2-Methyl-3-*n*-octadecylnaphthalene.—Two grams of the above hydrocarbon was subjected to sulfur dehydrogenation as already described for the preparation of 2-hexa· decylnaphthalene. After crystallization from acetone 0.7 g. (35%) of a product was obtained melting at 47–48°.

Anal. Calculated for $C_{29}H_{46}$: C, 88.25; H, 11.75. Found: C, 88.60; H, 11.30.

2-Methyl-3-*n*-octadecyl-1,4-naphthoquinone.—The oxidation of 0.5 g. of the above hydrocarbon with chromic acid gave 0.2 g. (37%) of quinone which melted at 95–97° after recrystallization from ether.

Anal. Calculated for $C_{29}H_{44}O_2$: C, 82.0; H, 10.4. Found: C, 81.7; H, 10.2.

Summary

The preparation and vitamin K activity of a number of naphthoquinones is described. None of these compounds surpasses or even approaches 2-methyl-1,4-naphthoquinone in activity.

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o-Halide Synthesis of 10-Methyl-1',9-methylene-1,2-benzanthracene

BY LOUIS F. FIESER AND JAMES CASON[1]

Since attempts to introduce methyl or hydroxyl groups into the interesting and reactive 1',9-methylene-1,2-benzanthracene[2] (XIII) by direct substitution offered little promise of success a second synthesis was investigated which affords a route not only to the parent hydrocarbon but to various 10-substituted derivatives. The method embodies the general scheme of synthesis of anthracene derivatives introduced by one of us and Seligman.[3] This involves the preparation, through a Grignard reaction and reduction, of a suitable *o*-halide of a diphenylmethane, conversion through the nitrile to the acid and ring closure. The halogen may be introduced in various sequences into either aromatic ring, giving considerable flexibility to the method.

For the present application of the *o*-halide synthesis 7-acenaphthenone (IV) was required as starting material, and as the ketone heretofore has been accessible only by quite laborious processes,[4] a new method was sought. Marquis[5] observed that the corresponding alcohol is produced in low yield in the form of the acetate by the oxidation of acenaphthene with lead dioxide in acetic acid, and in connection with a study of the action of lead tetraacetate on other hydrocarbons[6] Dr. E. B. Hershberg noted that 7-acenaphthenyl acetate can be obtained more smoothly

with the use of this reagent.[7] M. E. Gross then worked out a practical procedure for conducting the oxidation and this was employed initially in the present investigation. The experience of treating quantities of red lead with acetic acid for the preparation of a reagent for use in this solvent suggested that the operation might be dispensed with, and indeed it was found that acenaphthene can be converted smoothly and easily into the 7-acetoxy compound with red lead and acetic acid. The yield is equal to that obtained with purified lead tetraacetate, and considerable time is saved. The acetate is a liquid and is not easily freed from traces of acenaphthene and acenaphthenone, but pure crystalline 7-acenaphthenol is readily obtained in good over-all yield on saponification. Under the most favorable conditions found, oxidation with chromic acid and steam distillation of the product gave the pure ketone in reasonably good yield, and by this sequence of reactions 7-acenaphthenone is obtainable in quantity from the hydrocarbon in 47% over-all yield.

The further stages of the synthesis are indicated in the chart. The carbinol (V) resulting from the action of *o*-chlorophenylmagnesium bromide on 7-acenaphthenone was obtained in a crystalline condition only with considerable loss; the dehydration of the crude reaction product with acetic acid proceeded smoothly enough but in rather low over-all yield. The unsaturated compound VI crystallizes in brilliant orange prisms, while its dihydride VII is a colorless solid of lower

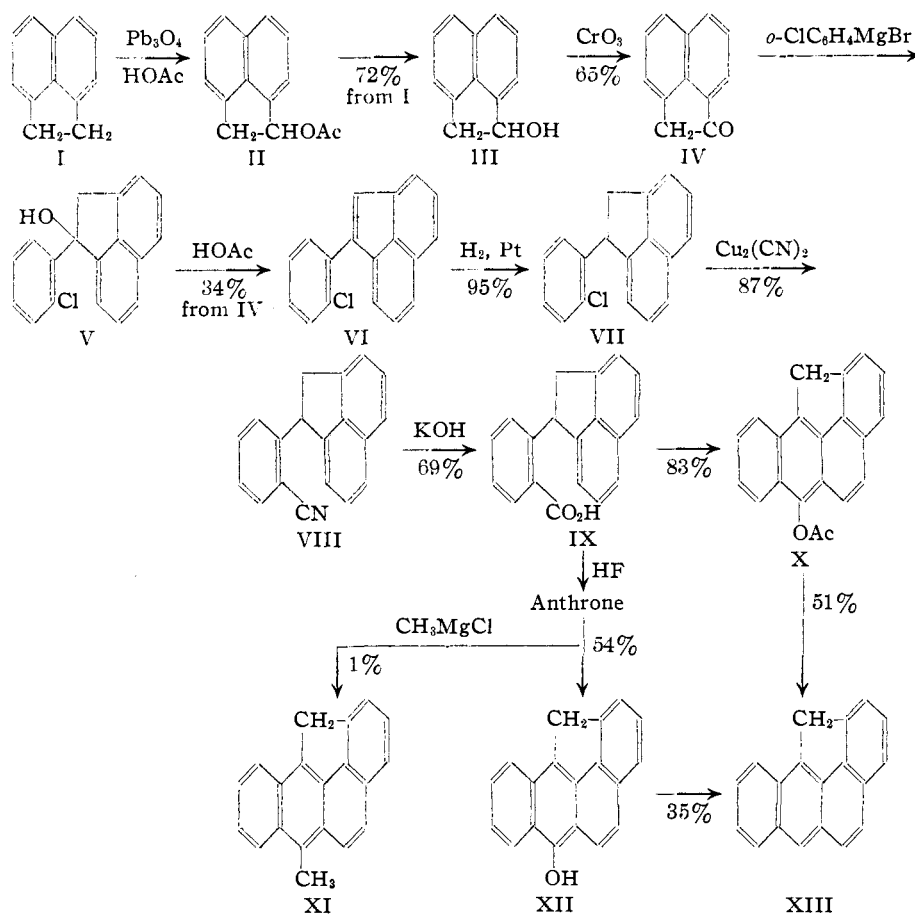(1) Research Fellow on funds from the National Cancer Institute.
(2) Fieser and Cason, THIS JOURNAL, **61**, 1740 (1939).
(3) Fieser and Seligman, *ibid.*, **61**, 136 (1939).
(4) Ewan and Cohen, *J. Chem. Soc.*, **55**, 580 (1889); Graebe and Gfeller, *Ann.*, **276**, 12 (1893); Graebe and Jequier, *ibid.*, **290**, 197 (1896); Badische Anilin u. Soda-Fabrik, German Patent 230,237 (1910); Ghigi, *Gazz. chim. ital.*, **68**, 184 (1938).
(5) Marquis, *Compt. rend.*, **182**, 1227 (1926).
(6) Fieser and Hershberg, THIS JOURNAL, **60**, 1893, 2542 (1938).

(7) Our results appear to be at variance with the observations reported by Monti, *C. A.*, **33**, 9316 (1939).

$$\underset{\substack{\text{CH}_2\text{-CH}_2 \\ \text{I}}}{} \xrightarrow[\text{HOAc}]{\text{Pb}_3\text{O}_4} \underset{\substack{\text{CH}_2\text{-CHOAc} \\ \text{II}}}{} \xrightarrow[\substack{72\% \\ \text{from I}}]{} \underset{\substack{\text{CH}_2\text{-CHOH} \\ \text{III}}}{} \xrightarrow[65\%]{\text{CrO}_3} \underset{\substack{\text{CH}_2\text{-CO} \\ \text{IV}}}{} \xrightarrow{o\text{-ClC}_6\text{H}_4\text{MgBr}}$$

$$\underset{\text{V}}{} \xrightarrow[\substack{34\% \\ \text{from IV}}]{\text{HOAc}} \underset{\text{VI}}{} \xrightarrow[95\%]{\text{H}_2,\ \text{Pt}} \underset{\text{VII}}{} \xrightarrow[87\%]{\text{Cu}_2(\text{CN})_2}$$

$$\underset{\substack{\text{CN} \\ \text{VIII}}}{} \xrightarrow[69\%]{\text{KOH}} \underset{\substack{\text{CO}_2\text{H} \\ \text{IX}}}{} \xrightarrow{83\%} \underset{\substack{\text{OAc} \\ \text{X}}}{}$$

IX $\xrightarrow{\text{HF}}$ Anthrone

CH$_3$MgCl ——— 54%

XI (CH$_3$)   1%   XII (OH)   51%   X

XII $\xrightarrow{35\%}$ XIII

melting point. Conversion to the nitrile VIII proceeded well, and saponification of this rather hindered derivative was accomplished satisfactorily by refluxing with alcoholic alkali for a prolonged period (ten days). Decomposition occurred at higher temperatures, and other methods of hydrolysis were tried without success. The acid IX was cyclized in good yield to 10-acetoxy-1',9-methylene-1,2-benzanthracene (X) by the zinc chloride–acetic acid–anhydride procedure,[8] and the acetate on reduction with zinc and alkali afforded 1',9-methylene-1,2-benzanthracene (XIII), identical with the material previously described.[2] The over-all yield from acenaphthene in ten steps is 3.8%, as compared with 3.4% by the previous five-step synthesis.[2]

For the preparation of the 10-methyl compound XI it was desirable to effect cyclization of the acid IX under conditions most favorable for retaining the product in the anthrone form and avoiding enolization. The hydrogen fluoride method[9]

appears superior to any other and has given good service in the synthesis of simpler 10-alkyl-1,2-benzanthracenes (56% yield), while the introduction of 9-alkyl groups is beset by greater difficulties and the yields are lower (14–35%[10]). The present case seems to be the most unfavorable yet encountered, for in a number of experiments under varying conditions cyclization of IX with hydrogen fluoride and treatment of the crude or crystallized product with methyl Grignard reagent afforded the hydrocarbon XI in no better than 1.2% yield, the bulk of the material being recovered as the crystalline anthranol XII. The anthranol is a sensitive substance and the yield on reduction to the parent hydrocarbon is lower than when the more stable acetate X is employed. By using adequate amounts of materials, a small supply of pure, pale orange 10-methyl-1',9-methylene-1,2-benzanthracene (XI), m. p. 181–181.4°, was obtained and the hydrocarbon is being tested for carcinogenic activity, along with the 10-hydroxy compound XII.

(8) Fieser and Hershberg, THIS JOURNAL, 59, 1028 (1937).
(9) Fieser and Hershberg, ibid., 61, 1272 (1939).
(10) Fieser and Hershberg, ibid., 62, 49 (1940).

The new hydrocarbon is of special interest because of its structural relationship to the potently carcinogenic 9,10-dimethyl-1,2-benzanthracene.[11]

Experimental Part[12]

7-Acenaphthenol (With M. E. Gross).—The "95% acenaphthene" (m. p. 92.5–93.5°) supplied by the Reilly Tar and Chemical Co. proved to be just as satisfactory for the preparation as recrystallized material (m. p. 93–93.5°) or a purer commercial grade (m. p. 93–93.5°). A solution of 154 g. of the crude hydrocarbon in 1.1 l. of glacial acetic acid (distilled over permanganate) was stirred mechanically at a temperature of 60–70° (maintained throughout the reaction) and treated with 820 g. of red lead (85–90%), added in 50-g. portions in the course of about forty minutes, a fresh addition being made on discharge of the red coloration. Very shortly after the addition of the oxidizing agent the test for lead tetraacetate with moistened starch–iodide paper was negative and the dark red, viscous solution was thereupon poured into 2 liters of water. The precipitated oil was extracted with 350 cc. of ether and then with 250 cc. of this solvent, and the washed extract was shaken with saturated sodium chloride solution and filtered through a layer of anhydrous sodium sulfate. After removal of the solvent, the 7-acenaphthenyl acetate was distilled at 5 mm., b. p. 166–168° from a bath at 180–185°; the yield of material (containing small amounts of acenaphthene and acenaphthenone) was 170–175 g. The result was the same using lead tetraacetate.

For hydrolysis the acetate was refluxed with 40 g. of sodium hydroxide in 400 cc. of water and 275 cc. of methanol for two hours. Crystalline acenaphthenol began to separate almost immediately and the liquor acquired a dark violet color probably due to the presence of acenaphthenone. After cooling to 20° the product was collected, washed well with water, dried and recrystallized from benzene, using 2 liters of solvent, and after clarification with Norite concentrated to a volume of 1 liter. The first crop amounted to 117–121 g. of pure acenaphthenol in the form of colorless needles, m. p. 144.5–145.5°, unchanged on further crystallization from benzene or alcohol, and the mother liquor afforded 3–5 g. of pure material; yield 70.5–74% (from acenaphthene). The melting points 146°[13] and 148°[5] have been reported.

7-Acenaphthenone (With M. E. Gross).—A suspension of 100 g. of acenaphthenol in 300 cc. of glacial acetic acid was stirred mechanically and treated with a solution prepared by dissolving 43 g. of chromic anhydride in the minimum amount of water and diluting with 240 g. of acetic acid. The solution was added in the course of fifty minutes while maintaining a reaction temperature of 28–32° by external cooling. The acenaphthenol dissolved giving a green solution and, after stirring for an additional hour at the same temperature, this was poured into 6 liters of ice water and the precipitated ketone collected on

a large Büchner funnel and washed well with water. As purification by direct crystallization or by vacuum distillation proved unsatisfactory, the moist product was distilled with steam, a method which proved very satisfactory. The distillate (35 liters) was cooled below 20° and the practically colorless ketone collected and dried. The acenaphthenone thus obtained melted at 118.5–120.5° and was satisfactory for the synthesis; the average yield in three runs was 64.5 g. (65%). Recrystallization from benzene–hexane (1:4) gave colorless needles, m. p. 121–121.5°.

7-(o-Chlorophenyl)-acenaphthylene (VI).—After trying numerous variations the following procedure was adopted as the most satisfactory. A solution of 45 g. of acenaphthenone in 125 cc. of benzene and 70 cc. of ether was added in fifteen minutes to the Grignard reagent from 7.8 g. of magnesium, 67.6 g. of o-bromochlorobenzene and 250 cc. of ether. After refluxing and stirring for forty-five minutes longer, the complex was decomposed with 55 cc. of saturated ammonium chloride solution and the benzene–ether layer was decanted from the pasty salt mass. By distilling most of the solvent and triturating the residue with hexane, crude **7-(o-chlorophenyl)-acenaphthenol-7 (V)** was obtained as an almost colorless powder. By recrystallization from benzene the carbinol was obtained as colorless, elongated prisms, m. p. 216–218° with decomposition to the orange acenaphthylene derivative, but the yield of crystalline product was only 15–20% and the material was not analytically pure. For the best over-all yield it was found expedient to dehydrate the crude product of the Grignard reaction. For this purpose the initial benzene–ether extract was concentrated on the steam cone and the solvent removed by heating to 70° at the water-pump. The reddish-orange oil remaining was refluxed with 225 cc. of glacial acetic acid for one hour and the dark solution poured into 500 cc. of water and extracted with benzene. After washing the extract free of acid it was filtered through anhydrous sodium sulfate, concentrated and passed through a 2.5 × 13-inch tower of activated alumina and Supercel (1:1). The column was washed with benzene until the dark orange zone due to the unsaturated chloro compound (which migrates rapidly) had passed into the filtrate. The column must be large enough to retain a more strongly adsorbed substance forming a broad yellow zone. The benzene in the filtrate was replaced by methanol and the product crystallized from a volume of about 350 cc. with seeding and slow cooling. This gave 20.5–22.5 g. of orange elongated prisms, m. p. 100–102° (softening at about 96°). No further crystalline material was obtained from the mother liquor on concentration, but on removing the solvent and distilling the residue at 2 mm. a red oil was obtained which yielded 3 g. of satisfactory product, m. p. 98–102°; the total yield of material suitable for the hydrogenation was 33–36%. When the total crude reaction product was distilled directly the yield fell to 15%, and attempted crystallization of material dehydrated with acetic acid but not purified by chromatographic adsorption proved difficult and unsatisfactory.

For analysis a sample of distilled material was crystallized three times from methanol and obtained as yellow-orange cubic or elongated prisms, m. p. 104–104.4°.

(11) Bachmann and Chemerda, THIS JOURNAL., **60**, 1023 (1938); Newman, *ibid.*, **60**, 1141 (1938).

(12) All melting points are corrected. Microanalyses by Lyon Southworth except as indicated.

(13) V. Braun and Bayer, *Ber.*, **59** 920 (1926).

Anal.[14] Calcd. for $C_{18}H_{11}Cl$: C, 82.25; H, 4.24; Cl, 13.52. Found: C, 82.15; H, 4.31; Cl, 13.33.

7-(o-Chlorophenyl)-acenaphthene (VII).—A solution of 32 g. of VI in 155 cc. of purified glacial acetic acid and 15 cc. of absolute ether was shaken with hydrogen at atmospheric pressure in the presence of 0.8 g. of Adams catalyst. The reaction was stopped after the absorption of one mole of hydrogen (three and one-half hours), for after saturation of the external double bond the aromatic nucleus is hydrogenated rather rapidly. After filtering and removing the solvent, the product distilled at 190–192° at 2 mm. (bath at 208–212°) as a light yellow oil which crystallized rapidly; yield 30.6 g. (95%). This material, m. p. 68–74°, was satisfactory for the next step. After three crystallizations from methanol a sample formed colorless elongated prisms melting constantly at 81–82°.

Anal.[15] Calcd. for $C_{18}H_{13}Cl$: Cl, 13.42. Found: Cl, 13.64.

o-(7-Acenaphthyl)-benzonitrile (VIII).—A mixture of 35.6 g. of the chloro compound VII, 11.5 g. of cuprous cyanide (dried in vacuum at 100°), 3 cc. of acetonitrile and 31 cc. of pyridine was heated in an autoclave under nitrogen (initial pressure, 800 pounds) at 243–245° for twenty-five hours. (Longer heating lowers the yield: thirty-five hours, 74.5%; forty-eight hours, 66.5%; a run conducted for sixty hours at 255° gave a 20% yield of a very poor product). The reaction mixture was shaken with dilute ammonia solution and benzene and after filtering the two-phase system with suction, the benzene layer was separated, washed with ammonia solution, water and dilute hydrochloric acid, filtered by suction, and washed free of acid. The residue from the benzene solution was distilled at 3 mm., the nitrile passing over as a reddish oil at 208–212° (bath, 220–230°); yield 25.9 g. (87%). The sample for analysis was passed through a tower of alumina in benzene solution and crystallized three times from hexane. It formed small, colorless needles, m. p. 79.7–80.5°. Impure samples often melt at higher temperatures.

Anal.[15] Calcd. for $C_{19}H_{13}N$: N, 5.49. Found: N, 5.55.

o-(7-Acenaphthyl)-benzoic Acid (IX).—A solution of 25.9 g. of distilled nitrile and 164 g. of potassium hydroxide in 1050 cc. of alcohol and 590 cc. of water was refluxed for 250 hours in a copper flask. The alcohol was expelled by distillation, water being added to maintain a volume of about 1200 cc., and the resulting hot aqueous solution was treated with Norite. The dark filtrate was acidified and cooled and the crude acid which separated was dried, refluxed in benzene with Norite and crystallized twice from benzene–hexane (1/1), giving 18 g. of acid crystallizing in soft, snow-white needles, m. p. 194–195°. The final mother liquor afforded 0.9 g. of pure product; total yield 69%. An additional crystallization from benzene–hexane gave a sample of constant m. p. 195–195.5°.

Anal. Calcd. for $C_{19}H_{14}O_2$: C, 83.18; H, 5.14. Found: C, 82.94; H, 5.29.

Attempts to shorten the reaction time by conducting the hydrolysis under pressure at higher temperatures resulted in extensive decomposition. After seventeen

hours at 120° only 5% of acid could be isolated from the tarry product, and on heating for sixty-five hours at 100° extensive decomposition was evident and only 40% of the acid could be isolated. On one occasion the nitrile was saponified as described, but for only one hundred hours; an oil separated after distillation of the alcohol and this crystallized on prolonged scratching. Crystallization from benzene–hexane (Norite) afforded o-(7-acenaphthyl)-benzamide, m. p. 177–180°, in 32.5% yield. Three crystallizations from benzene gave nearly colorless blades, m. p. 182–182.8°.

Anal.[15] Calcd. for $C_{19}H_{15}ON$: N, 5.13. Found: N, 4.98.

The alkaline filtrate from the amide on suitable working afforded the acid in 40.5% yield. Treatment of the amide with amyl nitrite in acetic acid solution gave only a non-acidic, amorphous red material.

10-Acetoxy-1',9-methylene-1,2-benzanthracene (X).—A mixture of 0.5 g. of the acenaphthylbenzoic acid IX, 3 cc. of glacial acetic acid, 2.5 cc. of acetic anhydride and a trace of zinc chloride was refluxed for one hour and water was added by drops to the hot solution until crystallization commenced. The acetoxy compound separated in fluffy, dull yellow plates, m. p. 171–178°; yield 0.45 g. (83%). Boiling with acetic acid caused decomposition, but alcohol proved satisfactory for recrystallization. A sample crystallized four times from this solvent formed fine, straw-colored needles, m. p. 175–179°, with softening at 171°. Further recrystallization did not give a product of sharp m. p.

Anal. Calcd. for $C_{21}H_{14}O_2$: C, 84.54; H, 4.73. Found: C, 84.66; H, 4.89.

1',9-Methylene-1,2-benzanthracene.[2]—The acetate X (60 mg.) was reduced with zinc and alkali according to Martin[16] and the fluorescent toluene layer passed through a tower of alumina and Supercel. The material recovered from the filtrate on crystallization from alcohol afforded 15 mg. of 1',9-methylene-1,2-benzanthracene, m. p. 122.5–123°, and 10 mg., m. p. 120–122° (51.5%). The pure sample showed no depression when mixed with the hydrocarbon prepared by the Elbs reaction.[2] Like the previous sample, the hydrocarbon appears colorless in artificial light and is a fluorescent greenish-yellow in daylight.

10-Methyl-1',9-methylene-1,2-benzanthracene (XI).—Five grams of o-(7-acenaphthyl)-benzoic acid was cyclized by standing in 100 g. of anhydrous hydrogen fluoride for fifteen minutes. The dark red solution was poured onto ice and the yellow precipitate was collected, washed and dried in vacuum. Attempted crystallization of the moist product from aqueous acetone was unpromising, for although some crystals were obtained, considerable tarry material resulted. For the Grignard reaction it was found best to use the crude dried product. This was added in half-gram lots with good stirring to 5 equivalents of methylmagnesium chloride in 150 cc. of ether; after refluxing for forty-five minutes and decomposing with dilute acid, the washed ether layer was concentrated until most of the ether had been removed and taken up in about 30 cc. of hot benzene. On cooling, 2–2.5 g. (43–54%) of **1',9-methylene-1,2-benz-10-anthranol** crystallized in slender, buff-

(14) Analysis by the Arlington Laboratories. Both this substance and its hydrogenation product are difficultly combustible.

(15) Analysis by H. S. Wight.

(16) Martin, This Journal, **58**, 1438 (1936).

colored needles, m. p. 160–164° dec. (see below). The filtrate was taken to dryness and the residue heated briefly on the hot-plate to effect dehydration and put through an alumina–Supercel tower in benzene solution. The hydrocarbon collected from the highly fluorescent filtrate was clarified with Norite in alcohol and crystallized from about 15 cc. of this solvent, giving 50–90 mg. (1.1–1.9%) of material melting at 174–177.5°. Three further crystallizations gave a product of the constant m. p. 181–181.4°; the first crop formed fine, pale orange needles (26–47 mg.) and the mother liquors gave 10 mg. more of good material; yield of pure hydrocarbon, 0.8–1.2%.

Anal. Calcd. for $C_{20}H_{14}$: C, 94.45; H, 5.55. Found: C, 94.25; H, 5.77.

The **trinitrobenzene derivative** when prepared from pure hydrocarbon crystallized from alcohol in bright orange-red needles, m. p. 182.5–183.2°.

Anal. Calcd. for $C_{26}H_{17}O_6N_3$: N, 8.99. Found: N, 9.13.

This derivative is not suitable for use in purifying the hydrocarbon, for when the crude reaction product from the tower was treated with trinitrobenzene the complex was brown-black and melted at about 162° and was not improved on repeated crystallization. The dark red picrate is too unstable to be useful as a derivative.

Many variations were tried in the procedure of conducting the Grignard reaction. In boiling benzene or toluene, in cold benzene, or in ether at −70°, the yield was either as reported or lower. Lithium methyl in ether gave the same yield as the Grignard reagent, and the addition of the crude cyclization product in benzene rather than as a solid lowered the yield. The low yield even under the most favorable conditions seems to be due to extensive and rapid isomerization to the anthranol.

1′,9-Methylene-1,2-benz-10-anthranol (XII).—The material separated from the Grignard reaction mixture by crystallization from benzene proved to be the anthranol in an essentially pure condition. The substance is quite sensitive and rapidly turns red in boiling benzene or in cold

dioxane. It is moderately soluble in ether or ligroin (b. p. 80–90°) and on three crystallizations from this solvent-pair formed pale yellow, fibrous needles, m. p. 160–164° with charring. The sample retained ligroin tenaciously and gave a satisfactory analysis only after drying for fifteen hours in high vacuum at 70°.

Anal. Calcd. for $C_{19}H_{12}O$: C, 89.03; H, 4.72. Found: C, 89.08; H, 4.85.

The substance is highly fluorescent in dilute alcoholic solution, and the addition of ferric chloride produces an orange-red coloration. Extensive decomposition occurred on attempted acetylation. The Bucherer reaction was tried at 140, 155, and 180° but gave only water-soluble products. On reduction of the anthranol with zinc and alkali, 1′,9-methylene-1,2-benzanthracene was obtained in 35% yield, and the result was the same using the crude cyclization product. That the yield was lower than from the acetate X is probably because of the great sensitivity of the anthranol and anthrone.

Summary

A synthesis has been developed for 1′,9-methylene-1,2-benzanthracene and its hitherto undescribed 10-methyl and 10-hydroxy derivatives starting with the reaction between o-chlorophenylmagnesium bromide and 7-acenaphthenone. A new method was found whereby the ketonic starting material can be produced readily in quantity. This consists in the oxidation of acenaphthene with red lead in acetic acid solution, saponification of the resulting 7-acetoxy compound and oxidation of 7-acenaphthenol to 7-acenaphthenone.

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Erythrina Alkaloids. VII. Isolation and Characterization of the New Alkaloids, Erythraline and Erythratine

BY KARL FOLKERS AND FRANK KONIUSZY

The isolation of erythramine from the seeds of *Erythrina sandwicensis* Deg. and *Erythrina subumbrans* (Hassk.) Merr. was described recently.[1] It was stated in that paper that erythramine had been isolated from other species of *Erythrina*, but that it was found associated with another alkaloid of such similar properties that its isolation was more difficult. This other new alkaloid has been named erythraline.

By fractional crystallization of the hydriodides

(1) Folkers and Koniuszy, THIS JOURNAL, **61**, 1232 (1939).

from the crude free alkaloids of *Erythrina glauca* Willd., with particular observance of the optical rotations, pure erythraline hydriodide was isolated as the major alkaloid and pure erythramine hydriodide as a minor alkaloid. A second minor alkaloid was also isolated as the hydriodide, and since it has been also characterized as new, it has been named erythratine. It was discovered later that pure erythratine base could be easily obtained directly by crystallization of the crude bases from ethanol, and the yield was twice that