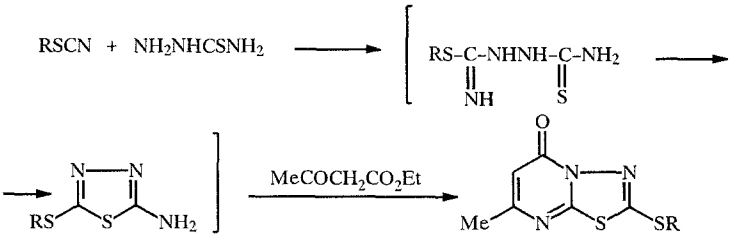
NEW SYNTHESIS OF 2-R-THIO-7-METHYL-5-OXO-5H-1,3,4-THIADIAZOLO[3,2-*a*]PYRIMIDINES

S. Sh. Shukurov and M. A. Kukaniev

The known methods for obtaining 2-R-thio-7-methyl-5-oxo-5H-1,3,4-thiadiazolo[3,2-*a*]pyrimidines I are based on the reactions of 2-amino-5-alkylthio(mercapto)-1,3,4-thiadiazoles with acetoacetic acid esters in the presence of cyclodehydrating agents such as concentrated H_2SO_4 or polyphosphoric acid (PPA) [1, 2]; a drawback common to these reactions is their multistep character.

We have found a single-reactor variation of the synthesis of I starting from alkyl and arylalkyl thiocyanates and thiosemicarbazide, without isolation of the intermediates, with subsequent reaction with acetoacetic ester in PPA at 90-100°C. The process takes no more than 3.5-4 h. It is possible that the reaction intermediates are alkylthio and arylalkylthio esters of hydrazodithiocarbamide, which are formed due to the addition of the hydrazine fragment of thiosemicarbazide to the cyano group of the alkyl and arylalkyl thiocyanates. They subsequently undergo cyclization to 2-amino-5-alkyl(arylalkyl)thio-1,3,4-thiadiazoles with subsequent conversion to Ia, b via the following scheme:



I a,b

Compound Ia (R = C_2H_5) was obtained in 77% yield and had mp 120-121°C [from dioxane—water (2:1)] (mp 120-121°C [2]).

Compound Ib (R = $C_6H_5CH_2$) was obtained in 86% yield and had mp 118-119°C (from dioxane). The samples of Ib obtained from 2-amino-5-benzylthio-1,3,4-thiadiazole [1] and by our proposed method were identical.

REFERENCES

1. Yu. Tokunaga, Ya. Tsuno, and J. Koshima; Japanese Patent Application No. 63-45286; *Ref. Zh. Khim.*, 130400P (1989).
2. K. Maekawa and S. Midzumitsu, Japanese Patent Application No. 52-118494; *Ref. Zh. Khim.*, 2102113P.

V. I. Nikitin Institute of Chemistry, Academy of Sciences of the Republic of Tadzhikistan, Dushanbe 734063.
Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 8, p. 1148, August, 1992. Original article submitted June 17, 1992.

0009-3122/92/2808-0970$12.50