# SIMPLIFIED SYNTHESIS OF 2-ALKYLTHIO-6-R-5-OXO-5H-1,3,4-THIADIAZOLO[2,3-c]-1,2,4-TRIAZINES

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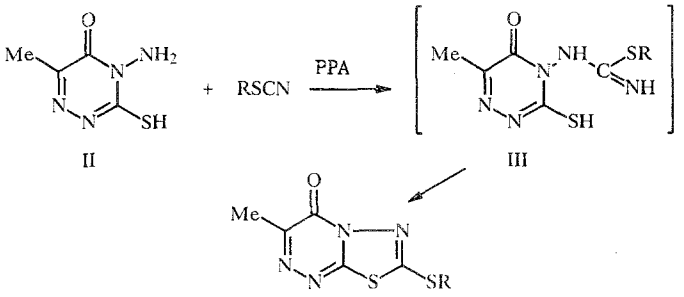
Two methods for obtaining the 2-alkylthio-6-R-5-oxo-5H-1,3,4-thiadiazolo[2,3-c]-1,2,4-triazine I system are known — condensation of 2-hydrazino-5-alkylthio-1,3,4-thiadiazoles with α-keto acids [1] and the reaction of 4-amino-3-X-6-$R^1$-5-oxo-4,5-dihydro-1,2,4-triazines II (X = NHPh, SMe) [2] with carbon disulfide in pyridine with subsequent alkylation of the mercapto group in an alkaline medium [3]. The possibilities of the first method are limited by the difficulties involved in obtaining the starting hydrazine, and the second method leads only to SH derivatives and requires an additional alkylation step.

On the basis of the data in [4], we proposed a one-step method for obtaining Ia, b that consists in the direct reaction of triazine II ($R^1$ = Me, X = SH) with thiocyanic acid esters in polyphosphoric acid (PPA).




I a, b

I aR = Me, b R = $CH_2Ph$

The amino group of II evidently first adds to the nitrile fragment of the thiocyanic acid ester to give isothiourea III, which undergoes intramolecular cyclization to I.

Thus a mixture of 0.02 mole of II, 0.02 mole of methyl or benzyl thiocyanate, and 25 g of PPA was heated at 95-100°C for 5-6 h, after which it was cooled and diluted with 100 ml of water. The precipitate was removed by filtration, washed with water, air dried, and recrystallized from dioxane (in the case of Ia the reaction mixture was neutralized with 15% ammonium hydroxide to pH 4-6. This procedure gave Ia, with mp 195-196°C (mp 195-196°C [3]), in 70% yield and Ib, with mp 170°C (mp 171°C [3]), in 78% yield.

## REFERENCES

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