SINGLE-REACTOR SYNTHESIS OF 2R-THIO-5,7-DIMETHYL-1,3,4-THIADIAZOLO[3,2-*a*]PYRIMIDINIUM PERCHLORATES

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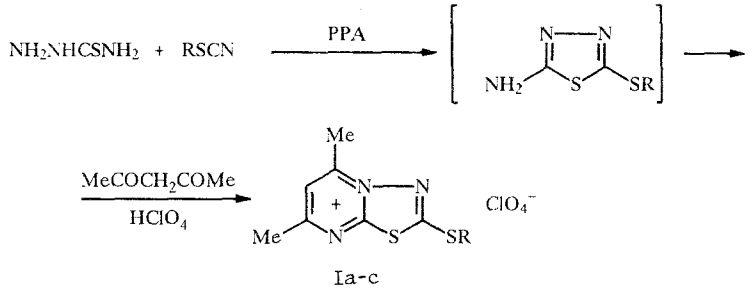
We found that the reaction of thiosemicarbazide with thiocyanic acid esters in PPA at 95-100°C (4-10 h) and subsequent condensation at 40-50°C of the intermediate reaction product II (without its isolation) with 2,4-pentanedione leads to the 2R-thio-5,7-dimethyl-1,3,4-thiadiazolo[3,2-*a*]pyrimidinium salts (I). Treatment of the aqueous solution of the reaction mixture with perchloric acid causes conversion into perchlorates of I.

The known methods of synthesis of I are based on the reaction of 2-amino-5R-thio-1,3,4-thiadiazoles (II) with pentane-2,4-dione or alkylation of 2-mercapto-I [1, 2].



Ia-c

Ia R = Me; Ib R = $PhCH_2$; Ic R = Ph

A countersynthesis of Ic was carried out by the reaction of 2-amino-5-phenylthio-1,3,4-thiadiazole and pentane-2,4-dione according to [1]. The properties of samples of Ic obtained by two independent methods were found to be identical.

2-Methylthio-5,7-dimethyl-1,3,4-thiadiazolo[3,2-*a*]pyrimidinium Perchlorate (Ia, $C_8H_{10}ClN_3O_4S_2$), yield 32%, mp 188°C (according to the data in [1], mp 188-189°C).

2-Benzylthio-5,7-dimethyl-1,3,4-thiadiazolo[3,2-*a*]pyrimidinium Perchlorate (Ib, $C_{14}H_{14}ClN_3O_4S_2$), yield 71%, mp 195°C (according to the data in [1], mp 193-195°C).

2-Phenylthio-5,7-dimethyl-1,3,4-thiadiazolo[3,2-*a*]pyrimidinium Perchlorate (Ic, $C_{13}H_{12}ClN_3O_4S_2$), yield 92%, mp 251-253°C.

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