

NOTES

1,2,3,4-Tetrahydro-2-methoxynaphthalene

BY HARRY A. ARBIT

The hydrogenation of 2-methoxynaphthalene to "ac-tetrahydro- β -naphthyl methyl ether" over Raney nickel in 71 to 73% has been described by Adkins and co-workers.¹ However, no physical properties of this compound other than a boiling point of 115–118° at 10 mm. pressure are given and no reason is presented for the statement that hydrogenation had taken place in the oxygenated ring.

We have carried out the hydrogenation² of 2-methoxynaphthalene as described by Musser and Adkins.¹ The product (58% yield) was a water-white liquid with a sweet odor, b. p. 113–115° (9 mm.), n_D^{25} 1.5293. In order to obtain clear evidence that the methoxyl group was on the reduced rather than on the aromatic ring, the following experiment was carried out. Two grams of the liquid was refluxed for ten minutes with 10 ml. of hydriodic acid (sp. gr. 1.7). Methyl iodide was evolved and the product obtained (b. p. 120° (10 mm.), n_D^{25} 1.5635) was insoluble in 10% sodium hydroxide and gave a positive qualitative test for iodine after sodium fusion. It was therefore 1,2,3,4-tetrahydro-2-iodonaphthalene, formed by action of the hydriodic acid on the secondary alcohol, ac-tetrahydro- β -naphthol, after demethylation of the original compound. This same iodide was obtained when a known sample of ac-tetrahydro- β -naphthol^{3,4} was similarly treated with hydriodic acid.

The compound formed by the hydrogenation of 2-methoxynaphthalene is thus shown to be 1,2,3,4-tetrahydro-2-methoxynaphthalene. The latter has been obtained by v. Braun and Weissbach⁵ by the hydrogenation of 3,4-dihydro-2-methoxynaphthalene. These authors give the boiling point of the compound as 123–124° (16 mm.), and mention its pleasant odor.

(1) Van Duzee and Adkins, *THIS JOURNAL*, **57**, 147 (1935); Musser and Adkins, *ibid.*, **60**, 664 (1938).

(2) The hydrogenation was carried out by Mr. W. M. Selby.

(3) Bamberger, *Ber.*, **23**, 197 (1890).

(4) Brochet and Cornubert, *Bull. soc. chim.*, [4] **31**, 1280 (1922).

(5) v. Braun and Weissbach, *Ber.*, **63**, 3052 (1930).

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A Simple Route to 2,3-Diphenyl-1,4-naphthoquinone

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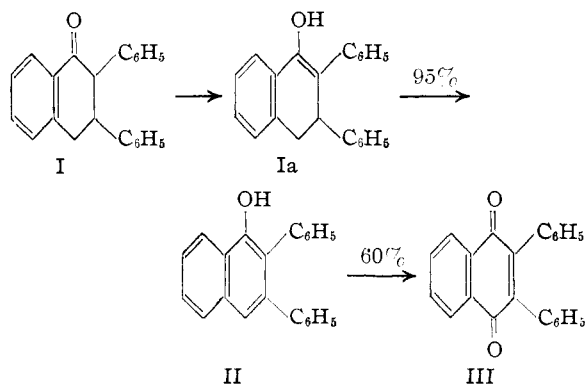
Crawford and Nelson¹ recently described an improved method for the conversion of 2,3-diphenyl-1-keto-1,2,3,4-tetrahydronaphthalene (I) into 2,3-diphenyl-1,4-naphthoquinone (III) in an over-all yield of 30%, using a four-step process. During our studies on polyphenylnaphthalenes² we observed that the ketone I shows a pronounced tendency to enolize to Ia. It seemed, therefore, possible that its direct dehydrogenation would lead to 2,3-diphenyl-1-naphthol (II). This is in-

(1) Crawford and Nelson, *THIS JOURNAL*, **68**, 134 (1946).

(2) P. Bergmann, Schapiro and Eschinazi, *ibid.*, **64**, 559 (1942).

deed the case. Heating of I with one equivalent of sulfur at 250–300° gives a nearly quantitative yield of the naphthol (II), which can be oxidized to the quinone (III) in about 60% yield.

Direct dehydrogenation of cyclic ketones to phenols has first been described by Darzens and Lévy.³ The method was applied in a number of cases, using either sulfur or selenium,⁴ but the yields are usually low. It is thus apparent that the enhanced enolizability of our ketone (I) has a favorable effect on the dehydrogenation to the corresponding naphthol (II), and one may presume that in every case the process consists essentially in the aromatization of an intermediate dihydronaphthalene derivative (such as Ia).



In this connection, it is of interest that catalytic dehydrogenation of cyclic ketones by means of palladium has been reported to give satisfactory yields (60–75%) of the corresponding phenols, especially if carried out in a solvent by prolonged heating.⁵

Experimental

2,3-Diphenyl-1-naphthol (II).—2,3-Diphenyl-1-keto-1,2,3,4-tetrahydronaphthalene (4.5 g.) and sulfur (530 mg.) were heated in a Pyrex tube. Reaction started at 250°, and this temperature was maintained for ten minutes and then raised slowly to 300° during thirty minutes. The black mass was distilled in a tube at 175–240° (0.6 mm.). The brown oil so obtained solidifies immediately; upon trituration with acetic acid, the substances showed a m. p. of 123–124°; yield, 4.3 g. The substance crystallized from acetic acid or petroleum ether (130°) in colorless plates, of m. p. 127–128°.⁶

Anal. Calcd. for $C_{22}H_{16}O$: C, 89.1; H, 5.4. Found: C, 88.9; H, 5.4.

(3) Darzens and Lévy, *Compt. rend.*, **194**, 181 (1932).

(4) (a) Cook and Hewett, *J. Chem. Soc.*, 403 (1933); (b) Fieser, Hershberg and Newman, *THIS JOURNAL*, **57**, 1509 (1935); (c) Peak and Robinson, *J. Chem. Soc.*, 759 (1936); (d) Ruzicka, *Helv. Chim. Acta*, **19**, 419 (1936).

(5) (a) Mosettig and Duvall, *THIS JOURNAL*, **59**, 367 (1937); (b) Wilds and Shunk, *ibid.*, **65**, 469 (1943); Cook and Schoental, *J. Chem. Soc.*, 288 (1945).

(6) Weiss and Sonnenschein, *Ber.*, **58**, 1043 (1925), report a m. p. of 128–131°.