ISSN 1070-3632, Russian Journal of General Chemistry, 2008, Vol. 78, No. 5, p.1018. © Pleiades Publishing, Ltd., 2008.
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LETTERS TO THE EDITOR

Thiocyanation of 6-Amino- and 6-Hydroxyaminouracyls

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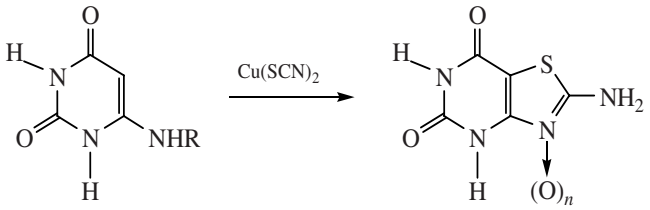
Received November 16, 2007

DOI: 10.1134/S1070363208050344

The thiazole *N*-oxides are a poorly studied class of compounds. 2-Aminothiazole 3-oxides can be obtained by reductive cyclization of *o*-thiocyanatonitrobenzene [1], and also by reaction of α-chloroximes with barium thiocyanate [2]. At the same time thiocyanation of aromatic amines is widely used in the synthesis of fused 2-aminothiazoles. Thiocyanation of heterocyclic amines is usually carried out with thiocyanogen formed in situ by treating a mixture of amine and thiocyanate with a solution of bromine in acetic acid [3, 4].

Available copper(II) thiocyanate proved to be an effective and easy for use reagent for thiocyanation of 6-aminouracyl **Ia** and 6-hydroxyaminouracyl **Ib**. While performing the reaction in water the corresponding 2-aminothiazolo [4,5-*d*]pyrimidin-5,7-dion (**IIa**) and its 3-oxide **IIb** were prepared.



Ia, Ib **IIa, IIb**

R = H (**Ia**), R = OH (**Ib**), *n* = 0 (**IIa**), *n* = 1 (**IIb**).

2-Aminothiazolo[4,5-*d*]-4,5,6,7-tetrahydropyrimidine-5,7-dione (IIa). A mixture of 0.65 g of 6-aminouracyl **Ia**, 1.8 g of copper(II) thiocyanate and 60 ml of water was heated with stirring at 40°C to complete conversion of black cupric salt to white cuprous one. After that the reaction mixture was heated to boiling and filtered. The filtrate was cooled, and the precipitate formed was filtered off, washed with a little ethanol, and dried in air. Yield 0.34 g (36%), mp > 310°C (with decomposition). Mass spectrum, *m/z* (I_{rel}, %): 184 (70), 141 (23), 99 (5), 64 (22), 56 (21), 43 (100). IR spectrum (mineral oil), ν, cm^{-1}: 3470, 3350–3000, 1750, 1740, 1720, 1710, 1700, 1650–1500. Found, %: C 32.60, H 2.20, N 30.40. $C_5H_4N_4O_2S$. Calculated, %: C 32.61, H 2.17, N 30.44.

2-Aminothiazolo[4,5-*d*]-4,5,6,7-tetrahydropyrimidine-5,7-dione N^3-oxide(IIb) was prepared from 6-hydroxyaminouracyl **Ib** [5] by analogous procedure. Yield 50%, mp > 260°C (with decomposition). Mass spectrum (FAB): $[M + H]^+$ 201. Mass spectrum, *m/z* (I_{rel}, %): 184 (55), 141 (12), 99 (5), 71 (14), 64 (24), 56 (21), 44 (69), 43 (100). IR spectrum (mineral oil), ν, cm^{-1}: 3500–3460, 3400–3000, 1720, 1710, 1690, 1680, 1660, 1640, 1610, 1590, 1560–1500. Found, %: C 30.20, H 1.90, N 28.10, $C_5H_4N_4O_3S$. Calculated, %: C 30.00, H 2.00, N 28.00.

The purity of the compounds obtained was checked by TLC on Silufol UV-254 plates, elution with 5 : 2 chloroform–ethanol.

Mass spectra were obtained on a MKh-1321 mass spectrometer with the direct admission of samples into the ion source, ionizing electrons energy 70 eV, and the temperature of the ion source 220°C. The fast atom bombardment mass spectra were registered on a VG Analytical 7070 EQ device. The IR spectra were recorded on a Specord M-80 spectrometer.

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