Reaction of VIII with Potassium *t*-Butoxide.—A suspension of 5.0 g. (0.0095 mole) of VIII was heated under reflux with 1.1 g. (0.010 mole) of potassium *t*-butoxide in dioxane for 2.5 hours. The mixture (green-black) was poured into 300 cc. of water. Extraction with Girard T reagent yielded 0.60 g. (23% yield) of 4,4′-dinitrobenzophenone, m.p. and mixed m.p. with authentic sample 188–190°. No other crystalline product could be obtained.

NEW YORK 27, N. Y. RECEIVED NOVEMBER 25, 1950

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY OF COLUMBIA UNIVERSITY]

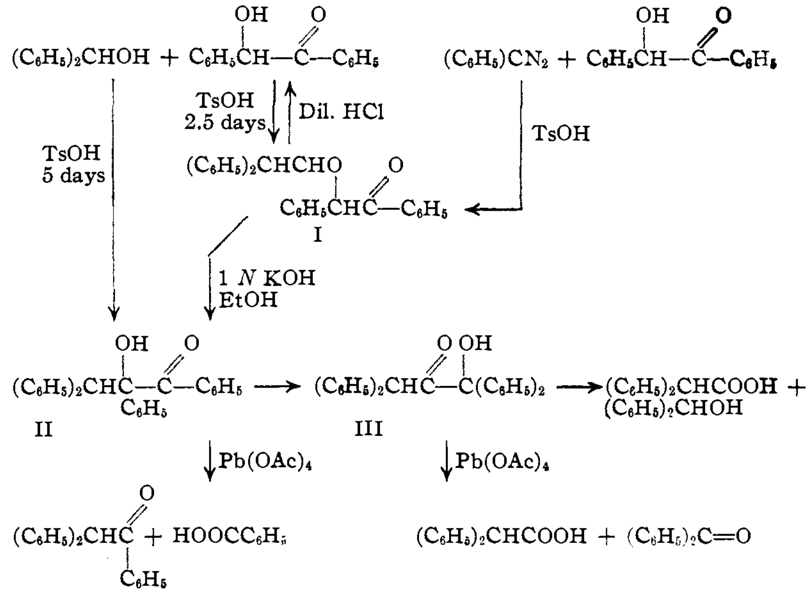
Cleavage and Rearrangement of Ethers with Base. II. Reaction of the Benzhydryl and Trityl Ethers of Benzoin with Potassium Hydroxide[1]

BY DAVID Y. CURTIN AND SIDNEY LESKOWITZ

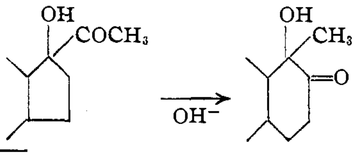
Benzhydryl desyl ether (I) has been prepared and found to rearrange on treatment with 1 N potassium hydroxide in ethanol to α-benzhydryl benzoin (II). II rearranges further to α-hydroxydibenzhydryl ketone (III). III can be cleaved to benzhydrol and diphenylacetic acid. Trityl desyl ether (VI) has also been prepared and found to react with 1 N potassium hydroxide in ethanol to give benzilic acid and triphenylmethane. The significance of these reactions is discussed.

In a continuation of the work on the reaction of ethers with bases discussed in the first paper of this series[2] it was considered desirable to investigate certain compounds which it was hoped would react under milder conditions than the ones previously studied. For this reason an investigation of benzhydryl desyl ether (I) was undertaken. This compound was readily prepared by the reaction of benzhydrol and benzoin in the presence of *p*-toluenesulfonic acid. The structure of I was confirmed by its ready hydrolysis to benzoin with dilute acid and by an alternative synthesis from the reaction of diphenyldiazomethane with benzhydrol in the presence of *p*-toluenesulfonic acid. An attempt to prepare I by the reaction of phenylbenzoyldiazomethane with benzhydrol in the presence of *p*-toluenesulfonic acid gave only desyl *p*-toluenesulfonate.

When I was treated for one hour under reflux with a 1 N solution of potassium hydroxide in ethanol it was converted to a mixture of products from which were isolated two new solid substances (II and III), benzhydrol, diphenylacetic acid and small amounts of tetraphenylethane and benzoic acid. II could also be obtained in 56% yield from benzoin and benzhydrol in the presence of acid when the reaction was allowed to proceed for a longer time than was used for the preparation of I. II was shown to be α-benzhydrylbenzoin by cleavage with lead tetraacetate to benzoic acid (81% yield) and phenyl benzhydryl ketone (50% yield). III was identified as α-hydroxydibenzhydryl ketone by cleavage with lead tetraacetate to benzophenone (68%) and diphenylacetic acid (80%). It was further found that II could be converted to III in 74% yield with ethanolic potassium hydroxide. When III was treated with a refluxing solution of 1 N potassium hydroxide for 90 minutes, benzhydrol (35%), diphenylacetic acid (45%) and unreacted starting material (40%) were obtained. We feel, therefore, that the initial reaction of I is a rearrangement to II followed by rearrangement of II to III and finally cleavage of III to benzhydrol and diphenylacetic acid. These reactions are summarized as shown.



The base-catalyzed rearrangement of I to II seems to be analogous to the rearrangement of dibenzyl ether with phenyllithium[3] or potassium amide[4] or to the Stevens rearrangement[5] and probably proceeds by a similar mechanism. The isolation of tetraphenylethane is of interest since benzhydryl 4,4′-dinitrobenzhydryl ether gives this substance as one of the principal products on treat-



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[1] Abstracted from the Ph.D. thesis submitted to Columbia University by Sidney Leskowitz.

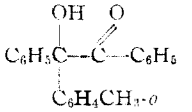
[2] D. Y. Curtin and S. Leskowitz, THIS JOURNAL, **73**, 2630 (1951).

[3] G. Wittig and L. Lohmann, *Ann.*, **550**, 260 (1942).

[4] C. R. Hauser and S. W. Kantor, Abstracts of Papers presented at the 116th Meeting of the American Chemical Society, September, 1949, p. 46M.

[5] See J. L. Dunn and T. S. Stevens, *J. Chem. Soc.*, 279 (1934).

ment with base.[2] The rearrangement of II to III is of unusual interest since it is apparently the first example in a relatively simple molecule of a rearrangement discovered by Stavely[6] in the steroids. Shoppee and Prins[7] extended this rearrangement in the steroid series and proposed that it is an intramolecular rearrangement of the conjugate base of the alcohol. A similar mechanism seems applicable to the rearrangement of II to III. This rearrangement is probably reversible and it is likely that III is the thermodynamically more stable product because of its decreased steric strain. Roger and McGregor[8] have also reported results which may be explained by postulation of a hydroxyketone rearrangement of this type. They allowed *o*-tolylmagnesium bromide to react with benzil and obtained a compound which was not characterized but which may be presumed to have been α-*o*-tolylbenzoin (IV). When IV was treated with base, instead of benzoic acid and phenyl *o*-tolyl ketone as they expected, the products were *o*-toluic acid and benzhydrol. These results suggest that (IV) underwent a base-catalyzed rearrangement to (V) before being cleaved.

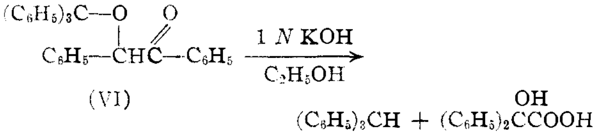


(IV)

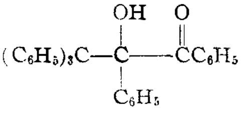
$o\text{-}CH_3\text{—}C_6H_4COCOH(C_6H_5)_2$

(V)

We have also prepared desyl trityl ether (VI) from triphenylcarbinol and benzoin. When VI was treated with 1 *N* potassium hydroxide in refluxing ethanol for one hour it was converted to benzilic acid (88%) and triphenylmethane (93%). This reaction may proceed by a direct β-elimination



leading to benzil (which can rearrange to benzilic acid) and triphenylmethide ion or, alternatively, by a rearrangement to α-tritylbenzoin (VII) followed by a base-catalyzed cleavage of VII to



$$(C_6H_5)_3C\text{—}\overset{\overset{\displaystyle OH}{|}}{\underset{\underset{\displaystyle C_6H_5}{|}}{C}}\text{——}\overset{\overset{\displaystyle O}{\|}}{C}C_6H_5$$

(VII)

benzil and triphenylmethide ion. With the hope of isolating VII, the reaction of VI with potassium hydroxide was allowed to proceed for only five minutes. Triphenylmethane (26%), benzilic acid (33%) and unreacted starting material (64%) were obtained but no VII could be isolated. Attempts to prepare VII by other methods have been unsuccessful.

Experimental[9]

Benzhydryl Desyl Ether (I) (a).—To a solution of 35 g. (0.16 mole) of benzoin in 300 ml. of boiling anhydrous benzene was added 30 g. (0.15 mole) of diphenyldiazomethane[10] and then five drops of concentrated sulfuric acid. An immediate vigorous reaction occurred. After two minutes the evolution of gas had ceased and the red color of the diazo compound had disappeared. The solution was refluxed for five hours and then the benzene was distilled under reduced pressure. The yellow glass was added to 500 ml. of hot alcohol. A small amount of insoluble solid was filtered from the hot solution and recrystallized from benzene to yield 0.5 g. of tetraphenylethylene, m.p. 223–224°. It was converted to the tetrabromo derivative,[11] m.p. 245–249°.

The alcoholic filtrate was treated with 15 g. (0.070 mole) of periodic acid monohydrate in 100 ml. of water for eight hours to remove unreacted benzoin. The solid was then collected, washed with water and recrystallized twice from ethanol, yielding 25 g. (63%) of I (flat needles, m.p. 87.5–88.5°).

Anal. Calcd. for $C_{27}H_{22}O_2$: C, 85.7; H, 5.9. Found: C, 85.9; H, 5.9.

When 0.5 g. (0.0013 mole) of I was refluxed for six hours with a solution of 0.5 g. (0.0025 mole) of 2,4-dinitrophenylhydrazine and 1 ml. of concentrated hydrochloric acid a red solid was obtained which on recrystallization from nitromethane melted with decomposition at 321–322° and had the correct analysis for **benzil 2,4-dinitrophenylosazone.**

Anal. Calcd. for $C_{26}H_{18}O_8N_8$: C, 54.7; H, 3.2; N, 19.6. Found: C, 55.1; H, 3.0; N, 19.4.

This compound could also be obtained from benzil or benzoin by a similar procedure.

When I (1.0 g., 0.0026 mole) was treated under gentle reflux for seven hours with 5 ml. of concentrated hydrochloric acid in 25 ml. of ethanol it was converted to benzoin (0.3 g., 53% yield), m.p. 131–134.5°.

Method (b).—Despite its lower yield, an alternative synthesis, which was a variation of the method of Pratt and Draper[12] was found to be preferable for the preparation of large amounts of I. Benzoin (53 g., 0.25 mole), benzhydrol (46 g., 0.25 mole) and toluenesulfonic acid (0.76 g., 0.004 mole) were heated in benzene in such a way that water was allowed to distil from the solution as it was formed. At the end of 12 hours 82% of the water had collected. An additional 0.76 g. (0.004 mole) of toluenesulfonic acid was added and the reaction was continued for 48 hours but no additional water collected. Unreacted benzoin was converted to benzaldehyde and benzoic acid by oxidation with periodic acid as above. The acid and aldehyde were extracted from ether with sodium bicarbonate solution and saturated aqueous sodium bisulfite. The ethereal solution remaining was then dried and the ether removed to give a yellow oil (81 g.) which was dissolved in 400 ml. of ethanol and 40 ml. of glacial acetic acid to which had been added 27 g. (0.16 mole) of Girard T reagent. The mixture was then refluxed for two hours, poured into 1 l. of ice-water and extracted with ether. To the aqueous solution remaining was added 25 ml. of concentrated sulfuric acid and the solution was allowed to stand for two days at room temperature. The solid which was formed was triturated with 5 g. of periodic acid monohydrate in 100 ml. of water and 200 ml. of ethanol. Recrystallization from ethanol gave 22.7 g. (24% yield) of I, m.p. 86–88°.

Synthesis of α-Benzhydrylbenzoin (II).—The procedure in Method (b) above was used except that 2.0 g. (0.0105 mole) of *p*-toluenesulfonic acid was added initially and the reaction was allowed to proceed for a total of five days. When the reaction was completed the benzene was distilled under reduced pressure to give a yellow solid which was triturated with a solution of 10 g. (0.05 mole) of periodic acid monohydrate in 50 ml. of water and 50 ml. of ethanol. The white solid was filtered and washed twice with ethanol to give 53 g. (56%) of II, m.p. 158–160°. Recrystallization from ethanol raised the m.p. to 161–162°. The

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[6] H. E. Stavely, This Journal, **62**, 489 (1940).

[7] C. W. Shoppee and D. A. Prins, *Helv. Chim. Acta*, **26**, 185 (1943), and succeeding papers.

[8] R. Roger and A. McGregor, *J. Chem. Soc.*, 442 (1934).

[9] All melting points are corrected. Analyses were carried out by the Clark Microanalytical Laboratory, Urbana, Illinois; the Elek Microanalytical Laboratories, Los Angeles, California; and the Schwarzkopf Microanalytical Laboratory, Elmhurst, Long Island.

[10] L. I. Smith, *Organic Syntheses*, **24**, 53 (1944).

[11] W. Manchot and P. Krische, *Ann.*, **337**, 194 (1904).

[12] E. F. Pratt and J. D. Draper, This Journal, **71**, 2846 (1949).

analysis was carried out on a sample of II obtained from the base-catalyzed rearrangement of I described below.

Anal. Calcd. for $C_{27}H_{22}O_2$: C, 85.7; H, 5.9. Found: C, 85.3; H, 5.7.

Reaction of II with Lead Tetraacetate.—Lead tetraacetate (2.6 g., 0.0059 mole) was added to a solution of 2.0 g. (0.0033 mole) in 50 ml. of 90% acetic acid at 50–55°. Benzene (15 ml.) was added to keep the reaction mixture homogeneous and the reaction was continued with stirring for two hours. Concentrated sulfuric acid (0.34 ml.) was added to the cold reaction mixture and the precipitate of lead sulfate filtered. The filtrate was evaporated to dryness to give a white crystalline residue which was dissolved in 100 ml. of ether. Benzoic acid, m.p. 118–120° (0.3 g., 81% based on unrecovered starting material) was obtained by extraction of the ethereal solution with 5% sodium carbonate followed by sublimation. After removal of the benzoic acid the ethereal solution was evaporated to dryness and the solid remaining recrystallized from ethanol to give 0.85 g. (42%) of unreacted II, m.p. 158–160°.

The alcoholic filtrate was diluted with 50 ml. of water and cooled in an ice-bath. The tan solid which formed was collected and recrystallized from ethanol after decolorization with activated charcoal yielding 0.4 g. (49%) of phenyl benzhydryl ketone, m.p. 130–131°. Mixed melting points with an authentic sample were not depressed.

Reaction of I with Potassium Hydroxide.—A solution of 20 g. (0.053 mole) of I in 200 ml. of 1.0 *N* potassium hydroxide in ethanol was refluxed for one hour. The solution became purple immediately and darkened as the reaction proceeded. The hot black solution was filtered, to give a residue of 1.2 g. of II. After recrystallization from ethanol 1.0 g. (5%) of II, m.p. 161–162° was obtained.

Concentration of the alcoholic solution above by distillation of 75 ml. of ethanol under reduced pressure yielded 1.8 g. of white solid which after four recrystallizations from ethanol and two from glacial acetic acid gave 0.5 g. (0.0015 mole) of tetraphenylethane, m.p. 211–213°. Mixed melting points (several compositions) with an authentic sample showed no depression. From the mother liquors an additional 0.5 g. (2.5%) of II was obtained.

The black alcoholic filtrate was then poured into 100 ml. of water, producing a voluminous white precipitate which was collected and recrystallized from ethanol to give **α-hydroxydibenzhydryl ketone** (III) (7.2 g., 36% yield, m.p. 121–122°).

Anal. Calcd. for $C_{27}H_{22}O_2$: C, 85.7; H, 5.9. Found: C, 85.4; H, 6.0.

The aqueous alcoholic filtrate was next extracted with three 100-ml. portions of ether. Removal of the ether and steam distillation followed by recrystallization from isoöctane gave 1.0 g. (18.5%) of benzhydrol, m.p. 65–67°. A mixed m.p. with an authentic sample showed no depression.

Finally, the aqueous extract was acidified and extracted with ether. Evaporation of the ether gave a white solid from which 0.4 g. (0.0033 mole) of benzoic acid, m.p. 122–124°, was separated by sublimation for one hour at 100° at 0.2 mm. pressure.

The residue from the sublimation was recrystallized from water after decolorization with charcoal to give 3.2 g. (51%) of diphenylacetic acid, m.p. 142–147°. Mixed melting points with an authentic sample melted at 143–147°.

Reaction of II with Potassium Hydroxide.—A solution of II (20.0 g., 0.053 mole) was refluxed for 30 minutes in 200 ml. of 1 *N* ethanolic potassium hydroxide. Filtration of the hot solution yielded 0.2 g. (1%) of unreacted II, m.p. 159–161°. Concentration of the solution to three-quarters of its volume followed by the addition of 100 ml. of water yielded crude III (74%, m.p. 110–115°) which was recrystallized from ethanol to give 11.0 g. (55%) of III, m.p. and mixed m.p. with III obtained above 120–122°. Extraction with ether of the filtrate left after the removal of III followed by evaporation of the ether to dryness and recrystallization of the residue from isoöctane gave 0.5 g. (20%) of benzhydrol, m.p. 64–66°. A mixed m.p. with an authentic sample showed no depression.

Acidification of the solution remaining from the ether extraction gave, after repeated recrystallization from water, 0.2 g. of benzilic acid, m.p. 142–147° (mixed m.p. with an authentic sample, 144–150°).

Reaction of III with Lead Tetraacetate.—This oxidation was carried out on 1 g. of III by the same procedure as was used for the oxidation of II described above. Diphenyl acetic acid (0.45 g., 80% yield, m.p. 146–148°) was isolated by extraction with sodium bicarbonate, acidification and recrystallization of the precipitate from water. A series of mixed melting points with an authentic sample showed no depression. The ether was distilled from the neutral fraction and it was heated for one hour under reflux with 1.0 g. of hydroxylamine hydrochloride, 1.0 g. of potassium hydroxide, 5 ml. of water and 25 ml. of ethanol. After filtration of sodium chloride, 5 ml. of water was added and the precipitate of benzophenone oxime obtained by cooling the solution was recrystallized from ethanol–water and then from ligroin. The yield of benzophenone oxime of 0.35 g., m.p. 142–144°, represents 0.32 g. (68%) of benzophenone. A mixed m.p. with an authentic sample showed no depression.

Reaction of III with Potassium Hydroxide.—When III (2.0 g., 0.0053 mole) was refluxed for 90 minutes in 20 ml. of 1.0 *N* potassium hydroxide in ethanol, extraction of the acidic fraction, neutralization and recrystallization of the product from water yielded 0.3 g. (45% based on unrecovered starting material) of diphenylacetic acid, m.p. 144–149°. A mixed m.p. showed no depression. When the ether was removed from the neutral fraction and the residue recrystallized from isoöctane, 0.8 g. (40%) of unreacted III, m.p. 119–121° (mixed m.p. showed no depression) was obtained. When the isoöctane filtrate was cooled in an ice-salt mixture, a yield of 0.2 g. (35%) of benzhydrol, m.p. 63–65°, was obtained. A mixed m.p. with an authentic sample showed no depression.

Reaction of Phenylbenzoyldiazomethane with Benzoin. Desyl *p*-Toluenesulfonate.—In an attempted synthesis of I, a solution of 22 g. (0.10 mole) of phenylbenzoyldiazomethane[13] in 100 ml. of dry benzene was added over one-half hour to 21 g. (0.10 mole) of benzoin and 19 g. (0.10 mole) of *p*-toluenesulfonic acid monohydrate from which the water had been previously distilled. The red color of the diazo compound disappeared instantly. The yellow solution was refluxed for two hours, cooled and washed with water. The benzene was then removed by distillation under reduced pressure and the glassy residue recrystallized from an ethanol–benzene mixture. After three recrystallizations from ethanol and three from a hexane–acetone mixture, 19.3 g. (54% yield) of desyl *p*-toluenesulfonate was obtained, m.p. 107–108.5°. On subsequent runs the product could be obtained in 90% yield by leaving out the benzoin and elimination of the recrystallizations from ethanol. An attempt to synthesize I by treating benzoin with desyl toluenesulfonate and pyridine in benzene (refluxing solution for 24 hours) gave only unreacted starting material (54%).

Anal. Calcd. for $C_{21}H_{18}O_4S$: C, 68.8; H, 5.0; S, 8.5. Found: C, 68.7; H, 5.3; S, 8.7.

Synthesis of Desyl Trityl Ether (VI).—The procedure used was similar to Method B for the synthesis of I. A solution of 26.5 g. (0.125 mole) of benzoin, 34.3 g. (0.130 mole) of triphenylcarbinol and 0.4 g. (0.0002 mole) of *p*-toluenesulfonic acid monohydrate in 500 ml. of benzene was refluxed in the apparatus used above for three days. The solution was washed with sodium bicarbonate solution and the benzene removed under reduced pressure. The residue was triturated with periodic acid (15 g., 0.078 mole) in 200 ml. of ethanol–water. The supernatant liquid was decanted from the yellow oil which after being triturated several times with ethanol crystallized. Recrystallization of the solid from ethanol yielded VI (15 g., 26%, m.p. 127–128°).

Anal. Calcd. for $C_{33}H_{26}O_2$: C, 87.2; H, 5.8. Found: C, 87.4; H, 5.8.

When VI (1.0 g., 0.0022 mole) was heated under reflux with 2 ml. of concentrated hydrochloric acid in 50 ml. of ethanol it was reconverted to benzoin (0.4 g., 85% yield, m.p. 131–134°) and 0.5 g. (82%) of triphenylcarbinol, m.p. 153–157°. Recrystallization from benzene raised the m.p. of the latter to 158–161°. In each case a mixed m.p. with an authentic sample showed no depression.

Reaction of VI with Potassium Hydroxide.—A solution of VI (15.0 g., 0.033 mole) in 200 ml. of 1 *N* potassium hydroxide in ethanol was refluxed for one hour. Concentra-

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[13] C. D. Nenitzescu and E. Solomonica, *Organic Syntheses*, **15**, 62 (1935).

tion of the solution and addition of 100 ml. of water gave 8.5 g. (93%) of triphenylmethane, m.p. 90–93°, after one recrystallization from ethanol. Mixed melting points with an authentic sample were not depressed. Sublimation of the acid fraction for one hour at 100° and 1 mm. pressure gave 0.3 g. of benzoic acid, m.p. 118–120° (no depression with an authentic sample). Crystallization of the residue from water gave 6.6 g. (88% yield) of benzilic acid, m.p. 148–150°. Mixed melting points with an authentic sample showed no depression.

A similar reaction carried out for only five minutes gave a yield of 26% of triphenylmethane, m.p. 85–91° and 33% of benzilic acid, m.p. 149–151°, as well as 64% recovered VI, m.p. 125–128°.

NEW YORK 27, N. Y. RECEIVED NOVEMBER 25, 1950

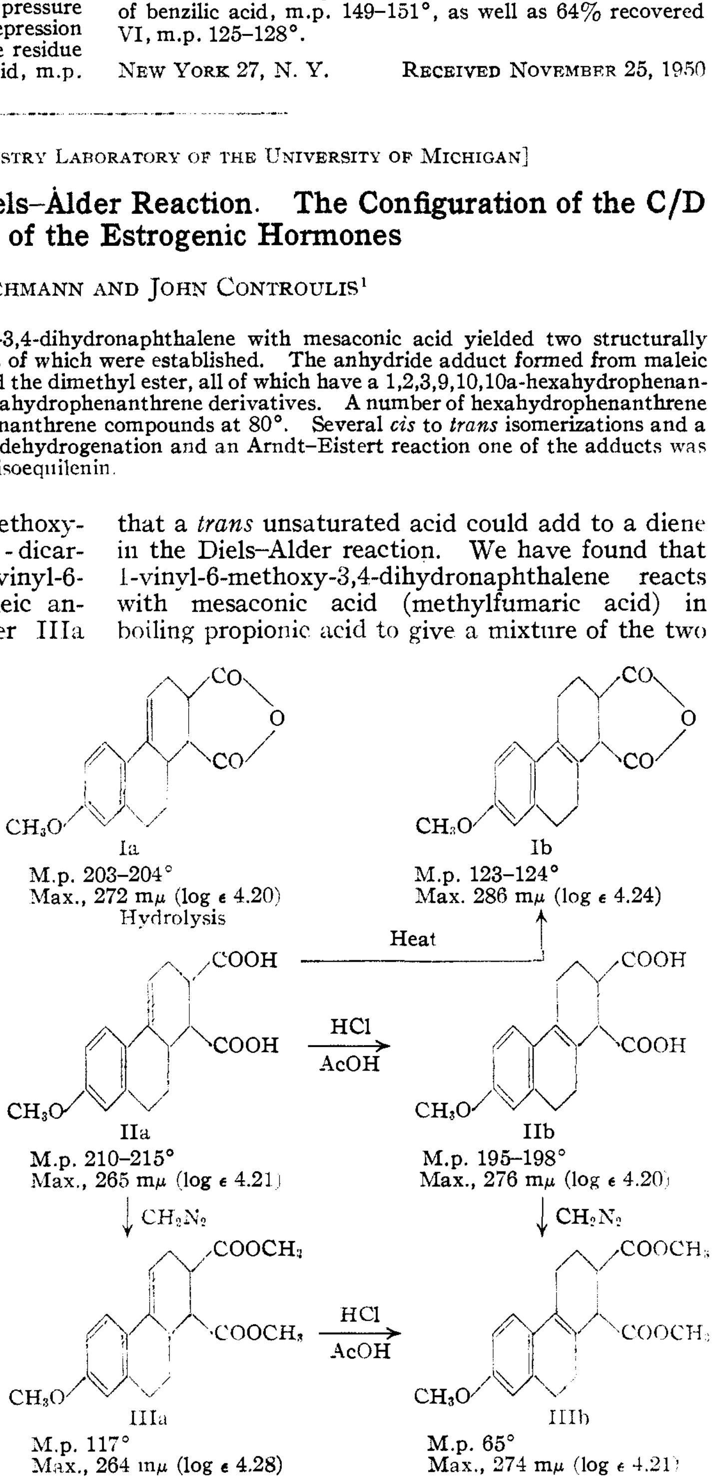
[CONTRIBUTION FROM THE CHEMISTRY LABORATORY OF THE UNIVERSITY OF MICHIGAN]

Synthesis of Isoequilenin via the Diels–Alder Reaction. The Configuration of the C/D Ring Juncture of the Estrogenic Hormones

BY W. E. BACHMANN AND JOHN CONTROULIS[1]

The Diels–Alder reaction of 1-vinyl-6-methoxy-3,4-dihydronaphthalene with mesaconic acid yielded two structurally isomeric adducts, the structures and configurations of which were established. The anhydride adduct formed from maleic anhydride and the diene, the corresponding acid and the dimethyl ester, all of which have a 1,2,3,9,10,10a-hexahydrophenanthrene structure, were isomerized to 1,2,3,4,9,10-hexahydrophenanthrene derivatives. A number of hexahydrophenanthrene compounds were dehydrogenated to tetrahydrophenanthrene compounds at 80°. Several *cis* to *trans* isomerizations and a *trans* to *cis* epimerization were carried out. By dehydrogenation and an Arndt–Eistert reaction one of the adducts was converted into an intermediate in the synthesis of isoequilenin.

Dane and co-workers[2] obtained *cis*-7-methoxy-1,2,3,9,10,10a-hexahydrophenanthrene-1,2-dicarboxylic anhydride (Ia) by the reaction of 1-vinyl-6-methoxy-3,4-dihydronaphthalene with maleic anhydride and prepared the dimethyl ester IIIa of the corresponding acid. By hydrolysis of the anhydride and of the ester we have now prepared the dicarboxylic acid IIa. When the acid was kept at its melting point (215°) a new anhydride was produced. The ultraviolet absorption spectra (Figs. 1 and 2) showed that the double bond had shifted to a new position of conjugation with the aromatic ring and the new anhydride must be *cis*-7-methoxy-1,2,3,4,9,10-hexahydro-1,2-dicarboxylic anhydride (Ib). The new location of the double bond would account for the increase in the position of the absorption maximum. When the acid IIa and its dimethyl ester IIIa were warmed with hydrogen chloride in acetic acid the isomeric acid IIb and ester IIc, respectively, were produced. The correspondence of the new acid and new ester was shown by the formation of the ester from the acid and diazomethane. As shown in Chart I, which shows the interrelationships, the melting points of the members of the "b" series were lower than those of the "a" series and the position of the maxima were shifted to the regions of higher wave length. The curves of the "a" series (Fig. 1) show an inflection or second maximum at 285–290 mμ which is not present in the members of the "b" series (Fig. 2).[3]



Ia
M.p. 203–204°
Max., 272 mμ (log ε 4.20)

Ib
M.p. 123–124°
Max. 286 mμ (log ε 4.24)

IIa
M.p. 210–215°
Max., 265 mμ (log ε 4.21)

IIb
M.p. 195–198°
Max., 276 mμ (log ε 4.20)

IIIa
M.p. 117°
Max., 264 mμ (log ε 4.28)

IIIb
M.p. 65°
Max., 274 mμ (log ε 4.21)

Bachmann and Scott[4] showed unequivocally that a *trans* unsaturated acid could add to a diene in the Diels–Alder reaction. We have found that 1-vinyl-6-methoxy-3,4-dihydronaphthalene reacts with mesaconic acid (methylfumaric acid) in boiling propionic acid to give a mixture of the two structurally isomeric adducts, *trans*-2-methyl-7-methoxy-1,2,3,4,9,10-hexahydrophenanthrene-1,2-dicarboxylic acid (IV) and *trans*-1-methyl-7-methoxy-1,2,3,4,9,10-hexahydrophenanthrene-1,2-dicarboxylic acid (VI). The reaction is slow (four to five days) and the yield of products is low (presumably on account of polymerization or de-

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[1] From the Ph.D. dissertation of John Controulis, June, 1950.

[2] E. Dane, O. Höss, A. W. Bindseil and J. Schmidt, *Ann.*, **532**, 39 (1937).

[3] These results are in agreement with those of J. Heer and K. Miescher, *Helv. Chim. Acta*, **31**, 219 (1948), on related compounds.

[4] W. E. Bachmann and L. B. Scott, THIS JOURNAL, **70**, 1458 (1948).