Russian Journal of Organic Chemistry, Vol. 39, No. 8, 2003, p. 1199. Translated from Zhurnal Organicheskoi Khimii, Vol. 39, No. 8, 2003, p. 1270.

SHORT COMMUNICATIONS

Dedicated to Professor G.I.Koldobskii on occasion of his 70th anniversary

2-Halo-2,4-dinitrothiolene-1,1-dioxides in Reaction with Pyridine

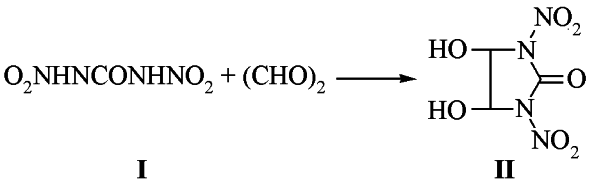
S. G. Ilyasov[1], A. A. Lobanova[2], and E.V.Rudakov[2]

[1]*Federal State Unitary Enterprise Federal Scientific and Production Center Altai, Biisk, 659322 Russia*
[2]*Institute of Problems in Chemicoenergetical Technologies, Siberian Division, Russian Academy of Sciences*

Received June 25, 2002

We have formerly reported on the synthesis of symmetrical dinitrourea [1] and its transformation into nitramide [2].

The condensation of dinitrourea with glyoxal is not described in the literature. We showed that at treating *N*,*N*'-dinitrourea with water solution of glyoxal 4,5-dihydroxy-1,3-dinitro-1,3-diazacyclopentan-2-one was formed.



I **II**

4,5-Dihydroxy-1,3-dinitro-1,3-diazacyclopentan-2-one. To 5 ml of 40% water solution of glyoxal (0.044 mol) was added at 20°C while stirring 5.6 g of dinitrourea containing 80% of the main compound (0.03 mol). After stirring for 5 min at 40°C to the reaction mixture 5 ml of water was added, and the mixture was cooled to 20°C. The precipitate was filtered off, washed with 5 ml of ice water, and dried at room temperature. Yield 2.6 g (41.7%), mp (decomp.) 167°C, mp (decomp.) 175°C (after reprecipitation from acetone solution into dichloromethane). IR spectrum, cm^{-1}: 3400 (OH), 1800 (C=O), 1605, 1590, 1315, 1290, 1255 (NO_2). ^1H NMR spectrum, δ, ppm: 5.83 s (2H, CH–CH), 7.03 s (2H, OH). ^{13}C NMR spectrum, δ, ppm: 83.06 (CH), 140.7 (C=O). Found, %: C 17.8; H 2.3; N 24.6. $C_3H_4N_4O_7$. Calculated, %: C 17.3; H 1.92 N 26.9.

IR spectra were recorded on spectrophotometer Specord M-80 from KBr pellets, NMR spectra were registered in acetone-d_6 on spectrometer Bruker AM-200 at operating frequencies 200 (^1H) and 50 MHz (^{13}C), internal reference TMS.

REFERENCES

1. Lobanova, A.A., Sataev, R.R., Popov, N.I., and Il'yasov, S.G., *Zh. Org. Khim.*, 2000, vol. 36, p. 188.
2. Lobanova, A.A., Il'yasov, S.G., Popov, N.I., and Sataev, R.R., *Zh. Org. Khim.*, 2002, vol. 38, p. 11.

1070-4280/03/3908-1199$25.00 © 2003 MAIK "Nauka/Interperiodica"