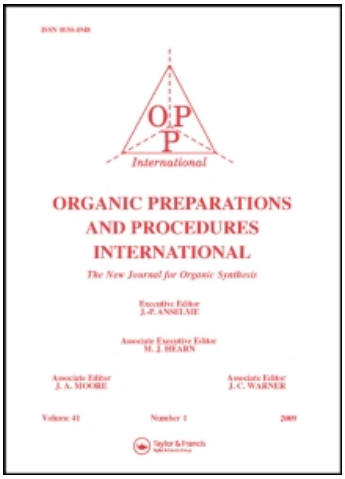

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4-AMINO-3-HYDRAZINO-5-MERCAPTO-1,2,4-TRIAZOLE

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4-AMINO-3-HYDRAZINO-5-MERCAPTO-1,2,4-TRIAZOLE

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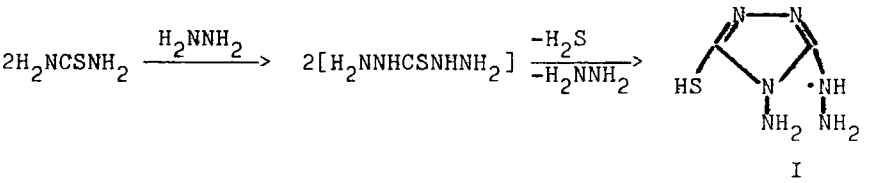
4-Amino-3-hydrazino-5-mercapto-1,2,4-triazole (I) can be prepared from a great variety of acyclic and cyclic compounds containing the thioureido grouping by reaction with hydrazine.[1] The title compound is of special interest because it has



I

recently been shown to be a specific, yet highly sensitive reagent for the detection of aliphatic and aromatic aldehydes.[2] The qualitative test depends upon the rapid formation of purple or magenta colored 6-mercapto-3-substituted-s-triazolo-(4,3-b)-s-tetrazine derivatives when one drop of an aldehyde is added to a freshly prepared solution of 0.1 g of the reagent in 2 ml of N-sodium hydroxide and the mixture is aerated using a micro-bubbler. The intense color of the triazolo-tetrazine dye develops rapidly within 1 minute.

EXPERIMENTAL

Thiourea (1.0 mole, 76 g) and 98% hydrazine hydrate (4.0 moles, 200 g), is a 1-liter flask equipped with a reflux condenser and mechanical stirrer, were heated at 100° (steam) for 3 hrs. (fume-hood). The reaction mixture was cooled, diluted with water (300 ml), neutralized (pH 6.5) by addition of conc. hydrochloric acid (∿ 20 ml) and refrigerated at 0° for 1/2 hr. The crude product was filtered, washed with ice-water (100 ml) and digested in dilute hydrochloric acid (2M, 750 ml) for 15 min. After removal of acid-insoluble impurities (mainly sulfur), the filtrate was neutralized to pH 6.5 with sodium hydroxide and refrigerated at 0° for 1/2 hr. to yield 4-amino-3-hydrazine-5-mercapto-1,2,4-triazole (42-48 g; 57-65%). Dried at 110°, the product in this form had mp. 226-228° (dec.); one recrystallization from water (700 parts; 87% recovery after 12 hr. at 0-5°) gave the triazole as colorless needles mp. 231-233° (dec.), lit.[3] mp. 228°. The infrared absorption spectrum exhibited major absorption bands at 3050-3320 (NH stretch); 1645,1595, 1500 (NH deformation freq.); 1155 (C=S).

Anal. Calcd. for $C_2H_6N_6S$: C, 16.45; H, 4.15; N, 57.50.
Found: C, 16.30; H, 4.00; N, 56.90

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