

**Table V. Effect of Granule Drying Time on P<sub>2</sub>O<sub>5</sub> Availability of Cured Product<sup>a</sup>**

Drying Time, <sup>b</sup> Min.	Moisture Content of Granules After Drying, %	Composition of Cured Product, <sup>c</sup> %				P <sub>2</sub> O <sub>5</sub> Availability, %	
		P <sub>2</sub> O <sub>5</sub>					
		Total	A.P.A.	W.S.	F.A.		H <sub>2</sub> O
15	2.9	22.3	21.5	19.3	1.2	1.5	96
4	4.8	21.7	21.2	19.5	1.1	1.9	98
0	5.8	22.2	21.9	19.8	0.7	1.2	99

<sup>a</sup> High-grade rock; 96% H<sub>2</sub>SO<sub>4</sub>.

<sup>b</sup> With warm (100° to 150° F.) air.

<sup>c</sup> Cured 3 to 7 weeks.

with either 96% acid and finely ground rock or with fuming acid and a standard grind of rock. It was necessary to use slightly more acid with the latter combination. It has been estimated that fine grinding of rock to the size desired for this process would increase the cost of the rock by about 40 cents per ton or about 25 cents per ton of superphosphate. At published prices, the use of a higher acidulation with fuming acid and the standard grind of rock would increase production cost approximately the same amount. Thus, the choice between the two might depend on the cost of acid at the particular plant location.

The process has the following advantages over the usual method for granulating superphosphate. The usual acid dilution equipment is eliminated, as well as the cutter commonly used with mechanical dens. The superphosphate is

granulated before curing, thereby eliminating the extra handling involved in digging cured superphosphate from the pile, transferring to the granulation area, and grinding prior to granulation. The cost of drying the granulated superphosphate is reduced or eliminated. Any surface drying required to facilitate screening or reduce caking in the pile can be accomplished by short contact with warm air, possibly within the granulating drum. Although little or no drying is required, the moisture content of the product is relatively low and it is therefore easier to make standard 0-20-0 from low-grade rock. The evolution of fluorine is increased by twofold or more, thereby improving the economics of fluorine recovery.

Although the procedures were designed to simulate commercial operation, a larger scale investigation of the process appears warranted. Points on which

further information is needed include the type of mixer best suited to mixing of strong acid and rock, granulation efficiency obtainable in larger equipment, the degree of surface drying, if any, needed to prevent caking in the pile, and ammoniation characteristics of the product. Four to 5 pounds of ammonia per unit of P<sub>2</sub>O<sub>5</sub> were held in small-scale tests.

The process should be adaptable to existing superphosphate granulation plants with little change in equipment, assuming that the rock-acid mixer is suitable. Standard dens (perhaps with cutter eliminated) should be appropriate for the dry, semigranular acidulate, and the rotary drum commonly used in granulation plants should be suitable for granulation of the acidulate.

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## ISOTOPE-LABELED INSECTICIDES

### The Synthesis of Tertiary Carbon Deuterated DDT and DDT Analogs

TO UTILIZE one of several contemplated experimental approaches to the testing of a new chemical hypothesis (10) on the toxic action of certain contact insecticides, it was necessary to prepare the deuterated analogs of some of these. The reactivity of DDT under certain conditions which ordinarily favor free radical reactions had led the authors to believe that the toxic action of DDT might be the result of an in vivo free radical reaction of DDT at the susceptible site.

The nature of several reported metabolites of DDT, viz., DDE (27), *p,p'*-dichlorobenzophenone (14), *p,p'*-dichlorobenzohydroxy (20), and Kelthane (14, 22) suggests that detoxication proceeds

via enzymatic processes which may also be free radical in type.

The first two of these metabolites have been obtained in this laboratory by the bromine-sensitized photoelimination and autooxidation reaction.

Barker (2) reacted deuteriochloral and 1-deuteriodichloroacetaldehyde with chlorobenzene to prepare 1,1,1-trichloro-2,2-bis(*p*-chlorophenyl)ethane-2-d (d-DDT, d-I) and 1,1-dichloro-2,2-bis(*p*-chlorophenyl)ethane-2-d (d-DDT, d-I) and 1,1-dichloro-2,2-bis(*p*-chlorophenyl)ethane-2-d (d-TDE, d-II), respectively.

Among the products of the sulfuric acid condensation of chloral and chlorobenzene is the compound, *p*-chloro-

$\alpha$ -trichloromethylbenzyl alcohol (IA), which is probably an intermediate in the formation of I (3). The preparation of the 1-d analog of this compound by the reduction of 2,2,2,4'-tetrachloroacetophenone (IK) with lithium aluminum deuteride to yield d-IA appeared feasible in the light of the successful reduction of hexachloroacetone to 1,1,1,3,3,3-hexachloro-2-propanol (8) and of 2-fluoro-2,2,4'-trichloroacetophenone to *p*-chloro- $\alpha$ -(dichlorofluoromethyl)benzyl alcohol (5).

Chlorination of *p*-chloroacetophenone gave IK and 2,2,4'-trichloroacetophenone (IIK). A model reduction with lithium aluminum hydride of IK and of IIK gave IA and IIA, respectively, in

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The syntheses of 1,1,1-trichloro-2,2-bis(*p*-chlorophenyl)ethane-2-d(d-DDT,d-I); 1,1-dichloro-2,2-bis(*p*-chlorophenyl)ethane-2-d(d-TDE,d-II); 1,1,1-trichloro-2,2-bis(*p*-bromophenyl)ethane-2-d(d-DBrDT, d-III); and 1,1,1-trichloro-2,2-bis(*p*-methoxyphenyl)ethane-2-d(d-methoxychlor, d-IV) are described. A new synthesis of 2,2-dimethyl-1,1-bis(*p*-methoxyphenyl)propane (DANP, V) is described, and this synthesis is used to prepare 2,2-dimethyl-1,1-bis(*p*-methoxyphenyl)propane-1-d (d-V).

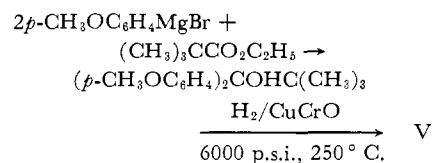
good yields. IA was also synthesized by an alternate route from *p*-chlorobenzaldehyde and chloroform (4). The two samples of IA had identical infrared spectra.

With chlorobenzene, IA yielded I in the presence of sulfuric acid (12). IK was reduced by lithium aluminum deuteride to d-IA which was then converted to d-I.

The absence of a significant quantity of deuterium on the aromatic rings was suggested by the exact identity of the infrared spectra of samples of 1,1-dichloro-2,2-bis(*p*-chlorophenyl)ethylene (DDE, VII) prepared from I and d-I. The NMR spectra gave further support to the restriction of substitution by deuterium to the desired position.

Similar procedures were used to prepare d-II, 1,1,1-trichloro-2,2-bis(*p*-bromophenyl)ethane-2-d(d-DBrDT, d-III); 1,1,1-trichloro-2,2-bis(*p*-methoxyphenyl)ethane-2-d(d-methoxychlor, d-IV); and 2,2-dimethyl-1,1-bis(*p*-methoxyphenyl)propane-1-d(d-DANP, d-V). d-II was prepared by the reaction of 2,2,4'-trichloroacetophenone (IIK) with lithium aluminum deuteride and condensation of the resulting alcohol, d-IIA, with chlorobenzene. d-III was prepared by the reaction of *p*-bromophenyl magnesium bromide with chloral, and the resulting alcohol, IIIA, was oxidized to the ketone, IIK, which was converted to d-IIIA by lithium aluminum deuteride. This alcohol was then condensed with bromobenzene in the usual way. d-IV was prepared from 2,2,2-trichloro-4'-methoxyacetophenone (IVK) by reducing with lithium aluminum deuteride and condensing the resulting alcohol, d-IVA, with anisole.

V has been prepared by Rogers *et al.* (17) who used the following reaction sequence:



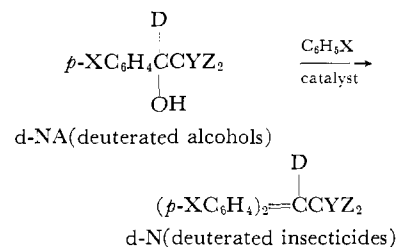
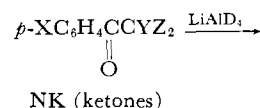
Deuteration at 6000 p.s.i. and 250° C. was believed almost surely to cause random exchange. The successful synthesis of d-IV led the authors to consider a similar route to d-V, viz., by the reaction of d-VA and anisole. It has been stated that this Friedel-Crafts step in the

synthesis would proceed necessarily through the carbonium ion. The implication was then made that since neopentyl carbonium ion yields predominantly rearranged products, this synthetic route is not feasible (17). Neither Rogers *et al.* nor Skerret and Woodcock (19) could secure 1,1-di(*p*-chlorophenyl)-2,2-dimethylpropane by this route. No attempt, however, seems to have been made to synthesize V in this way.

The rather considerable difference in the polar effects of the *p*-chlorophenyl and *p*-anisyl groups seemed to invalidate the implied analogy. Success was anticipated on either of two possible grounds—the anisylneopentyl carbonium ion, if formed, might not rearrange before its electrophilic substitution on anisole, or the anisylneopentyl carbonium ion might not be the reacting form of the alcohol but that the reaction might proceed by an S<sub>N</sub>2 mechanism (6).

Because of the success of others (15) in alkylating without rearrangement by using aluminum chloride or ferric chloride, these catalysts were employed. Chance favored the use of aluminum chloride in that the concentrations and conditions chosen were favorable to the formation of V. In subsequent experiments, under different conditions and/or concentrations, rearrangement was demonstrated, not however of the anisylneopentyl carbonium ion but of V itself, presumably through the dianisylneopentyl carbonium ion as has been predicted by Rogers *et al.* (17). The product of this rearrangement was 2,2-dianisyl-3-methylbutane (VI). V appears to be the kinetically favored and VI the thermodynamically favored product of the reaction. This is interesting because in a related situation, viz., that of the alkylation of benzene by 2-chloro-2,3-dimethylbutane, the thermodynamically less stable isomer, 2,3-dimethyl-2-phenylbutane, has been observed by Schmerling and West (18) to rearrange to 2,2-dimethyl-3-phenylbutane.

The use of ferric chloride as the catalyst was not plagued by the tendency toward rearrangement. The general synthetic scheme for the final steps in the synthesis of all five tertiary deuterated insecticides may be represented as follows:



N	X	Y	Z
I	Cl	Cl	Cl
II	Cl	H	Cl
III	Br	Cl	Cl
IV	OCH <sub>3</sub>	Cl	Cl
V	OCH <sub>3</sub>	CH <sub>3</sub>	CH <sub>3</sub>

The physical appearance of each insecticide and its deuterated analog was identical. Mixed melting points showed no depression. Infrared spectra of 5% solutions in carbon disulfide and using the 0.1-mm. cell showed the following differentiating bands for the insecticides and their deuterated analogs. I: 11.23 μ, 11.77 μ, 12.40 μ; d-I: 10.51 μ, 11.86 μ, 12.64 μ; II: 11.36 μ, 11.77 μ, 12.50 μ; d-II: 10.17 μ, 11.10 μ, 11.94 μ, 12.15 μ, 12.77 μ; III: 11.25 μ, 11.78 μ, 12.45 μ; d-III: 10.53 μ, 11.88 μ; IV: 7.83 μ, 11.22 μ, 11.76 μ; d-IV: 10.56 μ, 12.42 μ, 12.64 μ; V: 7.93 μ, 12.86 μ; d-V: 7.75 μ, 13.04 μ, 13.26 μ.

Infrared spectra of 20% solutions of d-DDT, d-BrDT, and d-methoxy-chlor using calcium fluoride optics showed absorption at 4.6 μ where no absorption was observed for DDT, DBrDT, and methoxychlor. On the other hand, DDT and DBrDT showed a sharp absorption at 3.4 μ which was missing in the spectra of the deuterated insecticides. Methoxychlor, because of the methoxy group, gave strong absorption in the 3.2-μ to 3.5-μ region for both the deuterated and nondeuterated forms.

Infrared spectra of the intermediate alcohols and their deuterated analogs also showed differentiating bands. A 0.1-mm. cell containing a 5% solution of the alcohol in carbon disulfide was used with the following results: IA: 8.25 μ, 9.27 μ, 11.08 μ, 12.25 μ; d-IA: 7.48 μ, 10.38 μ, 10.58 μ, 11.34 μ; 2,2-dichloro-1-*p*-chlorophenylethanol (IIA): 7.25 μ, 9.50 μ, 10.62 μ, 11.69 μ, 12.14 μ; d-IIA: 7.51 μ, 8.82 μ, 9.70 μ, 11.13 μ, 12.00 μ; IIIA: 7.70 μ, 11.62 μ; d-IIIA: 7.51 μ, 9.16 μ, 10.58 μ, 11.85 μ; IVA: 9.33 μ, 10.11 μ, 11.58 μ, 12.58 μ; d-IVA:

9.16  $\mu$ , 10.39  $\mu$ , 10.60  $\mu$ , 11.88  $\mu$ ; 2,2 - dimethyl - 1 - *p* - methoxyphenylpropanol-1 (VA): 7.84  $\mu$ , 11.12  $\mu$ , 13.05  $\mu$ ; d-VA: 9.36  $\mu$ , 11.70  $\mu$ , 12.25  $\mu$ .

NMR spectra taken and analyzed by Varian Associates, Palo Alto, Calif., on I, IV, and V, and their respective deuterated analogs showed the deuterium to be in the position assigned to the extent of 98% for d-I and d-IV and not less than 95% for d-V. No NMR spectra were made of II and III, but their infrared spectra and behavior in reactions in which the isotope effect was measured are considered to be satisfactory evidence that they too had the expected deuterium content at the tertiary aliphatic position. The isotope rate effect in free radical reactions of d-I, d-III, and d-IV was investigated. The results of this investigation will be published separately. Insect toxicological and pharmacological studies on the deuterated compounds by entomologists who are collaborating in this study will also be published separately.

### Experimental

All infrared determinations were made on the Perkin-Elmer Model 21 spectrophotometer. Melting points were taken on the Fisher-Johns Melting Point Apparatus and are uncorrected.

***p*-Chloroacetophenone.** This compound was synthesized by the procedure of Adams and Noller (7).

**2,2,2,4' - Tetrachloroacetophenone (IK).** This compound was prepared by the procedure of Barry (3).

***p*-Chloro- $\alpha$ -trichloromethylbenzyl Alcohol (IA).** PROCEDURE A. A solution of IK (10 grams, 0.04 mole) dissolved in 25 ml. of anhydrous ether was slowly added to a solution of lithium aluminum hydride (0.5 gram, 0.013 mole) in 75 ml. of anhydrous ether. Spontaneous refluxing occurred at first, after which the mixture was gently refluxed for 3 hours. The complex was decomposed by adding 15 ml. of water and 20 ml. of 10% sulfuric acid with stirring continued until both layers were clear. The ether layer was then separated and evaporated after washing with water and drying over potassium carbonate.

PROCEDURE B. IA was also prepared by the procedure of Bergmann *et al.* (4).

***p*-Chloro- $\alpha$ -trichloromethylbenzyl- $\alpha$ -d Alcohol (d-IA).** This compound was prepared following the Procedure A for IA, except that lithium aluminum deuteride (Metal Hydrides Inc., Beverly, Mass.) was used.

**1,1,1 - Trichloro - 2,2 - bis(*p*-chlorophenyl)ethane-2-d (d-I).** To 5 grams (0.019 mole) of d-IA and 9.5 grams (0.084 mole) of chlorobenzene was added 70 grams of concentrated sulfuric acid and 5 grams of 15% fuming sulfuric acid, and the mixture was shaken for 1

hour. The oily layer was separated from the acid and dissolved immediately in 95% ethanol, from which d-I crystallized. Recrystallization from 95% ethanol afforded the product (50 to 70%), m.p. 108–109.5° C. A mixed melting point with I gave no depression.

**2,2,4'-Trichloroacetophenone (IIK).** Chlorine was passed into 116 grams (0.75 mole) of *p*-chloroacetophenone at 60° C. with stirring until there was a weight increase of 35 grams at which time a considerable amount of solid was present. The temperature was raised to 100° C. and chlorination continued until an additional 17 grams increase in weight was registered. The reaction mixture was stirred with 1500 ml. of warm petroleum ether (30° to 60° C.), and the supernatant extract was separated and chilled overnight. The residue was retained for further extraction. The mother liquor was poured off the crystalline solid and was used to extract the residue which had been reserved for this purpose. This extract also deposited crystals on standing in the refrigerator. The combined crystalline products were recrystallized from *n*-hexane to give 112 grams of product (68%), m.p. 58–59° C.

***p*-Chloro- $\alpha$ -dichloromethylbenzyl- $\alpha$ -d Alcohol (d-IIA).** The procedure was identical with that used for the preparation of d-I using IIK in place of IK.

**1,1 - Dichloro - 2,2 - bis(*p*-chlorophenyl)ethane-2-d (d-II).** The procedure which employed d-IIA was the same as that used for the preparation of d-I except that after removal of the sulfuric acid, the pasty upper layer was triturated with 25 ml. of 75% ethanol. The product, collected by filtration, washed with a small amount of 75% ethanol was recrystallized from 95% ethanol. Yield, 3 grams (53%); m.p. 110° C. A mixed melting point with authentic II gave no depression.

***p*-Bromo- $\alpha$ -trichloromethylbenzyl Alcohol (IIIA).** *p*-Bromophenyl magnesium bromide was prepared from 118 grams (0.5 mole) of *p*-dibromobenzene using the procedure of Evans and Duffard (7). The slow addition of 73 grams (0.5 mole) of chloral was followed by stirring overnight after which the reaction product was worked up in the usual manner. Distillation of the residue after removal of the ether yielded a fraction distilling from 120° to 170° C. (3 mm.). Redistillation yielded 32 grams of product distilling at 142° C. (3 mm.).

**2,2,2 - Trichloro - 4' - bromoacetophenone (IIIK).** To a warm solution composed of 19 grams (0.065 mole) of potassium dichromate, 85 ml. of water, 55 ml. of acetic acid, and 13 grams (0.13 mole) of sulfuric acid was added 22 grams (0.07 mole) of IIIA, and the mixture was refluxed with stirring for 4 hours. The ether extract of the cooled reaction mixture was washed

successively with water, saturated aqueous sodium bicarbonate, and saturated aqueous sodium chloride, then dried over sodium sulfate. The product after removal of the ether distilled at 132° C. (4 mm.). Yield, 15 grams (70%); m.p. 13° C.

***p*-Bromo- $\alpha$ -trichloromethylbenzyl- $\alpha$ -d Alcohol (d-IIIA).** The procedure was identical with that used for the preparation of d-IA using IIIK in place of IK.

**1,1,1 - Trichloro - 2,2 - bis(*p*-bromophenyl)ethane (III).** The procedure was identical with that used for the preparation of I from IA and chlorobenzene with equivalent amounts of IIIA and bromobenzene being used in this case. Yield, 78%, m.p. 145° C.

**1,1,1 - Trichloro - 2,2 - bis(*p*-bromophenyl)ethane-2-d (d-III).** The procedure is identical with that for III except that d-IIIA is employed in place of IIIA. Yield, 84%, m.p. 144–146° C. A mixed melting point with III showed no depression.

**Trichloroacetonitrile.** The procedure of McBee and Pierce (13) was followed.

**2,2,2 - Trichloro - 4' - methoxyacetophenone (IVK).** The procedure of Houben and Fischer (17) was followed. These authors reported a 70% yield, whereas the present authors were unable to obtain better than 35%.

***p*-Methoxy- $\alpha$ -trichloromethylbenzyl Alcohol (IVA).** This compound was prepared from the corresponding ketone, IVK, in the same manner as IA following Procedure A. B.p. 160–163° C. (3–5 mm.).

***p*-Methoxy- $\alpha$ -trichloromethylbenzyl- $\alpha$ -d Alcohol (d-IVA).** Procedure A used in preparing IVA was followed except that lithium aluminum deuteride was used.

**1,1,1 - Trichloro - 2,2 - bis(*p*-methoxyphenyl)ethane-2-d (d-IV).** To a mixture of d-IVA (2 gram, 0.008 mole) and anisole (0.92 gram, 0.009 mole) in 2.2 ml. of glacial acetic acid, 4.4 ml. of 98% sulfuric acid was slowly added, taking care that the temperature did not rise above 45° C. The mixture was stirred for 40 minutes. The acid was then removed, and the violet gummy residue was triturated in a mortar under cold 70% ethanol until it became a fine white powder. This was then recrystallized from 95% ethanol. Yield, 70%, m.p. 85–86° C. A mixed melting point with IV showed no depression.

**2,2 - Dimethyl - 1 - *p* - methoxyphenylpropanol-1 (VA).** *t*-Butyl magnesium chloride was prepared from 165.6 grams (1.8 mole) of *t*-butylchloride and 36.5 grams (1.5 mole) of magnesium turnings in absolute ether (16). The mixture was cooled and 72 grams (0.53 mole) of anisaldehyde in ether was added slowly. After standing overnight, the mixture was decomposed with ice and

dilute sulfuric acid, filtered, and the ether layer removed. The ether solution was successively washed with water, sodium carbonate solution, water, and saturated sodium chloride solution, and then dried over sodium sulfate. After removing the ether, the product distilled at 117–120° C. (3 mm.). It was a pale yellow liquid which crystallized on standing. Yield, 80.3 grams (76%), m.p. 38–39° C. from *n*-hexane.

***t*-Butyl-*p*-methoxyphenyl Ketone (VK).** A cold solution of 21 grams (0.08 mole) of sodium dichromate in 100 ml. of water and 17.5 grams (0.17 mole) of concentrated sulfuric acid was allowed to drop slowly upon 19.4 grams (0.10 mole) of melted VA while stirring. The temperature rose to 45° C. and was then maintained at 35° to 45° C. After addition of the dichromate solution was complete, the reaction mixture was stirred for 4 hours and then left standing overnight. The mixture was extracted with ether, and the ether extract was washed successively with water, sodium bicarbonate solution, water, and saturated sodium chloride solution, and then dried over sodium sulfate. The product, after removal of the ether, distilled at 104–107° C. (0.5 mm.). Yield, 13.1 grams (68%). Semicarbazide, m.p. 152–154° C.

**2,2 - Dimethyl - 1 - *p* - methoxyphenylpropanol-1-d (d-VA).** Using VK, the procedure was identical with that used for the preparation of d-IA.

**2,2 - Dimethyl - 1,1 - bis(*p* - methoxyphenyl)propane (V).** PROCEDURE A. To a cold solution of 5 grams (0.03 mole) of VA and 15 grams (0.14 mole) of anisole in a test tube was added 2 grams (0.015 mole) of anhydrous aluminum chloride in small portions with thorough cooling and stirring. After the addition was complete, the test tube was closed with a drying tube and allowed to warm up to room temperature overnight (about 13 hours). The reaction mixture was decomposed with ice and hydrochloric acid and extracted with ether. The ether extract was washed successively with water, sodium bicarbonate solution, and water. After removal of the ether, the excess anisole was removed by steam distillation. The residue was washed with cold methanol and then crystallized from methanol. Yield, 3.8 grams (52%), m.p. 60–61° C.

PROCEDURE B. The above procedure was repeated using 5 grams (0.03 mole) of ferric chloride as catalyst. The reaction mixture, dissolved in five volumes of benzene was repeatedly washed with 30 ml. portions of 3*N* hydrochloric acid and then with water and 3% ammonia.

The excess anisole was removed by steam distillation. The residue was taken up in petroleum ether (30–60° C.), dried over calcium chloride, treated with norite, and filtered. The filtrate was passed through a 20 × 0.7 cm. column of activated alumina, the eluate allowed to evaporate and the nearly colorless crystalline residue weighing 4.1 grams recrystallized from methanol. Yield, 3.5 grams (48%); m.p. 59–60° C. The melting point of mixtures of V with an authentic sample (furnished by E. F. Rogers, Merck & Co., Rahway, N.J.) was not depressed. The infrared spectrum of V was identical with that of the authentic sample.

**2,2 - Dimethyl - 1,1 - bis(*p* - methoxyphenyl)propane - 1 - d (d-V).** Using d-VA, the procedure was identical with Procedure A used in preparing V.

**2,2-Dianisyl-3-methylbutane (VI).** PROCEDURE A. The procedure was identical with that for the preparation of V, except that 4.0 grams (0.03 mole) of aluminum chloride were used. The white crystalline product melted at 101.5–103° C. and had an infrared spectrum identical with that of a sample prepared by Rogers *et al.* (17). When the reaction was allowed to proceed for only 1/2 hour at room temperature, V was isolated in 44% yield. When 3.0 grams of catalyst with 15 grams of anisole or 2.0 grams of catalyst with only 5 grams of anisole were used, both V and VI were obtained by fractional crystallization from methanol.

PROCEDURE B. To a thoroughly cooled solution of 1 gram (0.0035 mole) of V in 3 grams (0.028 mole) of anisole, 1 gram (0.008 mole) of anhydrous aluminum chloride was added with stirring. The mixture warmed to room temperature overnight and was then worked up as in the procedure for preparing V. The only product isolated was 0.5 gram of white crystals, m.p. 101.5–102° C., identical with the product obtained by Procedure A.

**1,1 - Dichloro - 2,2 - bis(*p* - chlorophenyl)ethylene (VIII).** The general procedure of Harris and Frankforter (9) was used on I and d-I.

**Alkaline Hydrolysis of IK.** A mixture of 5 grams (0.02 mole) of 1K and 16 ml. of 10% sodium hydroxide was heated in a distilling flask on a steam bath. The distillate, dried with a few granules of calcium chloride, weighed 2.2 grams, boiled at 61° C., and had the typical odor of chloroform. The alkaline residue was acidified, and the precipitate which weighed 2.7 grams was recrystallized from ethanol, m.p. 235° C. (uncorr.), neutral equivalent 155.

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