CATALYTIC METHOD OF SYNTHESIS OF 2,4-DIPHENYLFURAN AND 2-PHENYLINDOLE

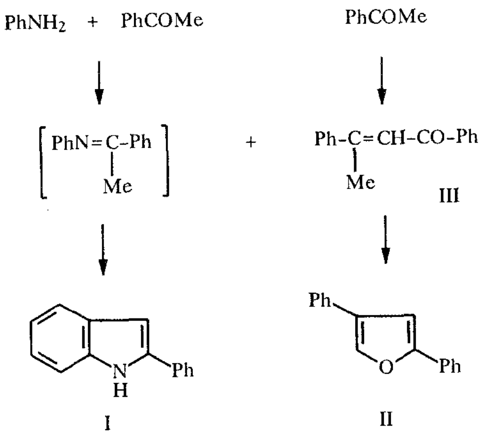
B. N. Anisimov, A. A. Obynochnyi, and N. S. Prostakov

2,4-Diphenylfuran has been obtained from acetophenone and its mixture with aniline, over K-16 catalyst. It has been established that the reaction proceeds through a stage of dypnone formation. When the aniline—acetophenone mixture is used as the starting material, small amounts of 2-phenylindole are formed.

We have used dehydrocyclization reactions to obtain nitrogen-containing heterocyclic compounds with various structures. In these syntheses we used K-12 and K-16 commercial dehydrogenation catalysts [1]. Here we are presenting results from experiments on the catalytic conversion of acetophenone, an aniline—acetophenone mixture, and dypnone over K-16 catalyst at 360-380°C.

We can assume that in the case of the aniline—acetophenone mixture, the Schiff base acetophenonanil is formed and is then converted upon dehydrocyclization to 2-phenylindole (I), which is recovered with a yield of only 2%. The main product of the reaction is 2,4-diphenylfuran (II), yield 16%. In the dehydrocyclization of the acetophenone itself, the yield of the diphenylfuran II is 31% of the acetophenone reacted. The conversion of acetophenone to the substituted furan II proceeds through a stage of acetophenone condensation to form dypnone, i.e., 1,3-diphenyl-2-buten-1-one (III), which then undergoes dehydrocyclization.

Catalytic dehydrocyclization of dypnone (III) gives 2,4-diphenylfuran (II) with a 68% yield [2].



EXPERIMENTAL

The course of the reaction, the individuality of the substances, and their separation were monitored by TLC on Silufol UV-254 plates in an ether—heptane system (1:1). The mass spectra were obtained in an MKh-1303 mass spectrometer. The PMR spectra were taken in a Bruker WP-80 instrument.

The elementary analyses of the compounds that were obtained are in agreement with the calculated values.

2-Phenylindole (I) and 2,4-Diphenylfuran (II). A. A solution of 5 g (0.054 mole) of aniline and 5 g (0.04 mole) of acetophenone in 20 ml of benzene was passed through a contact tube containing 30 ml of catalyst for a period of 2 h. The temperature in the catalyst zone was 360-380°C. Collected 1.5 liters of gas (21°C, 755 mm Hg) consisting of: 54.3% H_2, 43.7% C_nH_{2n+2}, 1.4% CO_2, and 0.6% C_nH_{2n}. From the catalyst, 3.2 g of aniline and 1.7 g of acetophenone were distilled. The residue (1.5 g) was chromatographed on aluminum oxide (H = 19 cm, d = 3.5 cm, eluent a mixture of heptane and ether, 2:1). Recovered 0.1 g of 2,4-diphenylfuran (II), R_f 0.57 (mixture of heptane with ether, 1:1), mp 104-106°C [2]. PMR spectrum ($CDCl_3$): 7.31 (1H, d, 3-H, J = 0.8 Hz), 7.77 (2H, m, o-Ph), 7.65 (2H, m, o-Ph), 7.20-7.42 (2H, m, p-Ph), 7.33-7.45 ppm (4H, m, p-Ph).

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Found: M + 220. $C_{16}H_{12}O$. Calculated: M 220. Last eluted from the chromatographic column was 0.87 g of 2-phenylindole (I), R_f 0.32, mp 189-190°C [3]. Yields relative to amount of acetophenone reacted: 2% of compound I, 16.4% of compound II.

B. At the same catalyst temperature, a solution of 25 g (0.2 mole) of acetophenone in 50 ml of benzene was passed through the contact tube over the course of 6 h. Collected 3.5 liters of gas (21°C, 760 mm Hg). From the liquid reaction product, recovered 0.7 g (3% yield) of the substituted furan II, mp 104-106°C.

C. At the same catalyst temperature, a solution of 4.5 g (0.02 mole) of dypnone (III) in 25 ml of benzene was passed through the contact tube over the course of 1.5 h. Collected 0.6 liter of gas (22°C, 757 mm). By chromatography, recovered 3.6 g (68%) of diphenylfuran II, R_f 0.57, mp 104-106°C.

LITERATURE CITED

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3. K. Weygand and H. Hilgetage, *Methods of Experiment in Organic Chemistry* [Russian translation], Khimiya, Moscow (1968), p. 886.