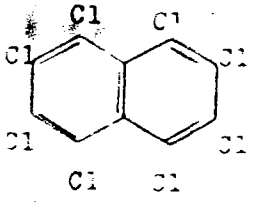
6. Arclor 1270 $\xrightarrow[\text{Excess}]{\text{aniline}}$



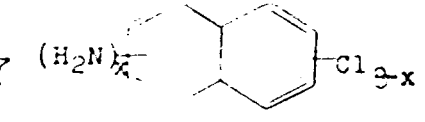
7. Aroclor 1262 $\xrightarrow[\text{excess}]{HN(CH_3)_2}$



8.



$\xrightarrow[\text{aq. NH}_4\text{OH}]{\text{excess}}$



AMMONOLYSIS OF AROCLORS AND OCTACHLORONAPHTHALENE Aroclors 1270, 1260, and 1262 react rapidly with excess aqueous ammonia, giving good conversions to diamines. Of the three substitution reactions of Aroclors studied, ammonolysis is the most complete reaction giving di-functional derivatives. Hydrolysis and thiolysis work is reported separately. This high reactivity extends to the lower chlorinated Aroclor 1262, which is much less active in the other reactions.

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[illegible] of octachloronaphthalene [illegible] on aminolysis and thiolysis, respectively. Each molecule contains the greatest number of labile chlorine atoms of all perchlorinated aromatic compounds studied. More than 4 chlorine atoms were replaced. (Eq. 8).

AROCHLOR 1270 AMINE REACTION PRODUCTS Aroclor 1270 reacts easily with methylamine (Eq. 2), diethylenetriamine (Eq. 3), mono- and diethanolamine (Eq. 4 and 5), and aniline (Eq. 6). Reactions shown in Eq. 3, 4, and 5 were carried out at atmospheric pressure between 150 and 200°C. Conversions and yields were all very high. In the case of diethylenetriamine, close to 3 moles of amine substituted each mole of Aroclor 1270.

Aroclor 1270 reacts with mono- and diethanolamine, respectively, at the nitrogen atoms, as shown by analysis for hydroxyl. Again, high conversions and yields were obtained. Aniline reacted more slowly than all other amines tested, probably because of low base strength.

Our earliest scouting on aminolysis of Aroclors was made in 1955, when gaseous dimethylamine was passed through Aroclor 1262 at atmospheric pressure. A 33% conversion to dimethylamino compound was obtained.

An excellent raw material position would exist for Monsanto on Aroclor ammonolysis products, but the structural relationship of the products to carcinogenic compounds precludes further interest. Tests for carcinogenic action of octachlorobenzidine would cost Monsanto a minimum of $25,000 (R-3). These tests are far from reliable, particularly if negative.

In toxicity tests, exclusive of carcinogenic activity, Aroclor 1270-ammonia product was classified as mild (R-4).

Five patent disclosures were submitted as a result of this work on Aroclor-nitrogen chemistry.

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These few experiments have shown definitely that Aroclors react smoothly with ammonia, primary and secondary amines to yield difunctional products. All are most likely new, with the exception of 4,4'-diamminooctachlorobiphenyl(R-6). The ease of reaction is far greater than in hydrolysis, particularly with lower Aroclors. 1262 gave difunctional amine, whereas it did not undergo di-hydrolysis or di-thiolysis.

Excellent raw material positions would exist for Monsanto on Aroclor-aminolysis products, and raw materials costs seem low.

The similarity of these compounds to carcinogenic benzidines, the cost of a carcinogenic test program, and unreliability of negative results precludes further interest in this area (R-3). It may be important, however, to conduct several experiments for patent purposes, to afford protection if this type of amine is cleared in the future (R-3).

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The literature should be checked carefully for references to carcinogenic action of chlorinated benzidines. Cancer research organizations, such as Sloan-Kettering, might be interested in testing purified samples of the Aroclor-nitrogen derivatives for carcinogenic or anti-cancer activity.

A few more reactions between Aroclors and amines should be tried, as suggested in R-3, p. 5, due care being taken to avoid exposure to the products. This might broaden patent protection for Monsanto, if clearance on these compounds ever develops. Samples should be screened at Dayton as curing agents for epoxy resins and at Creve Coeur as agricultural chemicals.

Also in the Aroclor-nitrogen derivative area, [illegible] probably not a aspect of carcinogenicity, Aroclor dicyanide synthesis should be attempted. Chlorine replacement in Aroclor 127[illegible] and 12[illegible] by cyanide group using $Cu(CN)_2$ or an alkali cyanide, using solvents such as dimethylformamide and dimethylsulfoxide, should be tried. The nitriles should be hydrolyzed to the corresponding acids. Further experimental work should be [illegible] on Aroclor [illegible] - nitrogen compounds on the assumption that the terphenyl structure is less related to [illegible] than the Aroclor 12[illegible] series, (R-[illegible], Part III.).

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DATA AND DISCUSSION

AMMONOLYSIS OF AROCLORS Aroclors 1270, 1268, and 1262 were contacted with excess aqueous ammonium hydroxide. Ammonolysis of Aroclor 1270 went rapidly (1-2 hr.) in 91% conversion at 300°C. A continuous ammonolysis is conceivable. The first experiment (Expt. 3) was made at 250°C as recommended for ammonolysis of hexachlorobenzene (R-5). No reaction took place. This may be due to low solubility since Aroclor 1270 is solid at this temperature (m p 285°C). Hexachlorobenzene, a liquid at 250°C, reacts smoothly at this temperature (R-5).

The Aroclor 1270 ammonolysis product was selectively extracted with 96% sulfuric acid and then precipitated (R-5). Since very low amounts of unreacted Aroclor were isolated (Expt. 1 and 2), it was considered unnecessary to apply this separatory step to the ammonolysis products of Aroclor 1268 and 1262 (Expt. 4 and 5).

Based on elemental analyses, the ammonolysis products of Aroclor 1268 and 1262 are diamines. This contrasts with failure to obtain complete difunctionality via hydrolysis and thiolysis reactions, particularly with Aroclor 1262. The basicity of the amine has a definite influence on the rate of reaction and degree of conversion of the Aroclor to diamine.

The above ammonolysis process was successfully demonstrated with octachloronaphthalene (Halowax-1051) which reacted readily at 280°C (Expt. 6). Although chlorine analyses were inconclusive by the H-t-57 Cl method, ionic Cl found in the reaction mixture, equal to the Cl replaced in the Halowax, was equal to 6.6 atoms of Cl for each mole of the chloro compound charged. No explanation can be found for the very high carbon content or the high melting point (311°C) of the ammonolysis product of octachloronaphthalene. The latter may indicate polymer formation.

Contact with this Halowax may have induced the persistent dermatitis which this writer developed after these experiments. Halowax is known to cause dermatitis.

REACTION OF AROCLORS WITH AMINES AND ETHANOLAMINE Quantitative reaction takes place between Aroclor 1270 and excess methylamine on heating at 300°C in a bomb. Elemental analysis (Expt. 7) indicates that an average of 2.5 chlorine atoms per Aroclor 1270 molecule were replaced by the methylamino group.

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[illegible] chlorine atom substitution by amine groups [illegible] When dimethylamine is bubbled through Aroclor [illegible] at [illegible] displacement occurs. This successful scouting experiment led to the suggested program on Aroclor chemistry (R-2), and the subsequent experimental work.

Mono- and diethanolamine react smoothly and quantitatively with Aroclor 1270 on heating for several hours between 170-210°C (Expts. 9 and 10). Preferential condensation to give carbon-nitrogen linkage is shown by hydroxyl group analyses which check well for the corresconding mono- and di-β-hydroxyethylamino compounds. The rapidity of ammonolysis versus hydrolysis accounts for the selectivity shown in this reaction.

Diethylenetriamine reacts quantitatively and rapidly with Aroclor 1270 at 150-180°C. Elemental analyses and material balance indicate almost three molecules of diethylenetriamine have reacted per Aroclor 1270 molecule. It is conceivable that four diethylenetriamino groups could be introduced under forcing conditions. This specific amino compound was desired for use as a curing agent for epoxy resins since it contains a multiplicity of amine moieties, hence may lead to novel and rapid cures.

Aniline reacted more slowly and less completely (Expt. 12) than all other amines investigated, probably because of low base strength.

It is conceivable that the reactions shown in Eqns. 1-7 (Summary) could be adapted to continuous process conditions since the reaction time is short in all reactions investigated. Reactions shown in Eqns. 1-7 should also be applicable to Aroclor 5460.

EVALUATION DATA None of the Aroclor amine derivatives were screened for special applications. The similarity of these to chlorinated benzidines probably precludes such evaluation due to potential carcinogenicity. The main use originally proposed for these was in epoxy-curing. By combining the diethylenetriamine with Aroclor 1270, a curing agent with low volatility would result. Many amines now used for epoxy-curing have undesirably high volatility; this complicates epoxy during, especially when toxic amines are used.

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APPARATUS: All experiments at atmospheric pressure were conducted in standard-taper, Pyrex-glass equipment in the absence of water. The flask was heated by heating mantle. Solvent was removed under stirring and good temperature control.

Experiments under pressure (Expts. 1, 3-5) were made in stainless steel, 1-liter bomb No. 15. In Expt. 2 the old 3-liter stainless steel bomb No. 2 was used; the bombs were heated electrically.

EXPERIMENTS

Ammonolysis of Aroclors

Experiment 1 (360210-11) A mixture consisting of Aroclor 1270 (0.242 mole, 120 g) and aqueous ammonia (5.6 moles, 357 ml, 28%) was heated in the 1-liter bomb to 300°C over a 65-minute period and then kept for 2 hours at this temperature. No exothermic reaction was noted. The pressure remained unchanged at 2150 psi. After cooling and venting, the dark solid reaction product was removed quantitatively from the bomb, filtered, washed with 150 ml of water and dried. The residue was added to 600 ml of 96% H_2SO_4 and stirred at 35°C for 1 hour. Three grams of iron oxide was removed by filtration. The filtrate was added over 50 minutes to an ice-water mixture (3 kilos) while the temperature was kept under 12°C. The precipitate filtered rapidly, giving 138 grams of the diamine sulfate salt. It was suspended in 800 ml. of water and neutralized to phenolphthalein at room temperature with 151 ml of 2.5N NaOH, giving 100 grams of diaminooctachlorodiphenyl. The material is pure as shown by C, H, Cl, and N analyses given in Table I. Conversion was 91%.

Experiment 2 (360212-3) Experiment 1 was duplicated using 4-fold increase in charge; this consisted of 0.968 mole of Aroclor 1270 and 27 moles of aqueous ammonia. The pressure increased to 2500 psi during the 2.3 hour heating at 304°C; total crude diamine collected was 401 grams. It was dissolved in 2.8 liters of 96% H_2SO_4 and added to 3.2-liters of ice and water. Filtration went very slowly, giving 441 grams of sulfate salt of the amine. It was neutralized with 278 ml of 3N NaOH to give 336 grams of free base. This material, however, failed to dissolve in the reagent used in the H-8-57 modified Cl analytical method. Therefore, it was necessary to use the

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[illegible] values found show that somewhat more than 2 chlorine atoms were replaced by amino groups.

Experiment 3 (360206): The first ammonolysis experiment was made at 250°C using the directions given in R-5 for the preparation of pentachloroaniline. Aroclor 1270 (0.193 mole, 96 g) and NH_3 (4.7 moles, equal to 285 ml 28% aqueous solution) were heated for 1 hour at 250°C while the pressure went up to 1000 psi. The light reaction mixture was low in ionic chlorine as shown by a test tube experiment. The solid material isolated was starting material, insoluble in concentrated H_2SO_4. However, at 300°C the next two experiments (1 and 2) were successful.

Experiment 4 (360217-8) Aroclor 1268 (0.5 mole, 136.2 g) and 430 ml of 28% ammonia (11 moles) were treated for 4 hours at 300°C in bomb 15; the pressure reached 2400 psi. The reaction product was washed free of chloride and ammonia and then dried in a vacuum oven at [illegible]°C for 24 hours until free of amine odor. The diamine collected weighed 116 g, mp 73-75°C. Total ionic Cl found in the combined water washes (2000 ml) was equal to 0.285 mole. This is a good check with the moles of amine group replacing Cl in the final product. Conversion was 92.5%.

Experiment 5 (360280) Aroclor 1262 (0.35 mole, 137 g) was heated with 5 moles of aqueous ammonia at 300°C for 5.5 hours, the pressure reaching 2700 psi. The reaction mixture, a viscous orange material, was removed quantitatively from the bomb by ethanol washes. This solvent was distilled off and the organic material was washed at 80°C; good layer separation occurred. The final product was dried in a vacuum oven at 35°C/30 mm, giving 112 g of a black brittle material. Analyses checked well for a diamine (Table I). Conversion was 92.4%.

Ammonolysis of Octachloronaphthalene

Experiment 6 (360219) Octachloronaphthalene (Halowax 1051, 0.3 mole, 121.8 g) was combined with 7 moles of 28% NH_3 and heated for 3 hours at 250°C; over the next hour the temperature was gradually increased to 280°C and kept for 4 more hours at 280°C; maximum pressure was 1400 psi. The black reaction product was washed giving 99 g of material which was suspended in 1-liter of water. Filtration and drying

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gave only 0.1 g of solid. This indicates that the product must be somewhat soluble in water. The combined washes (2725 ml) contained 0.92 meq. of inorganic chloride. Based on this value, 6.6 chloro groups were replaced per molecule of octachloronaphthalene.

The isolated product melts above 311°C. The elemental analytical values are inconclusive, adding up only to 96.07%. The product failed to dissolve in the reagent used in the modified Cl analytical method H-8-57.

Anal. Found: C, 51.44, 51.55; H, 2.95, 3.02; Cl, 23.94, 24.61; N, 17.32

Calc'd.for $C_{10}Cl_4(NH_2)_4$: C, 36.8; H, 2.55; Cl, 43.6; N, 17.2

Calc'd.for $C_{10}Cl_2(NH_2)_6$: C, 41.9; H, 4.18; Cl, 24.8; N, 29.3

No definite conclusions can be drawn from these data, nor can the high carbon content be explained.

Reactions Between Aroclors and Various Amines

Reaction of Methylamine and Aroclor 1270

Experiment 7 (36-214) Aroclor 1270 (0.144 mole, 72 g) and 350 ml of methylamine was heated during 75 minutes to 144°C and kept for 3 more hours at this temperature; maximum pressure was 1800 psi. No indication of exothermic reaction was noted. The reaction product is completely soluble in 96% H_2SO_4, hence is free of unreacted Aroclor. It was washed with 400 ml. of water and dried at 70°C, giving 65 g of product, a 32.8% conversion. Analyses show that more than two (2.3) moles of methylamine were condensed with each mole of Aroclor 1270.

Anal. Calc'd. for Aroclor-Amine Product

	1:1 Ratio	1:2.3 Ratio	Found[a]
M. W.	495.59	482.5	
C	34.53	36.15	37.06
H	1.66	2.08	2.79
Cl	53.20	54.46	53.18[b]
N	5.76	7.27	7.22

[a] Average of 2 determinations.

[b] By the H-8-57 method.

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Reaction between Dimethylamine and Aroclor 1262

Experiment 8 (323123) Replacement of chlorine atoms in Aroclor by an amine was shown in 1955.

Aroclor 1262 (0.517 mole, 200 g) was placed in a cylindrical glass reactor (12" high x 3" i.d.), wrapped for electrical heating and fitted with a reflux condenser. The Aroclor was heated to 250-300°C and dimethylamine was passed in through a sintered glass disc at the bottom at a moderate rate for 1.5 hours. Amine hydrochloride salt was formed. The reaction mixture was filtered; the filtrate was flushed with N_2 at 100°C, and then heated to 200°C/0.5 mm. The residue was a mixture of unreacted Aroclor and the dimethylaminopolychlorobiphenyl. Based on N content, 33.3% of the Aroclor charged was converted to the diamine.

Anal. Found: C, 40.14, 40.17; H, 1.92, 2.00; N, 2.26, 2.36; Cl, 56.25, 56.29.

Reaction between Ethanolamine and Aroclor 1270

Experiment 9 (360225-7) A mixture of Aroclor 1270 (0.242 mole, 120 g) and ethanolamine (10 moles, 610 g, Eastman, white label) was stirred under reflux at 170-2°C for 6 hours. After 1.5 hours all Aroclor went in solution. No precipitation of reaction product occurred on cooling. The excess ethanolamine was distilled off at 140°C/18 mm (bath temperature). The residue was washed with stirring at 80°C with 3 x 300 ml portions of water, dried and kept at 160°C/1 mm under good stirring to collect 139 g of product. Analyses shows that 2 moles of ethanolamine reacted with each mole of Aroclor 1270. Reaction took place selectively between the amine nitrogen and the Aroclor molecule, as shown by the found OH values.

Anal. Calc'd. $C_{12}H_{10}Cl_8O_2N_2$ (M.W. 548): C, 35.0; H, 2.19; Cl, 51.7; N, 5.11; OH, 6.22

Found: C, 36.05; H, 2.7; Cl, 48.15, 48.27; N, 5.91, 6.0[illegible]; OH, 7.04, 7.05.

Analyses are for unrecrystallized material, which may contain traces of unreacted ethanolamine or trisubstituted product. Conversion is slightly over theory (104.9%).

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Reaction between Diethanolamine and Aroclor 1270

Experiment 10 (360234-6) Aroclor 1270 (0.242 mole, 121 g) was stirred with diethanolamine (7 moles, 736 g) and heated to 210°C over 1.25 hours. After three more hours at 210°C, all Aroclor had dissolved. At this point more Aroclor (24.2 g) was added and heated for 5 more hours at 210°C. The added Aroclor dissolved after 1.3 hours. Most of the excess diethanolamine was distilled off at 170°C/1-2 mm. No decomposition in low boilers occurred during the condensation step, as shown by a 98.5% material recovery. The distillation residue (388 g), still containing diethanolamine, was very soluble in ethanol, ethanol-water mixtures, and other common solvents. A 375 g portion of the distillation residue was dissolved in 750 g of absolute ethanol. To this solution was added 0.5 mole NaOH as 7.5 N solution; sodium chloride precipitated. This solution was added dropwise to a large excess of water (final volume ratio 1:20) and 108 g of a white solid, mp 142°C, was collected. This white material is very soluble in acetone, methanol, aqueous ethanol, etc. It is also somewhat water-soluble, explaining the low recovery (only 69.8%). No unreacted Aroclor was found.

Anal.	Calc'd. for $C_{20}H_{20}Cl_8N_2O_4$. MW 636	Found	
C	37.9	37.7	
H	3.15	3.03	
Cl[a]	44.60	45.22	45.26
N	4.39	4.07	4.09
OH	10.69	10.45	10.50

[a] by H-8-57 method

Reaction between Diethylenetriamine and Aroclor 1270

Experiment 11 (360232-3) Aroclor 1270 (120 g) was combined with diethylenetriamine (8 moles, 829 g) and stirred with heating. Since the Aroclor dissolved (or reacted) rapidly at 140°C, three additional 60 g portions of Aroclor were added. All went into solution immediately. Total Aroclor charged was 0.605 mole. This mixture was heated for 2 more hours at 150°C, and for an additional hour at 180-190°C, during which the solution turned somewhat darker. Excess amine was distilled off at 1-2 mm. The liquid residue (494 g, n_D^{25} 1.4833) was stirred at 90°C with water and separated by decantation. The residue was

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[illegible] yellow powder, [illegible] g, was obtained. No unreacted Aroclor was isolated. Elemental analysis indicate that between 2 and 3 diethylenetriamine moles reacted with each mole of Aroclor 1270. Unfortunately, the modified H-8-57 Cl method could not be used. The reported Cl values are probably low.

Anal.	X Calc'd. 1:2	Y Calc'd. 1:3	Found
C	38.1	41.40	40.00 - 40.33
H	3.84	5.18	5.05 - 5.25
Cl	44.80	35.32	35.52 - 35.54
N	13.32	18.10	16.72 - 16.84

X = MW 629.84, $C_{20}H_{24.05}Cl_{7.05}N_6$

Y = MW [illegible], $C_{24}H_{36.05}Cl_{[illegible]}N_{[illegible]}$

The sum of the found values is only [illegible]. This is due to the method used for Cl. The conversion found is equal to [illegible]% calculated as diamine and [illegible]% as triamine. This is additional supporting evidence that more than two moles of the amine reacted with Aroclor 1270.

Reaction between Aniline and Aroclor 1270

Experiment 12 (360237) A mixture consisting of Aroclor 1270 (0.293 mole, 145.3 g) and aniline (5 moles, 465 g of freshly distilled material) was stirred under reflux at 185°C. After 1.75 hours of reflux, all went in solution. As soon as the heating mantle was removed, precipitation of Aroclor started, indicating very slow reaction. The mixture then was refluxed for a total of 46 hours, cooled, and filtered. The precipitate was washed with benzene, giving 28 g (0.056 mole) of unreacted Aroclor. The filtrate was diluted with 400 ml. of water. 0.6 mole of 30% NaOH was added and the precipitate was washed several times. The wash water (935 ml) contained a total of 0.15 mole of Cl derived from Aroclor. The aniline was distilled off to isolate a semi-solid, dark product. It was not further purified since the chlorine found in the wash water indicated very incomplete reaction.

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PATENTS

Five patent disclosures were submitted as a result of this specific work on Aroclor chemistry.

D-2005 J. Dazzi. Amination of chlorinated biphenyls. Example using dimethylamine and Aroclor 1262 at atmospheric pressure; 10-31-55.

D-2467 J. Dazzi. Ammonolysis of Aroclor 1270, 1268, and 1262; 6-25-57.

D-2468 J. Dazzi. Ammonolysis of octachloronaphthalene to tetra and higher amines; 6-25-57.

D-2658 J. Dazzi. Reaction between Aroclor and diethylene-triamine, new compound, adding 2 or more moles of the amine; 4-2-58.

D-2659 J. Dazzi. Reaction between Aroclor 1271 and mono- and diethanolamine to yield the corresponding N,N'-β-hydroxyethyldiaminotetrachlorobiphenyl; 4-2-58.

No literature and patent search was made on compounds given in this report. Patents as new compositions of matter will require definition of applicability of these compounds. Suggestions are given in Section IV.

DSW 621532

STLCOPCB4095563

REFERENCES

1. Dazzi, J., "New Organic Chlorine (Aroclor) Derivatives" Memo to the Idea Review Committee, dated 4/26/55.

2. Dazzi, J., "The Aroclors and Other Halogenated Aromatics as Chemical Intermediates" Memo to Dr. C. A. Hochwalt, Part I; dated 1/16/56. Continuation, Part III, "Nitrogen Containing Compounds", dated 11/7/57.

3. Throdahl-Zienty, Memo to M. Kosmin, dated 10/10/57.

4. Younger Laboratories, St. Louis, reported by Elmer P. Wheeler to J. Dazzi, 7/15/57.

5. Rocklin, A. L., J. Org. Chem. 21, 1478-80 (1956) "Substitution Reaction of Hexachlorobenzene"

6. Roomalen, F. C., Rev. Trav. Chem. 53 359-79 (1934).

DSW 621533

STLCOPCB4095564

In toxicity tests, exclusive of carcinogenic acitivity, Aroclor 1270-ammonia product (Expt. 2) was classed as a mild irritant with most of the irritation being mechanical (R-4). A carcinogenic test on a compound such as this ammonolysis product of Aroclor 1270 would cost Monsanto a minimum of $25,000 as stated in R-3. Even then, according to this memo, the data can not be regarded as reliable.

A dermatitis which resisted treatment was the consequence of the writer's exposure over a long period of time to the Aroclors, to octachloronaphthalene, or their derivatives; the octachloronaphthalene is the most likely causative agent.

DSW 621534

STLCOPCB4095565

Carbon and hydrogen combustion were run at 800°C. Nitrogen was determined by the Dumas method.

Chlorine analyses were run by the H-8-57 method if the compound was soluble in the reagent; otherwise, the determination was made by the C-35-55 method (peroxide). This always gives low values for compounds high in chlorine such as Aroclor 1268-71 and their derivatives. The molecular weight of Aroclor 1270 is taken as 496.5 based on 71% Cl found as the average of 5 determinations, using the H-8-57 method (see NBP 360244).

We thank the various groups in Dayton for elemental and infrared analyses.

DSW 621535

STLCOPCB4095566

APPENDIX A. NOTEBOOK PAGE REFERENCES

323123	360217
323124	360218
360206	360219
360207	360220
360210	360225
360211	360226
360212	360227
360213	360230-37
360214	360239
360215	360244

APPENDIX B - Detailed List of Experiments

	Expt. No.
1) Ammonolysis of Aroclors	
Aroclor 1270	1
Aroclor 1270	2
Aroclor 1270	3
Aroclor 1268	4
Aroclor 1262	5
2) Ammonolysis of Octachloronaphthalene	6
3) Reactions Between Aroclor 1270 and Various Amines	
Methylamine	7
Dimethylamine (Aroclor 1262)	8
Ethanolamine	9
Diethanolamine	10
Diethylenetriamine	11
Aniline	12

DSW 621536

STLCOPCB4095567

APPENDIX C

TABLE I

ANALYSIS OF AMMONOLYSIS PRODUCTS OF

AROCLOR 1270, 1268, 1262

Aroclor	1270			1268		1262	
MW. Calc'd. as diamine	457.63	457.63		414.74		348.38	
Expt. No.	1	2		4		5	
Anal.[a]	Calc'd	Found	Found	Calc'd	Found	Calc'd	Found
C	31.4	31.7	33.91	34.76	35.90	41.37	[illegible]
H	0.88	1.21	1.56	1.28	1.31	2.12	[illegible]
Cl[b]	62.00	60.87	53.81[c]	57.48	56.80[b]	48.60	[illegible]
N	6.12	6.60	8.66	6.76	6.95	8.04	[illegible]

a. based on 2 determinations

b. H-8-57 method

c. C-35-55 method

DSW 621537

STLCOPCB4095568