The picrate crystallized from ethanol in orange-red, flat rods of m. p. 127–128°.

Anal. Calcd. for $C_{28}H_{19}O_8N_3$: C, 64.0; H, 3.6. Found: C, 64.4; H, 3.9.

2,3-Diphenyl-1,4-naphthoquinone (III).—The naphthol II (2 g.) was dissolved in acetic acid (50 cc.) and, after addition of potassium dichromate (2 g.) in acetic acid (30 cc.), heated to boiling for two minutes. Upon pouring onto ice, a yellow precipitate (1.7 g.) was obtained. It crystallized from ethanol in yellow prisms of m. p. 135–136°. An additional recrystallization from acetic acid raised the m. p. to 140–141°, as reported in the literature.[6] The yield of pure product was 1.2 g. (60%).

Anal. Calcd. for $C_{22}H_{14}O_2$: C, 85.1; H, 4.5. Found: C, 85.2; H, 4.7.

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RECEIVED APRIL 17, 1946

The Resistance to Hydrogenation of β-Stenols in Ethyl Acetate with Adams Platinum Oxide Catalyst

BY SEYMOUR BERNSTEIN AND LOUIS DORFMAN

It is known that dehydroergostenol on hydrogenation with either platinum oxide in glacial acetic acid[1] or with palladium in ethyl acetate[2] affords α-ergostenol and not δ-ergostenol since the unhydrogenated double bond migrates to the α-position. It was therefore surprising to find that dehydroergostenol was not hydrogenated with platinum oxide catalyst in ethyl acetate and the starting material was recovered unchanged. Likewise β-ergostenol could not be hydrogenated under these conditions.

This hitherto unsuspected fact that β-stenols are resistant to hydrogenation with platinum oxide in ethyl acetate should prove useful in future synthetic and structural work in the steroid field, *e. g.*, in the cardiac aglucons.

LEDERLE LABORATORIES, INC.
PEARL RIVER, NEW YORK
RECEIVED[3] APRIL 22, 1946

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[1] Morrison and Simpson, *J. Chem. Soc.*, 1710 (1932).

[2] Windaus and Lüttringhaus, *Ann.*, **481**, 119 (1930).

[3] Original manuscript received September 19, 1945.

Some Substituted Acetophenones[1]

BY E. CAMPAIGNE AND WM. BRADLEY REID, JR.

Ortho- and *meta*-methyl- and *ortho-* and *meta*-phenylacetophenones, required in another investigation, were prepared from acetic anhydride by the low temperature Grignard procedure of Newman and Booth.[2] The required Grignard reagent and yield of the corresponding methyl ketones were as follows: *o*-tolylmagnesium bromide, 48.2%; *m*-tolylmagnesium bromide, 46.4%; *o*-xenylmagnesium iodide, 61.8%; *m*-xenylmagnesium iodide, 26.8%.

The biphenyl compounds, being new, were characterized by means of their crystalline semicarbazones and 2,4-dinitrophenylhydrazones.

Experimental

***o*-Phenylacetophenone.**—2-Iodobiphenyl was produced in 82.7% yield by the toluene extraction of a mixture obtained by treating a diazotized solution of 2-aminobiphenyl (Monsanto Chemical Company) with excess potassium iodide solution. The 2-iodobiphenyl was converted to the Grignard reagent and treated with acetic anhydride, yielding *o*-phenylacetophenone as a yellow oil, b. p. 104–105° at 1 mm. This oil yielded a semicarbazone in white plates, melting at 197°.

Anal.[3] Calcd. for $C_{15}H_{15}N_3O$: N, 16.59. Found: N, 16.67.

A 2,4-dinitrophenylhydrazone was also prepared, and obtained in light orange plates, melting at 169–170°.

Anal. Calcd. for $C_{20}H_{16}N_4O_4$: N, 14.88. Found: N, 14.83.

3-Iodobiphenyl.—Using the method of Elks, Haworth and Hey,[4] *m*-nitroaniline was converted to 3-nitrobiphenyl in 43% yield. This nitro-compound, which melted at 59–61°, was reduced to the amine by hydrogenation over Adams platinum oxide catalyst in portions in 98.5% yield. The 3-aminobiphenyl, after distillation at 177–178° at 18 mm. pressure, solidified to a white solid melting at 31–31.5°. A solution of 53 g. (0.314 mole) of 3-aminobiphenyl in 500 ml. of 1.3 *M* sulfuric acid was diazotized with a solution of 22.5 g. (0.326 mole) of sodium nitrite in 50 ml. of water. The solid yellow diazo salt that formed was stirred vigorously in 500 ml. of toluene while a solution of 100.5 g. (0.628 mole) of potassium iodide in 250 ml. of water was added over a period of thirty minutes. The temperature of the reaction was maintained at +5° during the addition. The resulting red complex slowly decomposed at room temperature, and the black toluene layer that separated after several hours was dried and distilled at reduced pressure. The fraction which boiled at 145–155° at less than 1 mm. was redistilled at this pressure, and 3-iodobiphenyl was collected as a yellow oil, boiling at 149–152°. The yield was 42 g. or 48% of theoretical.

Anal. Calcd. for $C_{12}H_9I$: I, 45.42. Found: I, 45.69.

***m*-Phenylacetophenone.**—The Grignard reagent was prepared from 3-iodobiphenyl and converted to *m*-phenylacetophenone by treatment with acetic anhydride. The ketone was obtained as a light yellow oil boiling at 148–151° at less than 1 mm. pressure. It readily formed a semicarbazone which was obtained as white plates, melting at 222–223°.

Anal. Calcd. for $C_{15}H_{15}N_3O$: N, 16.59. Found: N, 16.47.

The 2,4-dinitrophenylhydrazone was also obtained as orange needles, m. p. 191–192°.

Anal. Calcd. for $C_{20}H_{16}N_4O_4$: N, 14.88. Found: N, 14.68.

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RECEIVED APRIL 26, 1946

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[1] Abstracted from a part of the thesis submitted by Wm. Bradley Reid, Jr., to the faculty of the Graduate School in partial fulfillment of the requirements for the Degree, Doctor of Philosophy, in the Department of Chemistry, Indiana University.

[2] Newman and Booth, THIS JOURNAL, **67**, 154 (1945).

[3] All analyses are by Mrs. W. B. Reid, Jr., of this Laboratory.

[4] Elks, Haworth and Hey, *J. Chem. Soc.*, 1285 (1940).

A Simple Purification Procedure for DDT[1]

BY KATHRYN H. COOK AND WALTER A. COOK

A survey of the literature on the new insecticide popularly known as DDT, discloses the fact

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[1] Presented before the Division of Organic Chemistry at the Atlantic City Meeting of the American Chemical Society, April 11, 1946.