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SYNTHESIS OF 1-(3-METHYLPYRAZOLYL-1)ANTHRAQUINONE

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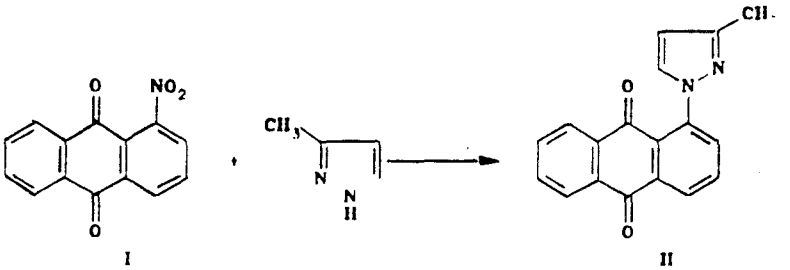
UDC 547.722.1:547.673.1:54.057

Nucleophilic replacement of the nitro group in 1-nitroanthraquinone (I) by amino takes place under quite drastic conditions.

We have found that 3(5)-methylpyrazole, a weak N-nucleophile (pK_a 3.32 [2]), reacts with 1-nitroanthraquinone under prolonged heating at 150°C to form one of two possible isomers, viz., 1-(3-methylpyrazolyl-1)anthraquinone (II). In the ^{13}C NMR spectrum (DMSO-D_6) of (II) the methyl signal at 13.25 ppm corresponds to the particular isomer, because the chemical shifts of the methyl carbons at positions 3 and 5 of the heterocycle are quite different from one another [3]. As is known, in going from $CDCl_3$ to DMSO-D_6 the PMR spectra of 1-substituted pyrazoles show a characteristic shift of the 5-H proton signal to the weak field [4]. Analogous changes in the spectrum of (II) also confirm its structure.



I II

1-(3-Methylpyrazolyl-1)anthraquinone (II). A mixture of 1.27 g of (I) and 5 ml of 3(5)-methylpyrazole was held at 150°C for 30 min. Methylpyrazole was distilled off in vacuum, and the residue was chromatographed on a column of 100/400 μm silica gel (benzene eluent). There was obtained 1.1 g (76%) of (II), mp 189-190°C, R_f 0.44. PMR spectrum ($CDCl_3$): 2.43 (3H, s, 3'-CH_3), 6.33 (1H, d, 4'-H), 7.59 (1H, d, 5'-H), 7.75-8.45 ppm (7H, m, $C_{14}H_7$). PMR spectrum (DMSO-D_6): 2.28 (3H, s, 3'-CH_3), 6.28 (1H, d, 4'-H), 7.90 (1H, d, 5'-H), 7.85-8.33 ppm (7H, m, $C_{14}H_7$). M^+ 288.

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Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 12, p. 1695, December, 1990. Original article submitted March 12, 1990.