CONDENSATION OF 5-AMINO-1,2,3,4-THIATRIAZOLE WITH ACETYLACETONE

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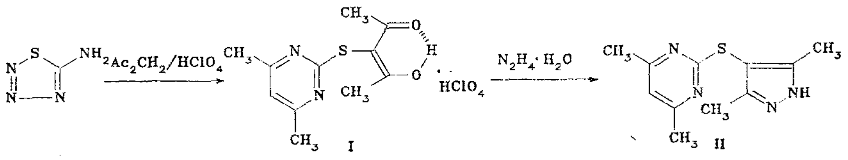
We have observed that nitrogen evolution (1 mole per mole of aminothiatriazole) occurs when a solution of 5-amino-1,2,3,4-thiatriazole in acid (hydrochloric, perchloric, or trifluoroacetic acid) is mixed with acetylacetone. 4-Hydroxy-3-[(4,6-dimethyl-2-pyrimidinyl)-thio]pent-3-en-2-one perchlorate (I), with mp 182-184°C (dec., from isopropyl alcohol), was obtained in 67% yield from the reaction mixture after nitrogen evolution when the reaction was carried out in an anhydrous mixture of perchloric and acetic acids. PMR spectrum (in CF_3COOH with respect to hexamethyldisiloxane): 2.05 (6H, s, CH_3), 2.35 (6H, s, 4'- and 6'-CH_3), and 7.00 ppm (1H, s, 5'-H).



Compound I reacts with hydrazine hydrate at room temperature in alcohol solution to give (3,5-dimethyl-4-pyrazolyl)-4,6-dimethyl-2-pyrimidinyl sulfide (II) with mp 139-140°C [from ethanol–water (1:4)] in quantitative yield. PMR spectrum: 2.20 and 2.33 (6H each, s, CH_3); 7.00 ppm (1H, s, pyrimidine proton).

Other β-diketones, β-chlorovinyl carbonyl compounds, β-keto aldehyde and malonaldehyde acetals, and acetoacetic ester also react with 5-amino-1,2,3,4-thiatriazole in the presence of acids at room temperature with nitrogen evolution to give various (depending on the nature of the reagents, the reaction conditions, and the way in which the reaction products are isolated) 2-mercaptopyrimidine derivatives. The results of elementary analysis of the compounds obtained were in agreement with the calculated values.

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