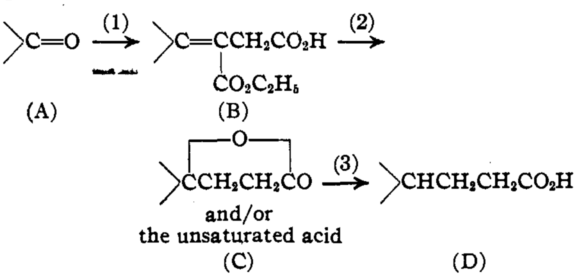
[CONTRIBUTION FROM THE LABORATORY OF ORGANIC CHEMISTRY OF THE UNIVERSITY OF WISCONSIN]

The Stobbe Condensation with 2-Acetylnaphthalene. A Synthesis of 2,4-Dimethylphenanthrene

BY WILLIAM S. JOHNSON, ARTHUR GOLDMAN[1] AND WILLIAM P. SCHNEIDER

In this communication we wish to introduce procedures for (1) the use of potassium *t*-butoxide in the Stobbe condensation[2] and (2) the acid-catalyzed decarbethoxylation of the resulting half-ester (B) to give the γ-lactone or derivative (C). The combination of these two steps followed by reduction (step 3) affords a method of executing an important synthetic process: namely, the introduction of a propionic acid residue at the site of the carbonyl group (A) of a ketone.[3] The general scheme is represented as



$$\rangle C{=}O \xrightarrow{(1)} \rangle C{=}\underset{\substack{|\\ CO_2C_2H_5}}{C}CH_2CO_2H \xrightarrow{(2)}$$

(A) (B)

$$\rangle CCH_2CH_2CO \xrightarrow{(3)} \rangle CHCH_2CH_2CO_2H$$

and/or
the unsaturated acid
(C) (D)

Included in accompanying papers is a description of the application of this chain-lengthening process to the ring ketones tetralone-1[4] and 1-keto-1,2,3,4-tetrahydrophenanthrene.[5] The present work deals with the application of the process to an open-chain ketone, 2-acetylnaphthalene. The preparation of 2,4-dimethylphenanthrene from the resulting lactone II is also described as typifying a use of the chain-lengthening process in polycyclic aromatic synthesis. The general plan bears some resemblance to Cook and Robinson's[6] approach to 8-methyl-1,2-benzanthracene, but differs fundamentally at the decarboxylation step.

The condensation of 2-acetylnaphthalene with diethyl succinate in the presence of sodium ethoxide according to conventional procedures has already been described.[7] When the crude mixture of isomeric half-esters produced in this way was treated with a refluxing mixture of acetic and hydrochloric acids, carbon dioxide was evolved slowly and the crystalline γ-methyl-γ-(2-naphthyl)-butyrolactone, II, was produced in 57–69% yields. In contrast when the Stobbe condensation was conducted in *t*-butyl alcohol containing potassium *t*-butoxide, the reaction was very much more rapid (forty minutes instead of several days), the product was much cleaner, and the yield of practically pure lactone II was 82%.[8] It was found that the decarbethoxylation step could be accelerated considerably by the use of hydrobromic in place of hydrochloric acid. Robinson and Slater[9] have prepared the lactone II by the interaction of β-naphthylmagnesium bromide and ethyl levulinate. The reported yield of crude material was 38%.

For the synthesis of 1,4-dimethylphenanthrene, VIII, the reactions indicated in the accompanying flow sheet were employed. This hydrocarbon has been synthesized previously by two different methods. Akin, Stamatoff and Bogert,[10] using the Pschorr process, obtained it in about 25% over-all yield in six steps from 2,5-dimethylbenzyl chloride. Later Papa, Perlman and Bogert[11] in the same laboratories announced a successful synthesis by the Bogert–Cook method. The picrate of VIII thus was obtained in five steps from bromo-*p*-xylene (and *o*-methylcyclohexanone) in an over-all yield of about 21%. In the present work VIII was obtained in 54% over-all yield in six steps from 2-acetylnaphthalene, I. The lactone II was cleaved with warm dilute sodium hydroxide, and the aqueous solution of the salt of the hydroxy acid III was hydrogenated easily over copper–chromium oxide catalyst. In this way the crystalline γ-2-naphthylvaleric acid, IV, was obtained in 90% yield. From this point the synthesis was conventional. Cyclization with anhydrous hydrogen fluoride proceeded smoothly to give 4-keto-1-methyl-1,2,3,4-tetrahydrophenanthrene, V, in 93% yield. The addition of methylmagnesium iodide to this ketone resulted in a good (86%) yield of the crystalline 1,4-dimethyl-4-hydroxy-1,2,3,4-tetrahydrophenanthrene, VI, which on dehydrogenation over palladium–charcoal catalyst afforded 1,4-dimethylphenanthrene, VIII, in a yield of 91%. The

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[2] The alkoxide-catalyzed aldol type of condensation of succinic ester with a ketone (or aldehyde) was discovered and extensively studied by Stobbe and his collaborators, and is appropriately referred to as the "Stobbe condensation."

[3] For a brief summary of previous methods for effecting this transformation see Haberland and Heinrich, *Ber.*, **72B**, 1222 (1939).

[4] W. S. Johnson, H. C. E. Johnson and J. W. Petersen, THIS JOURNAL, **67**, 1360 (1945).

[5] Johnson and Petersen, *ibid.*, **67**, 1366 (1945).

[6] J. W. Cook and Mrs. A. M. Robinson, *J. Chem. Soc.*, 505 (1938).

[7] Johnson and Goldman, THIS JOURNAL, **66**, 1030 (1944).

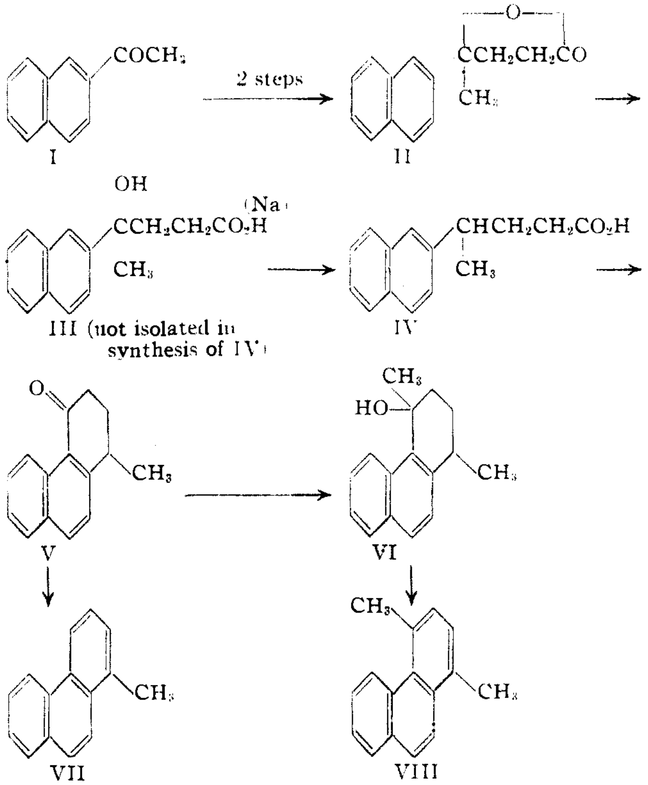
[8] An exhaustive study of the modified Stobbe condensation and the decarbethoxylation reaction is now in progress. The results of experiments with a number of ketones indicate that potassium *t*-butoxide is a superior reagent for the condensation. For example, Gilbert Stork and Chester E. Davis have found that with cyclohexanone 84% yields of the condensation product can be obtained; whereas by the conventional procedure the yield is reported to be about 40%: Alder, Pascher and Schmitz, *Ber.*, **76B**, 27 (1943). For other examples see refs. 4 and 5.

[9] Robinson and Slater, *J. Chem. Soc.*, 376 (1941).

[10] Akin, Stamatoff and Bogert, THIS JOURNAL, **59**, 1268 (1937).

[11] Papa, Perlman and Bogert, *ibid.*, **60**, 319 (1938).

melting points of the hydrocarbon and picrate were in agreement with those previously reported.[10,11]



Additional evidence for the structure of the ketone V was afforded by dehydrogenation over palladium–charcoal in a tube sealed under nitrogen according to a procedure of Fieser and Joshel.[12] The product was shown to be the expected 1-methylphenanthrene, VII, by comparison with an authentic specimen.

Since the modified Stobbe condensation seems to be generally applicable to the synthesis of lactones like II (or the corresponding unsaturated acids) from aryl ketones like I,[8] the scheme of reactions described above promises to be useful in the synthesis of certain types of polycyclic compounds which hitherto have not generally been easily obtained through the conventional cyclization process, because of difficulty experienced in obtaining the requisite γ,γ-disubstituted butyric acid (like IV). In preparing such acids for use in polycyclic syntheses, previous workers[13] have usually employed the reactions of the Grignard reagent with a γ-keto ester, an approach which is not generally satisfactory, and which in at least one instance has been reported to fail altogether.[13c]

The synthesis of 1,2,3,10-tetrahydroperinaphthanone-7[4] affords another example of the application of the process described in the present work. The reduction step was carried out with the unsaturated acid instead of with the lactone.

Experimental[14]

γ-Methyl-γ-(2-naphthyl)-butyrolactone (II). (*a*) *By the Sodium Ethoxide-Catalyzed Stobbe Condensation.*—The best results were realized when the Stobbe condensation was conducted in ether solution for five days with alcohol free sodium ethoxide according to a procedure already described in detail.[7] The crude oily mixture of half-esters obtained from 42.5 g. (0.25 mole) of 2-acetylnaphthalene and 43.5 g. (0.25 mole) of diethyl succinate and 0.5 mole of sodium ethoxide, was treated with 250 cc. of acetic acid and 150 cc. of concentrated hydrochloric acid. After refluxing for fifty-two hours (mercury trap) the solution was concentrated under reduced pressure. The residue was taken up in benzene, washed with water, followed by saturated sodium bicarbonate solution and dried over anhydrous sodium sulfate. Acidification of the bicarbonate extracts gave 10.0 g. of intractable brown acidic material which melted over a wide range, and which was largely unaffected by further acid hydrolysis. The neutral fraction was distilled rapidly in a two-bulb flask, and the small forerun (0.5 g.) was discarded. The main fraction distilled at 185–195° (2–3 mm.) giving a yellow oil which rapidly set to a light yellow solid, m. p. 74–77°, yield 39.0 g. (69%). Recrystallization from benzene–petroleum ether (60–68°) gave colorless plates, m. p. 76–77° (reported,[9] 77°). That this was truly γ-methyl-γ-(2-naphthyl)-butyrolactone follows from the reactions described below in connection with the synthesis of 1,4-dimethylphenanthrene.

When the time for the Stobbe condensation was shortened to three days the yield of crude lactone, m. p. 72–75°, was 65%. In another experiment the condensation was carried out at refluxing temperature for nine hours and the yield dropped to 54%. When a refluxing (nineteen hours) alcoholic solution of sodium ethoxide was used for the condensation according to a procedure already described,[7] the yield of lactone, m. p. 74–76°, was 57%.

(*b*) *By the Potassium t-Butoxide-Catalyzed Stobbe Condensation.*—To a refluxing solution of 2.15 g. (0.055 mole) of potassium in 45 cc. of *t*-butyl alcohol in an atmosphere of nitrogen, was added a mixture of 8.50 g. (0.05 mole) of 2-acetylnaphthalene and 13.1 g. (0.075 mole) of diethyl succinate, an additional 10 cc. of *t*-butyl alcohol being used to complete the transfer. After refluxing for forty minutes the orange-colored solution was chilled. 10 cc. of concentrated hydrochloric acid in 50 cc. of water was added, and the alcohol was removed under reduced pressure. The light yellow oily residue was taken up in ether, and was washed with 5% potassium carbonate solution. Acidification of the aqueous layers liberated the half-ester as a light yellow oil which was extracted with ether, washed with water followed by saturated salt solution, and dried over anhydrous sodium sulfate. The half-ester obtained after complete removal of the ether by warming under reduced pressure was treated with 45 cc. of acetic acid (distilled from potassium permanganate), 30 cc. of 48% hydrobromic acid and 15 cc. of water. After refluxing for three and one-quarter hours a total of about 1200 cc. of gas was collected (over saturated sodium chloride solution), and the rate of evolution had become quite slow. The acid solvents were then removed under reduced pressure and the residual oil was taken up in ether and washed with 5% potassium carbonate solution to remove acidic material. Evaporation of the dried (over anhydrous sodium sulfate), pale yellow ether solution afforded 8.12 g. of almost colorless crystalline lactone, m. p. 65–72°.

The acidic material which was separated by ether extraction of the acidified carbonate extracts amounted to

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(12) Fieser and Joshel, THIS JOURNAL, **61**, 2958 (1939).

(13) See for example (a) R. D. Haworth, *et al.*, *J. Chem. Soc.*, 1784, 2248 (1932); *ibid.*, 454, 864 (1934). (b) Fieser and Johnson, THIS JOURNAL, **61**, 1647 (1939). (c) Fieser and Joshel, *ibid.*, **61**, 2958 (1939). (d) Orcutt and Bogert, *ibid.*, **63**, 127 (1941).

(14) All melting points are corrected.

about 3.8 g. of a semi-solid. This was treated with 15 cc. of acetic acid, 10 cc. of hydrobromic acid and 5 cc. of water. After refluxing for four and one-half hours the solution was worked up as described above affording 2.25 g. of almost colorless lactone, m. p. 69–72°. The acidic fraction from this treatment amounted to 0.97 g., and on rehydrolysis with 9 cc. of acetic acid, 6 cc. of hydrobromic acid and 3 cc. of water for three hours it afforded an additional 0.27 g. (after trituration with 30–40° petroleum ether) of the lactone, m. p. 63–70°, making the total yield of crude lactone 10.64 g. or 94%. Comparable yields were obtained by the slower hydrochloric–acetic acid hydrolysis. Lactone of good quality could be obtained easily by warming the crude product with a slight excess of 2% sodium hydroxide solution for twenty minutes on the steam-bath. A small amount of undissolved oily impurity was removed with ether, and the aqueous layer after treatment with Norit was made acid (to congo red). This mixture was warmed on the steam-bath for one and one-half hours to relactonize the hydroxy acid, and then worked up by ether extraction, washing with saturated sodium bicarbonate, and drying. The recovery of colorless lactone, m. p. 73–75°, was 90% making the over-all yield of this material 85%. For catalytic reduction this material was recrystallized from ether–petroleum ether; m. p. 75–76°; over-all yield from I, 82%.

The neutral fraction which remained after the original Stobbe condensation amounted to 3.10 g. Distillation afforded 2.25 g. of liquid boiling at 210–215°, which was probably diethyl succinate. The higher-boiling fraction amounted to 0.3 g. of oil which on treatment with semicarbazide gave 0.13 g. of crude semicarbazone of 2-acetylnaphthalene, m. p. about 235° (dec.).

γ-Hydroxy-γ-(2-naphthyl)-valeric Acid (III).—A mixture of 1.60 g. of γ-methyl-γ-(2-naphthyl)-butyrolactone (II), and 25 cc. of 5% sodium hydroxide solution was warmed on the steam-bath until solution was complete. The solution was filtered, chilled, and then acidified slowly with dilute hydrochloric acid, care being taken to avoid an excess of mineral acid. The colorless crystalline hydroxy acid was washed thoroughly with water and dried at room temperature; yield 1.61 g. (93%); m. p. 101–105°. Because of its susceptibility to reversion to the lactone, the acid was recrystallized with considerable difficulty. When benzene was used as the solvent in flasks which had been washed free of traces of mineral acid it was possible to effect the recrystallization if the operation was carried out rapidly. After three recrystallizations the hydroxy acid was obtained as colorless needles, m. p. 109–110°. On standing, this material slowly underwent cyclodehydration to the lactone.

Anal. Calcd. for $C_{15}H_{16}O_3$: C, 73.75; H, 6.60. Found: C, 73.78; H, 6.58.

γ-(2-Naphthyl)-valeric Acid (IV).—Fieser and his coworkers[15] have successfully reduced β-aroylpropionic acid derivatives by hydrogenation of an aqueous solution of the sodium salt over copper–chromium oxide catalyst. It was expected, therefore, that the salt of a γ-aryl-γ-hydroxybutyric acid (like III) would be susceptible to reduction under similar conditions. This was indeed realized. A mixture of 34.4 g. (0.152 mole) of the lactone I and a slight excess of approximately 1 *N* sodium hydroxide solution was warmed until solution was complete. Acetic acid was added until the solution was only faintly basic to litmus, and the solution was filtered into a bomb of 500 cc. capacity. The hydrogenation was conducted in the presence of 5.0 g. of copper–chromium oxide catalyst (37 KAF) and at an initial pressure of 147 atmospheres of hydrogen. Hydrogenation proceeded rapidly at 160° and was complete after two and one-half hours. When higher temperatures were used the reduction went too far, presumably involving the naphthalene ring, and the product could not be crystallized. The filtered solution was acidified with an excess of hydrochloric acid and the mixture was boiled for one-half hour to decompose any of the high-melting material (see below). The acid was extracted with benzene and dried over anhydrous sodium sulfate. The solution was concentrated and petroleum ether (60–68°) was added to the warm solution to the point of incipient cloudiness. The reduced acid crystallized on cooling, m. p. 69–71°; total yield 31.2 g. (90%) of material suitable for the next step. Twice recrystallized, a sample of the acid was obtained in the form of colorless microscopic crystals, m. p. 70.5–71°, depressed on admixture with the lactone II.

Anal. Calcd. for $C_{15}H_{16}O_2$: C, 78.92; H, 7.07. Found: C, 78.79; H, 6.86.

In one experiment 30.0 g. of the lactone was hydrogenated over 4 g. of catalyst as described above. The solid product obtained on acidification was collected directly without heating. It amounted to 29.2 g., m. p. 61–68°. Crystallization from benzene–petroleum ether (60–68°) gave 13.5 g. of colorless solid, m. p. 94–100°. From the filtrate there was obtained 14.8 g. of fairly pure acid IV, m. p. 69–72°, not depressed on admixture with the analytical sample described above. The first crop was triturated with 300 cc. of boiling ether, and only about half of the material dissolved. Evaporation of the ether afforded an additional 7.60 g. of the acid IV, m. p. 68–70°. The ether-insoluble fraction was amorphous and amounted to 5.61 g., m. p. 182–186°. It was insoluble also in benzene and in chloroform, but dissolved in alcohol. Attempts to crystallize the substance from alcohol, however, were unsuccessful. It dissolved in aqueous alkali, and had a high neutral equivalent (929 and 932). A residue remained after the material was burned, suggesting that it might possibly be an acid salt.[16] The substance was not further investigated after it was found that it was easily decomposed into the desired acid IV by the action of warm mineral acid. A mixture of 2.21 g. in 5 cc. of hydrochloric acid was allowed to reflux for one-half hour. The solid which formed on cooling amounted to 2.07 g. of the acid IV, m. p. 69–72°, not depressed on admixture with pure IV.

4-Keto-1-methyl-1,2,3,4-tetrahydrophenanthrene (V).—A solution of 18.1 g. of γ-(2-naphthyl)-valeric acid (IV) in approximately 200 g. of anhydrous hydrogen fluoride was allowed to stand in a platinum vessel overnight. The solution was evaporated in a current of air, and the residue was taken up in ether, washed with 10% sodium carbonate solution, saturated salt solution, and dried over anhydrous potassium carbonate. Distillation of the oil obtained upon evaporation of the ether afforded 15.5 g. (93%) of slightly yellow, oily ketone, b. p. 179–181° (4–5 mm.). This material was satisfactory for the next step. Redistillation through a modified Widmer column gave colorless ketone, b. p. 145° (0.4–0.5 mm.).

Anal. Calcd. for $C_{15}H_{14}O$: C, 85.68; H, 6.71. Found: C, 85.63; H, 6.57.

The **semicarbazone** was prepared by the usual procedure in alcohol with semicarbazide hydrochloride and pyridine. After recrystallization from dilute alcohol it was obtained as colorless needles, m. p. 210–211° (dec.).

Anal. Calcd. for $C_{16}H_{17}ON_3$: C, 71.88; H, 6.41. Found: C, 71.89; H, 6.50.

1,4-Dimethyl-4-hydroxy-1,2,3,4-tetrahydrophenanthrene (VI).—A solution of 5.04 g. of the ketone V from the experiment described above in 20 cc. of dry benzene was added to a cooled solution of methylmagnesium iodide (from 1.4 g. of magnesium and 3.6 g. of methyl iodide) in 20 cc. of dry ether. The mixture was allowed to stand in the refrigerator overnight, and then the yellow supernatant liquid was decanted from the colorless salt cake onto ice. Excess 10% ammonium chloride solution was added to decompose the addition complex, and the organic layer was separated, washed with water, saturated salt solution, and dried over anhydrous potassium carbonate. The residue obtained on evaporation was recrystallized rapidly from ether–petroleum ether (b. p. 60–68°) in acid-

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(15) Fieser and Cason, THIS JOURNAL, **62**, 1293 (1940); Fieser and Daudt, *ibid.*, **63**, 782 (1941).

(16) *Cf.* The complex acid salt of abietic acid; Dupont, Desalbres and Bernette, *Bull. inst. du Pin*, No. **22**, 349 (1926).

free glassware; yield 0.86 g. of colorless carbinol, m. p. 126–128°. The aforementioned salt cake was treated with ice and ammonium chloride and worked up as described above. The crystallized carbinol thus obtained amounted to 3.81 g. of colorless microscopic crystals, m. p. 129–131°. The total yield was 4.67 g. or 86%. A sample purified for analysis by two recrystallizations from the same medium was obtained as colorless plates, m. p. 132–133°.

Anal. Calcd. for $C_{16}H_{18}O$: C, 84.91; H, 8.02. Found: C, 84.97; H, 8.25.

1,4-Dimethylphenanthrene (VIII).—A mixture of 4.50 g. of the carbinol VI and 0.45 g. of 30% palladium–charcoal catalyst[17] was heated at 310–320° for one hour by which time 96% of the calculated volume of hydrogen was evolved. The residue was dissolved in ether, filtered and evaporated. After trituration with a few cc. of cold methanol the residue was obtained as colorless needles, m. p. 49–50.5°; yield 3.14 g. From the triturate was obtained an additional 0.59 g. of the hydrocarbon, m. p. 47.5–49.5°, making the total yield 91%. After recrystallization from methanol a sample melted at 50–51° (reported,[11] 49.5–50°). A picrate was prepared according to the reported procedure,[11] and the product had the m. p. 142.5–143.5° (reported,[11] 143.5°).

1-Methylphenanthrene (VII).—According to the procedure of Fieser and Joshel[12] for the dehydrogenation of 1-keto-2-methyl-1,2,3,4-tetrahydrotriphenylene, a mixture of 1.04 g. of 4-keto-1-methyl-1,2,3,4-tetrahydrophenanthrene (V) and 0.1 g. of 30% palladium–charcoal[17] was sealed in a tube filled with nitrogen. After heating at 320–340° for six to seven hours, the mixture was triturated with ethanol and filtered. On concentration and dilution the filtrate yielded 0.62 g. of yellow needles, m. p. 107–115°. This product was purified by conversion to the picrate and cleavage of the latter in benzene by passage through a tower of activated alumina. In this way 0.42 g. (44% yield) of colorless plates of 1-methylphenanthrene was obtained. The m. p. 116.5–118° (reported,[18] 118°) was not depressed when the sample was mixed with an authentic specimen of the hydrocarbon prepared by Dr. F. C. Meyer from 1-keto-1,2,3,4-tetrahydrophenanthrene.

Summary

Potassium *t*-butoxide has been found to be an excellent agent for promoting the Stobbe condensation of a ketone with diethyl succinate.

Procedures are given for the introduction of a propionic acid residue at the site of the carbonyl group of an aryl ketone. The steps involve (1) a Stobbe condensation with potassium *t*-butoxide, (2) acid-catalyzed decarbethoxylation of the resulting half-ester and (3) reduction.

An application of the above process to the synthesis of 2,4-dimethylphenanthrene from 2-acetylnaphthalene is described.

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[17] Linstead and Thomas, *J. Chem. Soc.*, 1127 (1940).

[18] R. D. Haworth, *ibid.*, 1125 (1932).

[CONTRIBUTION FROM THE LABORATORY OF ORGANIC CHEMISTRY OF THE UNIVERSITY OF WISCONSIN]

The Stobbe Condensation with Tetralone-1. A Synthesis of 3'-Keto-3,4-dihydro-1,2-cyclopentenonaphthalene[1]

BY WILLIAM S. JOHNSON, HOWARD C. E. JOHNSON[2] AND JACK W. PETERSEN

In this communication is described the first phase of a study of the use of the Stobbe condensation[3] to build up the cyclopentanone fused ring structure which is characteristic of ring D of many of the sex hormones. By this method we have succeeded in synthesizing 3'-keto-3,4-dihydro-1,2-cyclopentenonaphthalene, VI, from tetralone-1, I, in 52–54% over-all yield (Scheme A) by a process which can be carried out in a few hours. The method promises to be applicable to the synthesis of molecules more closely resembling the steroids.[4]

The various reactions which were studied are outlined in the accompanying flow sheet. The condensation of tetralone-1 (I) with diethyl succinate proceeded well in the presence of sodium ethoxide, but the reaction was much improved by the use of potassium *t*-butoxide as the condensing agent.[5] The crystalline half-ester II thus was obtained in 89–94% yields. Treatment of II with a mixture of acetic anhydride and acetic acid containing a catalytic amount of zinc chloride[6] resulted in ring closure (Scheme A) not into the aromatic nucleus, but into the unsaturated alicyclic ring to produce a keto ester, probably III (position of double bond uncertain). Like a β-keto ester (of which formula III is a vinylog) the cyclized product was decarbethoxylated readily by the action of a mixture of hydrochloric and acetic acids to give the crystalline 3'-keto-3,4-dihydro-1,2-cyclopentenonaphthalene, VI. For the over-all synthesis it was unnecessary to isolate the keto ester III; the decarbethoxylation step (III → VI) could be effected simply by the addition of dilute hydrochloric acid to the cyclization mixture followed by a short period of heating. In this way the ketone VI was obtained as the semicarbazone in 58% yield from the half-ester II.

Evidence for the structure of the ketone VI was afforded by oxidation with permanganate to phthalic acid. Had the ring closed into the benzene nucleus, the expected oxidation product would have been benzene-1,2,3-tricarboxylic acid. The nuclear structure of VI was proved conclusively by sulfur dehydrogenation to the known

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[1] This work was assisted, in part, by a grant from the Wisconsin Alumni Research Foundation.

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[3] *Cf.* Johnson, Goldman and Schneider, THIS JOURNAL, **67**, 1357 (1945).

[4] Johnson and Petersen, *ibid.*, **67**, 1366 (1945).

[5] See ref. 3, footnote 8.

[6] *Cf.* Fieser and Hershberg, THIS JOURNAL, **59**, 1028 (1937).