

In an attempt to prepare XII from *trans*-IIIa by the action of 50–4 ml. acetic anhydride–concd. sulfuric acid mixture at room temperature for 16 hr., 82% of starting material was recovered.

**4-Bromo-2-acetoxy-2,5-diphenyl-3-furanone (XVI).**—A solution of 1.5 g. of IIb in 35 ml. of acetic anhydride and 3 ml. of concd. sulfuric acid, after standing for 24 hr. at room temperature and cooling, deposited crystals; recrystallized from absolute ethanol, 0.61 g. (43%), m.p. 185.5–187.5°; ultraviolet absorption,  $\lambda_{\text{max}}$  250, 322 m $\mu$ ;  $\epsilon$  8,080, 15,030.

*Anal.* Calcd. for  $\text{C}_{18}\text{H}_{13}\text{BrO}_4$ : C, 57.93; H, 3.51. Found: C, 57.96; H, 3.48.

**2,5-Diethoxy-2,3,4,5-tetraphenyl-2,5-dihydrofuran (IVcd)** was prepared like IVad; crystallized from absolute ethanol, 5.6 g. (47%), m.p. 156.5–157.5°; ultraviolet absorption  $\lambda_{\text{max}}$  260 m $\mu$ ,  $\epsilon$  15,520.

*Anal.* Calcd. for  $\text{C}_{32}\text{H}_{30}\text{O}_2$ : C, 83.08; H, 6.54. Found: C, 82.95; H, 6.30.

**Hydrolysis** of 0.5 g. by 60 ml. of 80% ethanol and 5 ml. of concd. hydrochloric acid, refluxing for 6 hr., processing as with IVad, gave 0.2 g. (48%) of IIIc. **Reductive condi-**

**tions**, zinc dust and boiling acetic acid for 2 min., converted IVcd to tetraphenylfuran (62%); IIIc under these conditions also gave tetraphenylfuran (75%). Acetic acid, boiling for 2 min., converted IVcd to IIIc (95%).

**Treatment of *cis*-dibenzoylstilbene (IIIc) with acetic anhydride concd. sulfuric acid mixture (25–1 ml.) for 24 hr. at room temperature, followed by hydrolysis and processing, gave 87% of starting material.**

**1-Chloro-2-(*N*-methylanilino)-1,2-dibenzoyl ethylene (XI).**—A solution of 2 g. of *cis*-IIIa and 2.8 g. of methylaniline in 50 ml. of dry ether reacted over 24 hr. at room temperature with deposition of methylaniline hydrochloride (filtered). After washing with water, drying over sodium sulfate and vac. evaporating, the resulting oil was crystallized from absolute ethanol; yield 0.88 g. (36%), orange crystals, m.p. 137–138°.

*Anal.* Calcd. for  $\text{C}_{23}\text{H}_{18}\text{ClNO}_2$ : C, 73.50; H, 4.83. Found: C, 73.65; H, 5.13.

*trans*-IIIa did not react under the above conditions, recovery 84%.

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## The Reaction of Olefins with Aromatic Substances in the Presence of Mercury Salts and Catalyst. III. Reaction of Various Olefins with Anisole

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RECEIVED AUGUST 4, 1958

The formation of  $\beta$ -arylethyl acetates and/or diarylethanes by the action of ethylenemercury salt addition compounds on aromatics in the presence of alkylating catalysts has been extended successfully to other olefins, *i.e.*, propylene, 2-butene and styrene. Structural determination of intermediate mercurials and final products shows that this reaction is of the  $\text{S}_{\text{N}}2$  type. Cyclohexene, a typical cycloolefin unlike other olefins reacts slowly with anisole to give methoxyphenylcyclohexene and not the expected *p*-methoxyphenylcyclohexyl acetate.

A new method of synthesis of  $\beta$ -arylethyl acetates and *sym*-diarylethanes by reaction of ethylene with aromatic compounds in the presence of mercury salts and catalyst was reported recently.<sup>2</sup>

It was established that this reaction proceeds through the intermediate mercurial  $\text{ArCH}_2\text{CH}_2\text{HgZ}$  ( $\text{Z}$  = acid radical) which is formed by the reaction of an ethylene–mercury salt complex compound with the aromatic.

Several features of the mechanism, however, remained unsettled and required further investigation.<sup>3</sup> For example, it was not determined whether the reaction of this complex with aromatics is a carbonium ion reaction or whether the aromatic participates directly in an  $\text{S}_{\text{N}}2$  type displacement on the complex.

This paper reports results of the application of this reaction to other olefins such as propylene, 2-butene, styrene and cyclohexene and discusses further, by bringing new experimental evidence to bear, the question of the mechanism mentioned above.

The results obtained are summarized in Table I.

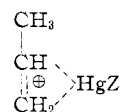
(1) Institute for Chemical Research, Kyoto University, Yoshida, Kyoto, Japan.

(2) K. Ichikawa, S. Fukushima, H. Ouchi and M. Tsuchida, *THIS JOURNAL*, **80**, 6005 (1958).

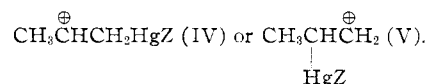
(3) Prof. G. F. Wright, University of Toronto, does not agree with our proposed mechanism of the reaction of  $\text{ArCH}_2\text{CH}_2\text{HgZ}$  to form  $\text{ArCH}_2\text{CH}_2\text{Z}$  and metallic mercury in the preceding paper II.<sup>2</sup> He suggested a radical reaction of  $\text{RHgZ} + \text{HgZ}_2 \rightarrow \text{RZ} + \text{Hg}_2\text{Z}_2$  and a subsequent reaction of  $\text{Hg}_2\text{Z}_2 \rightleftharpoons \text{HgZ}_2 + \text{Hg}$  (private communication); see also D. A. Shearer and G. E. Wright, *Can. J. Chem.*, **33**, 1002 (1955).

The reaction of the propylene–mercuric acetate addition compound with anisole gives 1-(*p*-methoxyphenyl)-2-propyl acetate (I) in yields of up to 67%.

If we assume that the reaction proceeds through a free carbonium ion, *i.e.*, the complex dissociates into ions before it reacts with the aromatic compound, then the complex

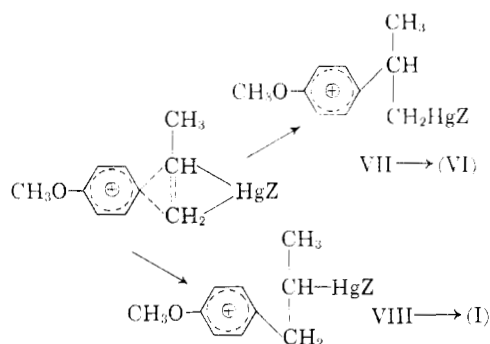


may give either of two carbonium ions



The secondary carbonium ion IV, much more stable than the primary ion V, will be formed preferentially and the reaction should result in the formation of 2-(*p*-methoxyphenyl)-1-propyl acetate (VI). The acetate actually obtained, however, proved to be I exclusively (demonstrated by hydrolysis to the alcohol and subsequent conversion to the *p*-toluenesulfonate). This eliminates reaction by the carbonium ion and makes the direct participation of anisole in an  $\text{S}_{\text{N}}2$  type reaction on the complex the more reasonable mechanism. Anisole can combine with either the central carbon atom of propylene to form VI or with the terminal carbon atom to form I through the intermediate VII or

VIII depicted below. Which is formed, VII or VIII, depends on the relative stabilities of the transition states in the two reaction paths. Considering the fact that 2-aryl-1-propyl *p*-toluene-



sulfonate is reported to rearrange to 1-aryl-2-propyl derivatives during solvolysis,<sup>4,5</sup> it is reasonable to assume that the activation energy of the path to VIII will be the lower and that the final product of this reaction should be I exclusively. This same kind of isomerization might be possible in the next step, *i.e.*, the formation of *p*-methoxyphenylpropyl acetate from VII or VIII. Intermediate mercurials were not isolated in this case, but this possibility was excluded by the results of the reaction of styrene which are described later. It is therefore concluded that the position of mercury in the entity which is formed under the reaction conditions is different from that expected from the conventional formula of the addition compound (2-acetoxypromercuric acetate)<sup>6</sup> and that the proposed form of the olefin-mercuric acetate complex is the reacting species.<sup>7</sup>

In the case of 2-butene, there is no possibility of the formation of isomers. The product is 3-(*p*-methoxyphenyl)-2-butyl acetate, the yield being lower than with propylene.

Reaction of the styrene-mercuric acetate addition compound with anisole gives 1-phenyl-1,2-bis-(*p*-methoxyphenyl)-ethane, which is the product of reaction between one mole of the addition compound and two moles of anisole. Phenyl-(*p*-methoxyphenyl)-ethyl acetate, the expected product of a mole-to-mole reaction, is not obtained under the usual conditions. Therefore, the question of whether the first anisole combines with an  $\alpha$ - or with a  $\beta$ -carbon atom of styrene cannot be determined by the structure of the final product. The point was cleared up, however, as follows: the reaction was stopped at the stage of the intermediate mercurial, and this mercurial was converted to an iodide by treatment with iodine and then to olefin by dehydriodination with alcoholic alkali. The olefin obtained was found to be a mixture of 1-phenyl-1-(*p*-methoxyphenyl)-ethylene (XI) and *p*-methoxystilbene (XII) in the ratio of roughly 2:1.

(4) S. Winstein and A. H. Fainberg, *THIS JOURNAL*, **80**, 459 (1958).

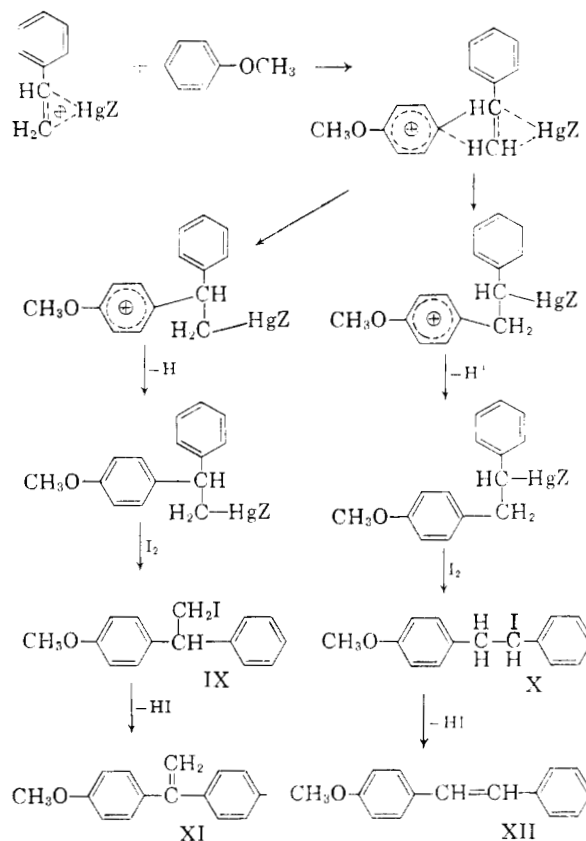
(5) D. B. Denney and B. Goldstein, *ibid.*, **79**, 4948 (1957).

(6) F. C. Whitmore, "Organic Compounds of Mercury," Chemical Catalog Co., (Reinhold Publ. Corp.), New York, N. Y., 1921, p. 115.

(7) Recently, C. W. Whitehead and J. J. Traverso assumed the same complex form in alkoxymercuration of allylic compounds; *THIS JOURNAL*, **80**, 2182 (1958).

Isomerization during dehydriodination must be considered, because quantitative separation of the iodide mixture was not carried out. One of the iodides was, however, separated in a pure form from the mixture by fractional recrystallization and was dehydriodinated to XI quantitatively. This iodide should be 2-phenyl-2-(*p*-methoxyphenyl)-ethyl iodide (IX), because benzyl-(*p*-methoxyphenyl)-carbinol is reported to give *p*-methoxystilbene by boiling with 50% sulfuric acid<sup>8</sup> and this suggests strongly that no isomerization occurs in the alkaline dehydriodination of 1-phenyl-2-(*p*-methoxyphenyl)-ethyl iodide (X) to XII. It can be concluded, therefore, that no isomerization occurs in the dehydriodination of the iodides.

Accordingly, the reaction scheme can be expressed by the equations



Thus the same mechanistic considerations as were used in the case of propylene can be successfully applied to this case.

As an example of a cyclic olefin, the cyclohexene-mercuric acetate addition compound was allowed to react with anisole in the presence of perchloric acid at 30–35°. Formation of free mercury was much slower here than in other cases, and the yield was lower as well. The product was mainly methoxycyclohexene with a small amount of a low boiling fraction and some di-(methoxyphenyl)-cyclohexane. This shows that the expected acetate was formed, but acetic acid was subsequently split off. The position of the double bond in this material was not determined, because the possibility

(8) A. Orekhoff and M. Tiffeneau, *Bull. soc. chim. France*, [4] **37**, 1415 (1925).

TABLE I  
 REACTION OF OLEFINS WITH ANISOLE IN THE PRESENCE OF MERCURIC ACETATE AND CATALYST

Olefin	Anisole, g.	Acetic acid, g.	Mercuric acetate, g.	Catalyst—Type	Wt., g.	Temp., °C.	Time, hr.	Product—		Yield, %
								Kind	Wt., g.	
Propylene	150	100	64	HClO <sub>4</sub> (60%)	34	45–46	3	I <sup>a</sup>	22.6	54
	150	60	64		34	45–46	2		23	55
	70	60	64	BF <sub>3</sub> ·HOAc	25	45–50	7.5		25.6	62 <sup>a</sup>
	100	60	64		30	48–52	3		28	67 <sup>b</sup>
	70	60	64	BF <sub>3</sub> ·Et <sub>2</sub> O	25	46–53	5		27.5	66
2-Butene	100	30	96	H <sub>3</sub> PO <sub>4</sub> (90%)	37	120–130	8	II	8.5	13
	100	100	64	HClO <sub>4</sub> (60%)	17	70	7		7.3	16
	100	30	96		51	70	1.2		10	15
	100	70	96	CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> SO <sub>3</sub> H	28	85–110	16.5		6.3	10 <sup>c</sup>
	50	30	32	BF <sub>3</sub> ·HOAc	17	38–44	3.5		9	40
Styrene	50	30	32	HClO <sub>4</sub> (60%)	17	40	2.5	III	20	63 <sup>d</sup>

<sup>a,b,c,d</sup> Free mercury was obtained in yields of 90, 75, 55 and 83%, respectively. <sup>a</sup> I, 1-(*p*-Methoxyphenyl)-2-propyl acetate; II, 3-(*p*-methoxyphenyl)-2-butyl acetate; III, 1-phenyl-1,2-bis-(*p*-methoxyphenyl)-ethane.

of acid-catalyzed isomerization would have invalidated the result.

### Experimental

The experimental procedure was method B of our previous paper.<sup>2</sup> The olefin-mercuric acetate addition compounds were prepared in acetic acid solution. First anisole, and then the catalyst were added to this solution with good stirring and the mixture was kept at the reaction temperature until formation of metallic mercury could no longer be observed.

**1-(*p*-Methoxyphenyl)-2-propyl Acetate (I).**—Reaction of propylenemercuric acetate with anisole gave 1-(*p*-methoxyphenyl)-2-propyl acetate, b.p. 122.5–124° at 4.5 mm., *n*<sub>D</sub><sup>20</sup> 1.4997 (reported<sup>9</sup> b.p. 113° at 1.7 mm., *n*<sub>D</sub><sup>20</sup> 1.4989).

*Anal.* Calcd. for C<sub>12</sub>H<sub>16</sub>O<sub>3</sub>: C, 69.21; H, 7.74. Found: C, 69.30; H, 7.94.

**1-(*p*-Methoxyphenyl)-2-propanol (XIII).**—With 37 g. of 20% ethanolic potassium hydroxide, 12.4 g. of I was hydrolyzed by boiling for 10 hr. After removal of ethanol, the reaction mixture was extracted with ether. The ether extract was washed with water, neutralized with dilute sulfuric acid, washed again with water and then dried over anhydrous sodium sulfate. Distillation gave 8.4 g. of a viscous liquid, b.p. 121–122° at 4 mm., *n*<sub>D</sub><sup>25</sup> 1.5250 (reported<sup>9</sup> b.p. 119° at 4 mm., *n*<sub>D</sub><sup>25</sup> 1.5261).

**1-(*p*-Methoxyphenyl)-2-propyl *p*-Toluenesulfonate.**—To 2.8 g. of *p*-toluenesulfonyl chloride in 12 ml. of anhydrous pyridine, 1.7 g. of XIII obtained above was added slowly at –5 to –2°. The mixture was allowed to stand for 16 hr. at –1 to 0° and then 20 ml. of water was added slowly under cooling at –5 to –2°. The crude crystals weighed 3.2 g., m.p. 65–71°. Recrystallization from *n*-hexane gave 2.7 g. of 1-(*p*-methoxyphenyl)-2-propyl *p*-toluene sulfonate, m.p. 79–80° (reported<sup>9</sup> 80°), in 83% yield.

*Anal.* Calcd. for C<sub>17</sub>H<sub>20</sub>O<sub>4</sub>S: C, 63.72; H, 6.29. Found: C, 63.61; H, 6.46.

**3-(*p*-Methoxyphenyl)-2-butyl Acetate (II).**—The reaction of 2-butene-mercuric acetate with anisole gave 3-(*p*-methoxyphenyl)-2-butyl acetate, b.p. 124–128° at 3.5 mm., *n*<sub>D</sub><sup>20</sup> 1.5020.

*Anal.* Calcd. for C<sub>13</sub>H<sub>18</sub>O<sub>3</sub>: C, 70.24; H, 8.16. Found: C, 70.24; H, 8.15.

**3-(*p*-Methoxyphenyl)-2-butanol (XIV).**—Hydrolysis of 7.5 g. of II with ethanolic sodium hydroxide gave 4.7 g. of 3-(*p*-methoxyphenyl)-2-butanol, b.p. 140–142° at 14 mm., *n*<sub>D</sub><sup>20</sup> 1.5240 (reported<sup>10</sup> b.p. 149–149.5° at 14 mm., *n*<sub>D</sub><sup>20</sup> 1.5247).

*Anal.* Calcd. for C<sub>11</sub>H<sub>16</sub>O<sub>2</sub>: C, 73.30; H, 8.95. Found: C, 73.57; H, 8.83.

**3-(*p*-Methoxyphenyl)-2-butanone.**—To a solution of 1.8 g. of XIV in 10 ml. of benzene, 28 ml. of 3 *N* aqueous chromic acid was added slowly at 9–17°. After standing at

42–58° for 7 hr., the reaction mixture was extracted with ether. The ether extract was washed with aqueous sodium bicarbonate and then with water and was dried over anhydrous sodium sulfate. Distillation gave 1.3 g. (78% yield) of 3-(*p*-methoxyphenyl)-2-butanone, b.p. 131–134° at 14 mm. The semicarbazone melted at 170–170.5° after seven recrystallizations from aqueous 30–40% ethanol (reported<sup>10</sup> 170°).

**Oxidation of II with Potassium Permanganate.**—Oxidation of 2 g. of II with 8 g. of potassium permanganate, 0.5 g. of sodium hydroxide and 90 ml. of water under reflux for 5.5 hr. gave 0.4 g. of *p*-anisic acid, m.p. 184° (reported<sup>11</sup> 183–184°).

**1-Phenyl-1,2-bis-(*p*-methoxyphenyl)-ethane (III).**—The reaction of styrene-mercuric acetate with anisole gave a product boiling at 220–228° at 3 mm. Recrystallization of 20 g. of crude product from 30 ml. of ethanol gave 18 g. white crystals melting at 95–97°. After another recrystallization from benzene, the melting point was 98.1–98.7°. Mixed melting point with an authentic sample of 1-phenyl-1,2-bis-(*p*-methoxyphenyl)-ethane which was synthesized by the method described below showed no depression.

*Anal.* Calcd. for C<sub>22</sub>H<sub>22</sub>O<sub>2</sub>: C, 82.98; H, 6.96. Found: C, 83.02; H, 6.99.

The authentic sample was synthesized by the reaction of *p*-methoxybenzylmagnesium chloride with *p*-methoxybenzyl chloride. *p*-Methoxybenzyl chloride, b.p. 84–85.5° at 3 mm., *n*<sub>D</sub><sup>18</sup> 1.5498 (reported<sup>12</sup> b.p. 115–117° at 14 mm.) was prepared by the reaction of hydrochloric acid with *p*-methoxybenzyl alcohol, b.p. 109.5–111.5° at 3 mm. (reported<sup>12</sup> 140–141° at 12 mm.), which was made by a crossed Cannizzaro reaction between anisaldehyde and formalin according to the method proposed in the same report. In order to obtain a good yield of the chloride, complete removal of acid by drying over anhydrous potassium carbonate was found to be essential before distillation of the chlorinated product. *p*-Methoxybenzyl chloride, m.p. 61.2–62° (reported<sup>13</sup> 62–63°), was prepared by chlorination of *p*-methoxybenzyl alcohol, m.p. 65.5–66° (reported<sup>13</sup> 66–67°), with hydrochloric acid.

A quantity (5.8 g.) of *p*-methoxybenzyl chloride was added in several portions to a mixture of benzene (50 ml.) and an ethereal solution of *p*-methoxybenzylmagnesium chloride which was prepared from 6 g. of magnesium, 7.8 g. of the chloride and 50 ml. of ether according to the method proposed by Van Campen, *et al.*<sup>14</sup> The reaction mixture was worked up in the usual way and 3.2 g. of a product, b.p. 220–240° at 2 mm., m.p. 85–90°, was obtained. After three recrystallizations from alcohol, 1.8 g. of white needles

(11) H. Gilman, W. Langham and H. B. Willis, *THIS JOURNAL*, **62**, 347 (1940).

(12) T. F. Dankova, L. G. Evdokimova, I. I. Stepanov and N. A. Preobrazhenskii, *C. A.*, **43**, 2606 (1949).

(13) M. P. Balfe, M. A. Doughty, J. Kenyon and R. Poplett, *J. Chem. Soc.*, 608 (1942).

(14) M. G. Van Campen, D. F. Meisner and S. M. Farmerter, *THIS JOURNAL*, **70**, 2296 (1948).

(9) S. Winstein, M. Brown, K. C. Schreiber and A. H. Schlesinger, *THIS JOURNAL*, **74**, 1146 (1952).

(10) A. Sosa, *Ann. chim.* [11] **14**, 62 (1940).

of 1-phenyl-1,2-bis-(*p*-methoxyphenyl)-ethane with constant melting point, 98.1–98.6°, resulted.

*Anal.* Calcd. for  $C_{22}H_{22}O_2$ : C, 82.98; H, 6.96. Found: C, 83.14; H, 6.89.

**Preparation of the Intermediate Mercurial of the Reaction of Styrene–Mercuric Acetate with Anisole and the Determination of its Structure.**—At 18–20°, 33.5 g. of styrene was added to 96 g. of mercuric acetate in 90 ml. of acetic acid with stirring. Formation of an addition compound was complete within about 20 minutes. To this solution, 150 g. of anisole and then 51 g. of 60% perchloric acid were added with continued stirring at about 20°. After 20 minutes, the reaction mixture became faintly cloudy and the formation of a small amount of free mercury was observed. At this point, the reaction mixture was poured into 700 ml. of water. The resulting heavy oil was washed with water and then shaken vigorously with 300 ml. of 10% hydrochloric acid for about 20 minutes. After cooling at 0°, the resultant white crystals were separated from hydrochloric acid and were then washed with 200 ml. of methanol (77 g. (A), m.p. 102–108°). The mother liquor contained a layer of heavy oil. This oily layer was combined with the methanol which had been used for washing the crystals (A). The mixture was washed with water, dried over anhydrous sodium sulfate and then the solvent was evaporated under reduced pressure. The gummy mercurial (B) (46 g.) was obtained. When 10% sodium chloride solution was used instead of 10% hydrochloric acid mentioned above, the same results were obtained. At 72–75°, 68 g. of iodine in a solution of 136 g. of potassium iodide and 540 ml. of water was added to a mixture of 123 g. of mercurial (A + B), 280 ml. of benzene and 500 ml. of water during the course of about 20 minutes. The reaction mixture was kept at 72–75° for 10 minutes. After cooling to room temperature, excess iodine was decolorized with sodium thiosulfate solution, and the oily layer was separated from the water layer. The latter was extracted with benzene, the benzene extract was combined with the former and the whole was washed with water. After drying over anhydrous sodium sulfate, benzene was evaporated under reduced pressure below 40°. A mixture of iodides (100 g., m.p. 50–60°) was obtained. A quantitative fractional crystallization proved to be difficult, and so this mixture was submitted to alkaline dehydroiodination. A mixture of 50 g. of iodides, 200 ml. of methanol, 43 g. of potassium hydroxide and 8.6 g. of water was refluxed for 2 hr. After cooling, concentrated sodium chloride solution was added to the reaction mixture and the resultant solid was extracted with benzene. The benzene extract was washed with water and then dried over anhydrous sodium sulfate. After removal of the benzene, vacuum distillation gave 23 g. of a product, b.p. 146–159° at 2.5 mm. This was redistilled to give three fractions: (a) 13 g., b.p. 170–175° at 9 mm.; (b) 2.6 g., b.p. 176–184° at 9 mm.; (c) 6.5 g., b.p. 185–195° at 9 mm. Recrystallization of (a) from ethanol gave white plates of 1-phenyl-1-(*p*-methoxyphenyl)-ethylene (XI), m.p. 74.8–75° (reported<sup>15</sup> 75°).

*Anal.* Calcd. for  $C_{15}H_{14}O$ : C, 85.68; H, 6.71. Found: C, 85.39; H, 6.72.

From (c), white needles of *p*-methoxystilbene (XII), m.p.

136° (reported<sup>16</sup> 135–135.5°), were obtained by recrystallization from ethanol.

*Anal.* Calcd. for  $C_{15}H_{14}O$ : C, 85.68; H, 6.71. Found: C, 85.61; H, 6.77.

Fraction b was the mixture of (a) and (c), m.p. 69–70°.

*Anal.* Calcd. for  $C_{15}H_{14}O$ : C, 85.68; H, 6.71. Found: C, 85.70; H, 6.67.

It can be concluded, therefore, that the mercurial is the mixture of 2-phenyl-2-(*p*-methoxyphenyl)- and 1-phenyl-2-(*p*-methoxyphenyl)-ethylmercuric chlorides in a ratio of roughly 2:1.

**2-Phenyl-2-(*p*-methoxyphenyl)-ethyl Iodide (IX).**—A quantity (50 g.) of the iodide mixture obtained above was dissolved in 150 ml. of hot ethanol. The ethanol solution was cooled to 30° and the resulting crystals were recrystallized from ethanol (yield 13 g., m.p. 80–81°). Dehydroiodination by the same method as described above gave XI quantitatively. This iodide, therefore, is 2-phenyl-2-(*p*-methoxyphenyl)-ethyl iodide.

*Anal.* Calcd. for  $C_{15}H_{15}IO$ : C, 53.27; H, 4.47. Found: C, 53.50; H, 4.17.

**Reaction of Cyclohexene–Mercuric Acetate with Anisole in the Presence of Perchloric Acid.**—Cyclohexene (16.4 g., 0.2 mole) dissolved in 50 ml. of acetic acid was added to 80 g. (0.25 mole) of mercuric acetate. The excess mercuric acetate was filtered off after 30 minutes of agitation. To the filtrate, 108 g. of anisole and then 34 g. of 60% perchloric acid were added under stirring. Reaction was continued for 13 hr. at 30–34°. The resultant mercury (40 g.) was removed and the reaction mixture was added to water and this was extracted with benzene. The benzene extract was washed with water, neutralized with 5% sodium bicarbonate solution, washed again with water and then dried over anhydrous sodium sulfate. Distillation gave the fractions: (a) 2 g., b.p. 55–120° at 18 mm.; (b) 10 g., b.p. 120–157° at 17 mm.; and (c) 3 g., b.p. 210–220° at 5 mm. Identification of the fraction a was not performed. It had, however, a characteristic odor of cyclohexenyl acetate. Redistillation of fraction b gave 8 g. of a liquid, b.p. 145–147° at 14 mm.,  $n_D^{20}$  1.5505. This had the composition expected for methoxyphenylcyclohexene. Double bond determination by the method of Francis also supported this structure (91–92.4% of the theoretical value).

*Anal.* Calcd. for  $C_{13}H_{13}O$ : C, 82.93; H, 8.57. Found: C, 82.69; H, 8.43.

Redistillation of fraction c gave 2 g. of a liquid, b.p. 203–205° at 3 mm.,  $n_D^{20}$  1.5795 (reported<sup>17</sup>  $n_D^{20}$  1.5806 for *cis*-1,2-di-(*p*-methoxyphenyl)-cyclohexane). Bayer's test showed that this was a saturated compound. This is thought to be di-(methoxyphenyl)-cyclohexane. However, the detailed structure was not determined.

**Acknowledgment.**—The authors wish to express their thanks to Dr. A. J. Kresge for his encouraging discussion and for his kind help in the preparation of the manuscript, and to Sumitomo Chemical Co. for financial support.

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(17) E. C. Dodds, R. L. Huang, W. Lawson and R. Robinson, *Proc. Roy. Soc. (London)*, **B140**, 493 (1953).

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