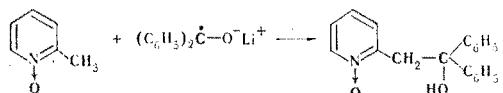


REACTION OF 2-PICOLINE N-OXIDE WITH BENZOPHENONE  
LITHIUM KETYL

A. S. Kurbatova, Yu. V. Kurbatov,  
and D. A. Niyazova

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We have found that 2-picoline N-oxide (I) does not react with benzophenone lithium ketyl in the ring, as in the case of pyridine N-oxide (I), but rather in the side chain with a high degree of selectivity to give only 1,1-diphenyl-2-(2'-pyridyl)ethanol N-oxide (II) in high yield:



The structure of II was confirmed by the PMR data and by deoxidation to give the corresponding pyridine derivative. The reaction may serve as a simple method for the synthesis of physiologically active diaryloxy-substituted oxides of azines.

A suspension of 0.35 g (0.05 mole) of finely cut up lithium in 10 ml of dioxane was added to a solution of 9.1 g (0.005 mole) of benzophenone (III) in 100 ml of absolute dioxane in a nitrogen atmosphere, and the mixture was stirred until the metal had dissolved completely. A solution of 5.45 g (0.05 mole) of oxide I in 50 ml of dioxane was added to the resulting dark-blue solution of the ketyl, and the mixture was stirred until a green coloration appeared. Water was added, and the precipitated 1,1-diphenyl-2-(2'-pyridyl)ethanol N-oxide was separated. The aqueous dioxane solution was extracted with hexane and benzene, and the hexane-dioxane extract was worked up to give 2.8 g (31%) of ketone III. Workup of the benzene extract gave an additional 0.5 g of alcohol II. The overall yield of alcohol II, with mp 214°C (from ethanol), was 67%. PMR spectrum (Varian XL-100, CF<sub>3</sub>COOH, hexamethyldisiloxane): 4.3 (s, 2H, CH<sub>2</sub>), 6.9-7.1 (m, phenyl protons), 7.5-7.6 (m, 2H, 3-H, 5-H, J<sub>5,6</sub> = 8.8 and J<sub>3,6</sub> = 7.4 Hz), 7.9 (t, 1H, 4-H, J<sub>3,4</sub> = J<sub>4,5</sub> = 7.0 Hz), and 8.4 ppm (d, 6-H, J<sub>5,6</sub> = 7.2 Hz). For deoxidation, a 0.5-g sample of alcohol II was refluxed in 7.5 ml of glacial acetic acid for 3 h with 0.1 g of iron powder, and the mixture was worked up in the usual manner to give 1,1-diphenyl-2-(2'-pyridyl)ethanol (70%) with mp 145°C (from ethanol). The results of elementary analysis for the nitrogen content were in agreement with the calculated value.

LITERATURE CITED

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A. Navoi Samarkand State University, Samarkand 703000. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 3, p. 419, March, 1982. Original article submitted July 15, 1981.