PHENALENODIAZEPINONE IN THE REACTION OF 2,3-DIAMINOPHENALENONE WITH ACETONE

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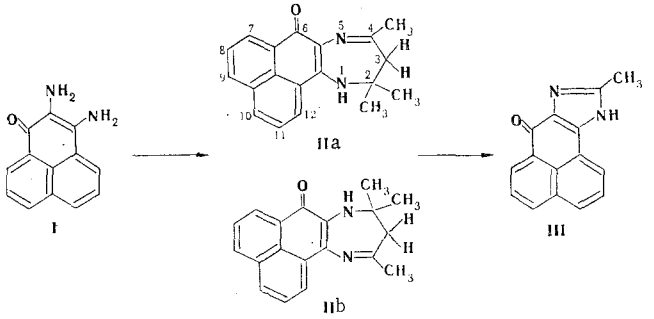
The literature contains no indications of the possibility of the cyclization of 2,3-diaminophenalenone (I) with acetone with the formation of a diazepine ring. We were able to realize this sort of transformation by refluxing diamine I in acetone solution or by reaction with mesityl oxide and obtained 1H-2,3-dihydro-2,2,4-trimethylphenaleno[2,3-b][1,5]diazepin-6-one (IIa). The structure of II was established on the basis of the PMR spectrum, which contains, in addition to signals from six aromatic protons (7.38-8.60 ppm), four singlet peaks with an intensity ratio of 6:3:2:1 at 1.44, 2.46, 2.48, and 5.08 ppm, which correspond to six protons of methyl groups in the 2 position, three protons of a methyl group in the 4 position, protons of a methylene group, and one proton of an imino group. A definitive choice between structures IIa and IIb cannot be made on the basis of the available data. It is evident that this sort of cyclization should include two steps, viz., the formation of an azomethine and addition to the double bond of mesityl oxide in any sequence [1]. Considering the ability of the α-amino group of enamino ketones to form Schiff bases [2], which is not characteristic for the enamino ketones themselves, preference should be given to structure IIa. This is also in agreement with the possibility of the addition of the amino group of enamino ketones to the double bond.



The yield of II when diamine I is heated in acetone is 43%, as compared with 62% in the reaction with mesityl oxide; the product has mp 166-167°C (from petroleum ether). IR spectrum (in KCl): 1638 (C=O) and 3300 cm^{-1} (NH). UV spectrum (in alcohol), λ_{max} (log ε): 245 (4.44), 269 (4.39), 351 (4.10), 372 (3.97) shoulder, and 503 nm (3.54). The results of elementary analysis and a mass-spectrometric determination of the molecular mass were in agreement with the calculated values. Thermal decomposition of II at 270-280°C gave 2-methylphenalenoimidazolone (III) in 39% yield.

LITERATURE CITED

1. W. Ried and P. Stahlhofen, Chem. Ber., 90, 815 (1957).
2. W. Ried and E. Torinus, Chem. Ber., 92, 2902 (1959).

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