rotation[6] at equilibrium (103 hr.) of −9.8° (*C*, 2.408; *t*, 24.9°; H_2O). The crystals are easily soluble in water, slightly in methanol, and insoluble in ethanol, ether, acetone and chloroform.

Anal. Calcd. for $C_6H_{13}O_6N$: C, 36.9; H, 6.66; N, 7.17. Found: C, 36.9, 37.1, 36.7; H, 6.15, 6.84, 6.86; N, 6.93, 7.04.

Mutarotation of D-Altrose Oxime.—A sample of 0.2422 g. of twice recrystallized material was dissolved into 10.06 cc. of aqueous solution. Twenty-two measurements of the rotations were made in a 2-dm. tube at 25.0 ± 0.5°. The reaction followed an unimolecular course, the calculated velocity constant ($k_1 + k_2$) being 0.0040 with an average variation of ±0.0001. In making this calculation the value obtained for the rotation at $t = 748$ min. was taken as the rotation at infinite time rather than that at $t = 6540$ min. since hydrolysis is appreciable after very long contact with water. The *p*H of the water was 5.8.

By plotting log ($\alpha_t - \alpha_\infty$) against time in minutes, a very nearly straight line was obtained. By extrapolation of this line to zero time, the value of log ($\alpha_0 - \alpha_\infty$) was determined graphically and the initial specific rotation[6] found to be −64.0°.

Summary

A large-scale and abbreviated procedure for the preparation of crystalline D-altrose from D-lactose by the method of Richtmyer and Hudson is described.

The oxime of D-altrose is described and measurements of its rate of mutarotation recorded.

CAMBRIDGE, MASSACHUSETTS
RECEIVED NOVEMBER 1, 1943

---

[6] All rotations referred to in this paper represent specific rotations of the D line of sodium unless specified otherwise. Concentrations are grams of solute in 100.0 cc. of solution.

[CONTRIBUTION FROM THE NOYES CHEMICAL LABORATORY, UNIVERSITY OF ILLINOIS]

Condensation of Cyclohexene Oxide, 1,2-Dichlorocyclohexane, and 3,4-Dichlorohexane with Anisole[1]

BY CHARLES C. PRICE AND GEORGE P. MUELLER[2]

This investigation was undertaken to explore the possibility of synthesizing vicinal *bis*-(*p*-hydroxyphenyl)-hexanes and cyclohexanes by some modification of the Friedel–Crafts type reaction. Such substances are of particular interest in connection with their ability to simulate the physiological activity of the hormone, estrone.

Kursanoff[3,4] has reported the isolation of a diphenylcyclohexane, m. p. 169–171°, which he believed to be the 1,2-isomer, from the aluminum chloride-catalyzed condensation of 1,2-dichlorocyclohexane with benzene. This material has now been shown to be the 1,4-isomer.[5,6] Nenitzescu and Curcaneanu isolated a greater amount of liquid 1,3-diphenylcyclohexane than the crystalline 1,4-isomer. It is thus obvious that, as so frequently happens with the Friedel–Crafts reaction, rearrangement occurred in the replacement of the chlorine atoms. Other similar rearrangements in vicinal, negatively-substituted halides also have been observed.[7]

It was hoped that, due to the greater reactivity of the aromatic nucleus in anisole or phenol, or by modification of the conditions of the Friedel–Crafts condensation, this tendency toward rearrangement might be modified. This hope was only partially realized. Condensations of anisole with 1,2-dichlorocyclohexane using aluminum chloride as the catalyst or with cyclohexene oxide or 1,2-dihydroxycyclohexane using boron fluoride as the catalyst produced poor yields of mixtures of condensation products from which two diastereoisomeric 1,3-dianisylcyclohexanes (I and II), *m*-dianisylbenzene (III) and anisylcyclohexane (IV) were isolated and identified. The fact that no 1,4-dianisylcyclohexane or *p*-dianisylbenzene was isolated is an indication that the tendency to rearrangement was at least somewhat less with anisole than with benzene.

The terphenyl was assigned the meta-orientation on the basis of the non-identity of the free phenol, its dimethyl ether and diacetate with the corresponding ortho and para isomers[8] and zinc dust distillation to yield a small amount of material with the proper melting point for *m*-terphenyl itself.

The 1,3-dianisylcyclohexane, the free phenol and its diacetate each differs from the known 1,1-isomers.[9]

The production of 1,3-dianisylcyclohexane, as well as *m*-dianisylbenzene and anisylcyclohexane, may be accounted for by the intermediate formation of 3-anisylcyclohexene (V). The anisyl group in the cycloölefin should direct addition of a second molecule of anisole to form 1,3-dianisylcyclohexane.[10] Disproportionation of hydrogen between the 3-anisylcyclohexene (V) and the 1,3-dianisylcyclohexanes would account for the

---

[1] Presented before the Organic Division at the 104th Meeting of the American Chemical Society, Buffalo, New York, September 7, 1942.

[2] Eli Lilly and Company Fellow, 1941–1943.

[3] Kursanoff, *Ann.*, **318**, 309 (1901).

[4] Bodroux, *Ann. chim.*, [10] **11**, 511 (1929).

[5] Nenitzescu and Curcaneanu, *Ber.*, **70B**, 346 (1937).

[6] Corson and Ipatieff, THIS JOURNAL, **60**, 747 (1938).

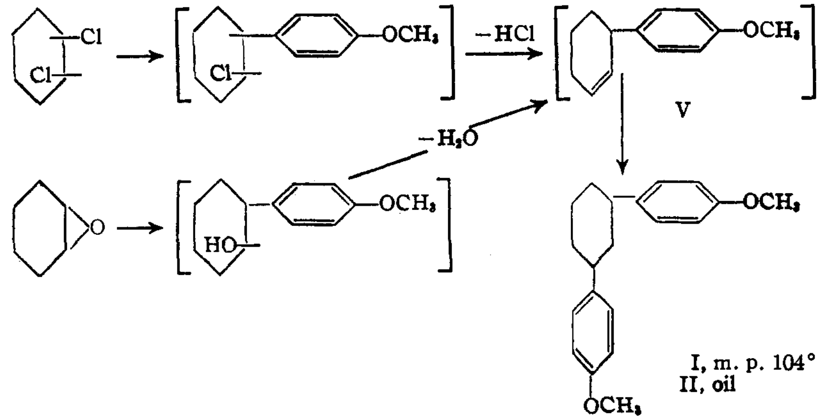
[7] Nenitzescu and Gavat, *Ann.*, **519**, 260 (1935); *Ber.*, **70B**, 1883 (1937); Nenitzescu and Przemetzki, *ibid.*, **69B**, 2706 (1936); Nenitzescu and Curcaneanu, *Bull. chim. soc. Romania*, [2] **1**, 125 (1939) (*C. A.*, **37**, 3740 (1943)).

[8] Price and Mueller, THIS JOURNAL, **66**, 632 (1944).

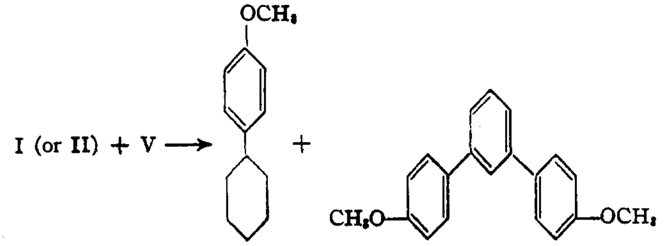
[9] Schmidlin and Lang, *Ber.*, **43**, 2819 (1910); von Braun, *Ann.*, **472**, 55 (1929).

[10] See, *e. g.*, Price and Karabinos, THIS JOURNAL, **62**, 1159 (1940).

anisylcyclohexane (IV) and *m*-dianisylbenzene (III) actually isolated.



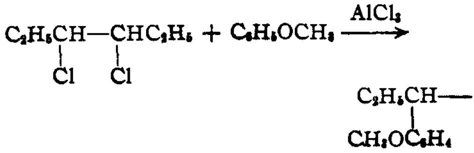
I, m. p. 104°
II, oil



IV, m. p. 58° III. m. p. 198°

For the case of the cyclohexene oxide, opening of the oxide ring has been shown in general to involve an inversion of configuration[11] and would thus produce the *trans*-alcohol as indicated. Dehydration of the corresponding alcohol without the methoxyl group has already been shown to produce 3-phenylcyclohexene.[10]

The open-chain analog of 1,2-dichlorocyclohexane, 3,4-dichlorohexane, was also condensed with anisole. This halide was extremely unreactive in the presence of aluminum chloride but it was possible to obtain a 14% yield of condensation product from which a 1% yield of hexestrol dimethyl ether was crystallized. In view of the work of Nenitzescu,[7,12] it seems likely that the remainder of the condensation product consisted of isomers of hexestrol.



$$\underset{\mathrm{Cl}}{C_2H_5CH}—\underset{\mathrm{Cl}}{CHC_2H_5} + C_6H_5OCH_3 \xrightarrow{AlCl_3} \underset{CH_3OC_6H_4}{C_2H_5CH}—\underset{C_6H_4OCH_3}{CHC_2H_5}$$

m. p. 142–143°
(1%)

The dihalide used was obtained by ethylation of acetylene with ethyl bromide[13] (20% yield), hydrogenation to *cis*-3-hexene[14] (95% yield) and chlorination[15] (84% yield).

The crystalline and oily isomers of 1,3-*bis*-(*p*-hydroxyphenyl)-cyclohexane and 4,4″-dihydroxy-*m*-terphenyl were found to possess no estrogenic activity when administered to spayed mice in doses up to 0.4 mg. per mouse. 1,3-*bis*-(*p*-Hydroxyphenyl)-cyclohexane showed no androgenic activity at a maximum total dose of 18 mg. per animal in castrated rats.[16]

Experimental[17]

Condensation of Cyclohexene Oxide with Phenol in the Presence of Boron Fluoride.—Boron fluoride was passed through a stirred mixture consisting of 28.8 g. (0.306 mole) of phenol and 5.0 g. (0.051 mole) of cyclohexene oxide at 40–70°. After six hours the purple complex was decomposed in water, extracted with 75 cc. of benzene and the benzene solution washed thoroughly with water and dried. The benzene and excess phenol were removed by distillation, the latter at 16 mm. pressure, and 7.5 g. of a dark oil remained as residue.

A portion of this was dissolved in aqueous sodium hydroxide; in a few minutes a crystalline sodium salt had separated from the solution. All qualitative tests for phenolic compounds failed as did attempts to crystallize the free phenol, its benzoate, 3,5-dinitrobenzoate, *p*-toluenesulfonate, picrate or aryloxyacetic acid derivatives.

A second reaction was carried out using five times the above quantities of reactants by passing boron fluoride through the mixture for ten hours. Fractional distillation of the crude oil yielded 8.9 g. of an oil, b. p. 135–175° (9 mm.). This crystallized upon cooling. It was redistilled and recrystallized, m. p. 103.4°, and proved to be *trans*-1,2-dihydroxycyclohexane. From this fraction a small amount of *p*-cyclohexylphenol was also obtained, m. p. 125–128°.[18] It was identified by conversion to the 3,5-dinitrobenzoate, m. p. 164–166°, and by analysis.

Anal. Calcd. for $C_{12}H_{16}O$: C, 81.76; H, 9.15. Found: C, 81.86; H, 8.71.

Condensation of Cyclohexene Oxide with Anisole in the Presence of Boron Fluoride.—Boron fluoride was bubbled into a mixture of 25 g. (0.255 mole) of cyclohexene oxide and 165 g. (1.53 mole) of anisole at 50° for three hours, with constant stirring. The dark-red complex was decomposed with 400 cc. of water. The organic liquid was extracted with ether, washed thoroughly with water and distilled at reduced pressure from a Claisen flask modified with a Vigreux column.

The first fraction collected was a colorless liquid, b. p. 89–91° (1 mm.). This was redistilled, b. p. 273–276° (740 mm.), and recrystallized several times from dilute ethyl alcohol. The colorless plates, m. p. 56.8–57.5°, were, according to these physical constants,[4] *p*-cyclohexylanisole. It was obtained in 8% yield.

A second fraction, 20 g., was collected, b. p. 95–183°

---

(11) Kuhn and Ebel, *Ber.*, **58**, 921 (1925); Böeseken, *Rec. trav. chim.*, **47**, 683 (1928); Wilson and Lucas, THIS JOURNAL, **58**, 2396 (1936).

(12) Nenitzescu, Gavat and Corcora, *Ber.*, **73**, 233 (1940).

(13) Bried and Hennion, THIS JOURNAL, **59**, 1310 (1937).

(14) Campbell and Eby, *ibid.*, **63**, 216 (1941).

(15) Spiegler and Tinker, *ibid.*, **61**, 940 (1939).

(16) We are indebted to Eli Lilly and Company for the results of these physiological tests.

(17) The analyses were carried out by Miss Margaret McCarthy, Miss Theta Spoor and Miss Dorothy Schneider.

(18) Schrauth and Görig, *Ber.*, **56**, 1900 (1923); Kursanoff, *Ann.* **318**, 325 (1901).

(1 mm.). Redistillation gave a colorless oil, b. p. 160–165° (1 mm.); n^{25} 1.5694. This proved to be the liquid isomer of 1,3-dianisylcyclohexane. It contained an impurity which gave the liquid a violet fluorescence.

Anal. Calcd. for $C_{20}H_{24}O_2$: C, 81.04; H, 8.16. Found: C, 81.50; H, 8.39.

The third fraction, b. p. 190–245° (1 mm.), was collected as a brown oil, part of which crystallized after standing for a few days. A little ether was added to the mass and the crystals collected. They were purified by recrystallization from petroleum ether (b. p. 96°), methylcyclohexane, and ethyl, butyl or amyl alcohol. The 4,4″-dimethoxy-*m*-terphenyl crystallized in large, colorless plates, m. p. 197–198° (cor.).

Anal. Calcd. for $C_{20}H_{18}O_2$: C, 82.73; H, 6.25. Found: C, 82.67; H, 6.55.

Condensation of 1,2-Dihydroxycyclohexane with Anisole in the Presence of Boron Fluoride.—1,2-Dihydroxycyclohexane was prepared by the method of Senderens and Aboulenc.[19] The equimolecular mixture of *dl* and *meso* forms was used, m. p. 73°.[20] As in the preceding experiment, 27 g. (0.233 mole) of the cyclohexane and 152 g. (1.4 mole) of anisole were stirred at 45° for three hours while boron fluoride was bubbled through the mixture. The same products as in the preceding condensation were isolated with an over-all yield of condensed material about one-fourth as great.

Condensation of 1,2-Dichlorocyclohexane with Anisole in the Presence of Anhydrous Aluminum Chloride.—A 500-cc. flask was equipped with a reflux condenser, thermometer, sealed stirrer and dry-solid addition appendix. 1,2-Dichlorocyclohexane,[21] 41.8 g. (0.273 mole), and anisole, 155 g. (1.43 mole), were placed in the flask and cooled to 5°; 15 g. of anhydrous aluminum chloride was added in small enough portions that the temperature did not rise appreciably. The mixture was stirred for six hours and permitted to stand for sixteen hours in the ice-bath. When worked up, as before, the complex yielded 20 g. of an oil which deposited 2 g. of 4,4″-dimethoxy-*m*-terphenyl upon standing. The crystals were separated and the oil distilled. The lower-boiling fractions yielded 1,2-dichlorocyclohexane and 3 g. of *p*-cyclohexylanisole.

A fraction, 8 g., b. p. 180–220° (3 mm.), was redistilled and a portion, b. p. 191–205° (1 mm.), partially crystallized. The whole was dissolved in a large volume of hot methyl alcohol. A small amount of the terphenyl crystallized and was separated from the solution. Upon standing overnight the solution deposited more crystals, 2 g. of long, white needles. These were recrystallized three times from methyl alcohol and once from ethyl alcohol, m. p. 102.8–104.0°. This proved to be the solid isomer of 1,3-dianisylcyclohexane.

Anal. Calcd. for $C_{20}H_{24}O_2$: C, 81.04; H, 8.16; mol. wt., 296.39. Found: C, 80.93; H, 7.81; mol. wt., 280 (Rast).

The oil remaining from evaporation of the recrystallization liquors was combined with the corresponding fraction from another run, and from 10 g. of oil 4 g. was collected, b. p. 207–210° (1 mm.); n^{25} 1.5769. This oil was the liquid form of 1,3-dianisylcyclohexane, isolated before from cyclohexene oxide and anisole. Its structure and relationship to the solid isomer above was shown by catalytic dehydrogenation to 4,4″-dimethoxy-*m*-terphenyl.

A similar reaction using 76.5 g. (0.5 mole) of the cyclohexane, 324 g. (3.0 mole) of anisole and 50 g. (0.37 mole) of aluminum chloride at 0° for fifty hours gave 16 g. of crude condensation products which were separated into 2 g. of *p*-cyclohexylanisole, 3.5 g. of the crystalline cyclohexane, 9 g. of the oily isomer and 0.5 g. of the terphenyl. The same reaction at 15° for sixty-five hours produced 30 g. of condensation products.

The course of the reaction did not appear to differ materially if carried out at 150–160° for two hours. An attempt to use carbon disulfide as a solvent resulted in a vigorous reaction which produced only ill-smelling, sulfur-containing oils and much tar.

Dehydrogenation of 1,3-Dianisylcyclohexane: 4,4″-Dimethoxy-*m*-terphenyl.—1,3-Dianisylcyclohexane, m. p. 104°, 0.10 g., was heated to 300° in the presence of 0.05 g. of 10% palladium black on Norite. The hydrogen evolved was swept into a nitrometer with a stream of carbon dioxide. The amount of hydrogen collected was 86% of three moles. The residue was suspended in hot ethyl alcohol and the solution filtered from the catalyst. The colorless plates obtained were 4,4″-dimethoxy-*m*-terphenyl, m. p. 193°, mixed m. p. 196–197° with this compound as previously obtained.

The oily isomer was likewise dehydrogenated by heating to 300° with the same catalyst and yielded 4,4″-dimethoxy-*m*-terphenyl, identified by melting point and mixed melting point.

1,3-*bis*-(*p*-Hydroxyphenyl)-cyclohexane.—A mixture of 2.47 g. of 1,3-dianisylcyclohexane, m. p. 104°, 7 g. of potassium hydroxide and 40 cc. of ethyl alcohol was heated at 200° for twenty-four hours in a high-pressure, steel bomb. The cooled solution was rinsed from the bomb with four times its volume of water and acidified with dilute hydrochloric acid. The precipitate was collected and recrystallized from dilute acetic acid or dilute methyl alcohol. The product appeared as long, colorless plates, m. p. 229–232°; yield 2.18 g. or 97%.

Anal. Calcd. for $C_{18}H_{20}O_2$: C, 80.56; H, 7.51. Found: C, 80.40; H, 7.76.

Similar treatment of the liquid isomer yielded only an oil which decomposed when attempts were made to distill it. An attempt to demethylate in hydriodic and acetic acids led only to a red, iodine-containing oil.

1,3-*bis*-(*p*-Acetoxyphenyl)-cyclohexane.—1,3-*bis*-(*p*-Hydroxyphenyl)-cyclohexane, 0.5 g. was warmed with 2 g. of freshly-fused sodium acetate and 10 cc. of acetic anhydride on a steam-bath for three hours. The mixture was hydrolyzed in water, yielding the theoretical amount of product. This was recrystallized four times from methyl alcohol, m. p. 74.5–75.5°.

Anal. Calcd. for $C_{22}H_{24}O_4$: C, 74.98; H, 6.86. Found: C, 74.87; H, 7.05.

4,4″-Dihydroxy-*m*-terphenyl.—The dimethoxy compound, 0.35 g., was heated with 1.0 g. of potassium hydroxide and 20 cc. of ethyl alcohol at 200° for twenty-four hours. The solution was treated as before and 0.29 g. (93%) of the diphenol was obtained. The product crystallized from dilute acetic acid or dilute methyl alcohol as fine plates having a slight lavender tint, m. p. 182–183°.

Anal. Calcd. for $C_{18}H_{14}O_2$: C, 82.42; H, 5.38. Found: C, 82.41; H, 5.51.

Demethylation was also accomplished in 94% yield by refluxing 0.85 g. of the ether with 3 cc. of hydriodic acid (d. 1.7) and 6 cc. of glacial acetic acid for three hours. Dilution and cooling precipitated the product.

The potassium hydroxide cleavage could not be successfully repeated on occasions. An experiment that behaved anomalously was one in which 2.5 g. of the ether was heated at 200° for twenty-four hours with 7.5 g. of potassium hydroxide and 20 cc. of ethyl alcohol. Considerable pressure developed in the bomb, present even after it had cooled. The products were not entirely soluble in the alcoholic alkali. The mixture was diluted and the suspension warmed and filtered. This insoluble residue from the reaction mixture proved to be 4,4″-dimethoxy-*m*-terphenyl.

Acidification of the filtered reaction mixture precipitated 1.25 g. of an impure solid which was recrystallized repeatedly from dilute acetic acid and from dilute ethyl alcohol; white plates, m. p. 66.0–67.5°, were obtained which were insoluble in 10% sodium hydroxide. This was probably 4,4″-dimethoxyperhydro-*m*-terphenyl. The analysis indicated a substance containing about four more hydrogen atoms than 4,4″-dimethoxyperhydroterphenyl.

Anal. Calcd. for $C_{20}H_{36}O_2$: C, 77.86; H, 11.76. Calcd. for $C_{20}H_{36}O_2 \cdot {}^1/_2C_2H_6O$: C, 76.08; H, 11.86. Found: C, 76.73, 75.99; H, 12.58, 12.89.

---

[19] Senderens and Aboulenc, *Compt. rend.*, **173**, 1365 (1921).

[20] Leroux, *ibid.*, **148**, 931 (1909).

[21] Kharasch and Brown, THIS JOURNAL, **61**, 3432 (1939).

In agreement with its formulation as the ether, this compound was resistant to the action of sodium dichromate and sulfuric acid in water at the reflux temperature. Demethylation with constant-boiling hydriodic acid in glacial acetic acid yielded a white product crystallizing from the acid mixture. Attempts to recrystallize this only gave gels, even from large amounts of benzene or alcohol. The gel was dried, m. p. 245–295°.

4,4′-Diacetoxy-*m*-terphenyl.—This derivative was obtained by treating the diphenol with fused sodium acetate and acetic anhydride, as described above. The compound was recrystallized from ethyl alcohol and obtained as white needles, m. p. 130.1–131.5°.

Anal. Calcd. for $C_{22}H_{18}O_4$: C, 76.28; H, 5.24. Found: C, 76.39; H, 5.79.

Oxidation of 4,4′-Dihydroxy-*m*-terphenyl.—The diphenol, 0.1 g., was refluxed with 8 cc. of water, 0.1 cc. of 10% sodium hydroxide and 1.0 g. of potassium permanganate. The solution was cooled, acidified and the manganese dioxide dissolved by additions of sodium bisulfite. The resulting solution contained only a slight haze of insoluble material. It was evaporated to dryness and some of the salt residue heated for three minutes at 160° with resorcinol and one drop of concentrated sulfuric acid. The fused mixture was cooled and diluted with water. The addition of sodium hydroxide produced a red-brown solution with a weak, but deep green fluorescence. This fluorescence and color had a very different character than that produced with phthalic acid. It matched perfectly with the colors obtained from iso- and terephthalic acid controls. The red solution obtained with resorcinol and sulfuric acid under the same conditions was of a distinctly different character.

Oxidations with 50% nitric acid yielded a yellow powder containing nitrogen. The crude product melted at 218–225°. This was not identified.

Zinc Dust Treatment.—A 0.05-g. sample of the cyclohexane was heated at 250° for one hour with 10% palladium-on-Norite catalyst. Zinc dust, 0.1 g., was then added to the melt. The heating was continued for two hours at 300°. The mixture was cooled, pulverized and extracted with benzene. When concentrated, decolorized, filtered and concentrated again to 0.3 cc., the benzene solution yielded a minute amount of crystals. The melting point of 85–90° indicated that this material was *m*-terphenyl (m. p. 89°).[22]

Condensation of Anisole with 3,4-Dichlorohexane in the Presence of Anhydrous Aluminum Chloride.—In the apparatus described previously for the Friedel–Crafts reactions were placed 7.75 g. (0.05 mole) of 3,4-dichlorohexane,[13,14,15] 14.4 g. (0.3 mole) of anisole and 50 cc. of petroleum ether (b. p. 95–96°). The reactants were cooled to 2° and 5 g. of anhydrous aluminum chloride was added in small portions to the stirred mixture. The reaction mixture was then heated to 80° for one and one-half hours, but no gas evolution was observed. Another 5-g. portion of catalyst was added, and subsequent heating to 95° brought about hydrogen chloride evolution for one hour. Heating was continued for ten hours.

The mixture was decomposed in water and treated in the usual manner. Distillation yielded 3 cc. of clear liquid, b. p. 90–123° (1 mm.), and 1 cc. of viscous straw-yellow liquid, b. p. 140–190° (1 mm.). The yield of the latter fraction was 7%.

To a mixture of 3.88 g. (0.025 mole) of 3,4-dichlorohexane, 5.4 g. (0.05 mole) of anisole and 25 cc. of petroleum ether was added 6.7 g. (0.05 mole) of anhydrous aluminum chloride during an hour. The reaction was heated on a steam-bath for four hours. The mixture was cooled and treated as before. This time 2.6 g. of liquid, b. p. 95–150° (1 mm.), and 0.9 g. of a viscous straw-colored oil, b. p. 150–190° (1 mm.), were collected. The latter fraction was combined with the similar product above and methylated, using alternately 2 cc. of dimethyl sulfate and 2.5 cc. of 20% sodium hydroxide. The excess dimethyl sulfate was destroyed with ammonia and the product extracted with ether.

Distillation of the dried ethereal extract yielded a cloudy, yellow oil, b. p. 180–190° (1 mm.). This was diluted with an equal volume of petroleum ether (b. p. 38–45°), seeded and placed in the refrigerator. After four days about 0.05 g. of crystals had been deposited. These were purified by recrystallization from methyl alcohol and appeared as needles, m. p. 142–143° (cor.). A mixed melting point with an authentic sample of hexestrol dimethyl ether[23] remained unchanged.

Anal. Calcd. for $C_{20}H_{26}O_2$: C, 80.59; H, 8.74. Found: C, 80.35; H, 8.89.

The yield of the unmethylated oil was about 14% and that of the crystals about 1%, calculations being based on the amount of 3,4-dichlorohexane used in both reactions.

Summary

1,2-Dichlorocyclohexane and cyclohexene oxide react with anisole in the presence of aluminum chloride or boron fluoride to form a solid and a liquid 1,3-dianisylcyclohexane, *p*-cyclohexylanisole and 4,4″-dimethoxyterphenyl.

3,4-Dichlorohexane condenses with anisole to give a small amount of oily products from which a 1% yield of hexestrol dimethyl ether was isolated.

URBANA, ILLINOIS RECEIVED DECEMBER 22, 1943

---

[22] France, Heilbron and Hey, *J. Chem. Soc.*, 369 (1940).

[23] This sample was furnished through the courtesy of Eli Lilly and Company.