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UNEXPECTED REACTION OF 4-SUBSTITUTED 6-CHLORO-5-NITROPYRIMIDINES WITH N-BENZYLPHENACYLAMINE

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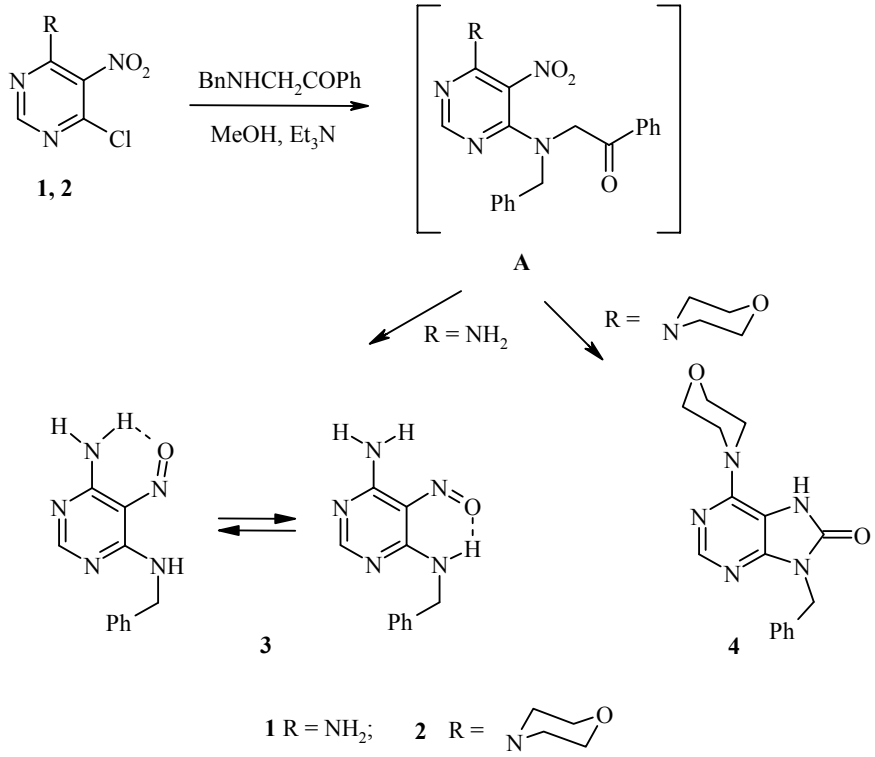
In continuing studies on 5-nitropyrimidines and their rearrangements when reacted with bases [1, 2], we observed that 6-chloro-5-nitropyrimidines **1, 2** when reacted with N-benzylphenacylamine in boiling methyl alcohol in the presence of triethylamine are converted to 4-amino-6-benzylamino-5-nitrosopyrimidine (**3**) and 9-benzyl-6-morpholinopurin-8(7H)-one (**4**) respectively. Products **3** and **4** are probably formed as a result of rearrangement of intermediate compound **A**.



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We should note that 5-nitrosopyrimidine **3** exists in solution as two conformers, evidence for which comes from its ^{1}H NMR spectrum, where we observe two sets (2:3) of characteristic signals.

The IR spectra were obtained on a Spectrum BX II FT-IR spectrophotometer (Perkin-Elmer) in vaseline oil. The ^{1}H and ^{13}C NMR spectra were recorded on a Varian Unity Inova (300 MHz and 75 MHz respectively) in DMSO-d_6, internal standard TMS; the mass spectra were recorded on a Micromass Quattro Ultima PT.

The starting 6-chloro-5-nitropyrimidines **1**, **2** were synthesized according to the methods in [3, 4].

Reaction of 4-Substituted 6-Chloro-5-nitropyrimidines (1, 2) with N-Benzylphenacylamine. Triethylamine (0.4 g, 4 mmol) was added to a solution of compound **1** (or **2**) (2 mmol) and N-benzylphenacylamine hydrochloride (0.52 g, 2 mmol) in anhydrous methanol (10 ml). The reaction mixture was boiled for 15 min. After cooling down to room temperature, the precipitate was filtered out and recrystallized, obtaining compounds **3** or **4**.

4-Amino-6-benzylamino-5-nitrosopyrimidine (3). Yield 82%; mp 189-190°C (DMF). IR spectrum, ν, cm^{-1}: 3350, 3352, 3277 (NH_2, NH). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.68, 4.76 (2H, d, *J* = 6, NCH_2); 7.29-7.39 (5H, m, ArH); 7.97, 8.04 (1H, s, CH); 9.74, 11.57 (1H, t, *J* = 6, NH); 8.22, 8.62, 9.27, 10.24 (2H, br. s, NH_2). Mass spectrum, *m*/*z* (*I*, %): 229 $[M]^+$ (100). Found, %: C 57.79; H 4.65; N 30.71. $C_{11}H_{11}N_5O$. Calculated, %: C 57.63; H 4.84; N 30.55.

9-Benzyl-6-morpholinopurin-8-one (4). Yield 90%; mp 259-260°C (MeOH). IR spectrum, ν, cm^{-1}: 3116 (NH), 1715 (CO). ^{1}H NMR spectrum, δ, ppm: 3.52-3.56 (4H, m, $N(CH_2)_2$); 3.68-3.71 (4H, m, $O(CH_2)_2$); 4.98 (2H, s, CH_2); 7.32 (5H, s, ArH); 8.19 (1H, s, CH); 11.18 (1H, br. s, NH). ^{13}C NMR spectrum, δ, ppm: 42.4, 45.8, 65.9, 105.1, 127.3, 127.4, 128.5, 136.9, 146.9, 149.0, 150.1, 152.8. Mass spectrum, *m*/*z* (*I*, %): 311 $[M]^+$ (45). Found, %: C 61.65; H 5.28; N 22.09. $C_{16}H_{17}N_5O_2$. Calculated, %: C 61.72; H 5.50; N 22.49.

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