SYNTHESIS OF NEW 5(4)-HYDROXYIMIDAZOLE DERIVATIVES

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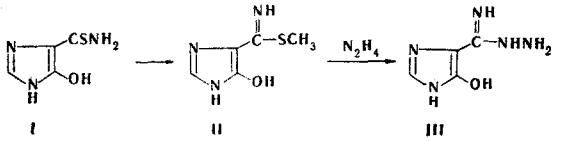
We have observed that in the methylation of 5(4)-hydroxyimidazole-4(5)-thiocarboxamide (I) with methyl iodide in methanol in the presence of sodium methoxide only S-methyl-5(4)-hydroxyimidazole-4(5)-thiocarboximidate (II) [mp 208°C (from water). UV spectrum (water), λ_{max} (log ε): 327 (4.27) and 215 nm (3.97). PMR spectrum (d_6-DMSO): 2.5 (s, 3H, SCH_3), 3.75 (NH), and 7.31 ppm (s, 1H, CH = N). IR spectrum (KBr): 685 cm^{-1} (SCH_3)] is formed, despite the presence in I of four possible sites for the incorporation of a methyl group.



I II III

Methyl mercaptan is evolved in the reaction of II with hydrazine hydrate, and 5(4)-hydroxyimidazole-4(5)-carboxamidrazone (III) is formed; this confirms the structure of II [IR spectrum of III: 3300, 3360, and 3400 cm^{-1} (NH). The PMR spectrum does not contain signals of a CH_3 group. UV spectrum (water), λ_{max} (log ε): 285 nm (4.2). The product was obtained in 80% yield and had mp 198°C (from water)].

Starting thioamide I was obtained by reaction of 5(4)-hydroxyimidazole-4(5)-carboxamide with P_4S_{10} in absolute dioxane [mp 202 (from water). UV spectrum (in 0.1 N HCl, λ_{max} (log ε): 209 (3.78), 256 (3.65), 273 (3.70), and 331 nm (4.32)].

The purity of I-III was monitored by thin-layer chromatography on Silufol in n-butanol–acetic acid–water (4:1:1), alcohol–chloroform (1:3), propanol–5% ammonium hydroxide (3:1), and alcohol–water (1:1) systems. The results of elementary analysis were in agreement with the calculated values.

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