6-Methyl-3-nitroso-1-phenethyl-4-(phenethylamino)-2(1H)-pyridinone

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In the course of our recent studies on the reactivity of some 4-amino-2-pyridones [1-3] we accomplished the nitrosation of the N,N-disubstituted 4-amino-2-pyridone 2. The 3-nitroso product 2 would be useful for the preparation of 3,4-fused heterocyclic systems, e.g. in Traube-type syntheses [4]. The starting 6-methyl-1-phenethyl-4-(phenethylamino)-2(1H)-pyridinone (1) was prepared according to the known procedure [2,5]. To a solution of pyridone 1 (1.99 g, 6 mmol) in glacial acetic acid (5 ml) was added dropwise a solution of sodium nitrite (620 mg, 9 mmol) in water (8 ml) with stirring at 20-25 °C. The reaction mixture was stirred for 1 h, before ice-cold water (10 ml) was added under stirring. The crystals that separated out were filtered off, washed twice with water and dried (2 h at 105°C) to afford the title compound 2. Yield: 1.48 g (69 %). Dark green needles.


1H NMR (300 MHz, CDCl3): 2.01 (s, 3H, 6-CH3), 2.94 (t, J = 7.2 Hz, 2H, 4-NHCH2CH2Ph), 3.08 (t, J = 7.2 Hz, 2H, 1-CH2CH2Ph), 3.47 (dt, J = 7.2 Hz, 2H, 4-NHCH2), 4.22 (t, J = 7.2 Hz, 2H, 1-CH2), 5.41 (s, 1H, H-5), 7.21-7.34 (m, 10H arom., two C6H5), 13.18 (broad s, 1H, NH).

FT IR (cm⁻¹, nujol): 1665, 1613, 1559, 1528, 1459, 1422, 1378, 1358, 1273, 1246, 1196, 1175, 1125, 1092, 1061, 1032, 980, 806, 752, 704, 688.

EI MS (70 eV; m/z (%)): 361 (M⁺, 14), 347 (14), 343 (17), 318 (10), 270 (11), 254 (33), 239 (82), 238 (43), 166 (22), 153 (10), 152 (31), 139 (46), 105 (85), 104 (29), 91 (100), 77 (30), 65 (31).


References

Sample availability: available from the authors and from MDPI.