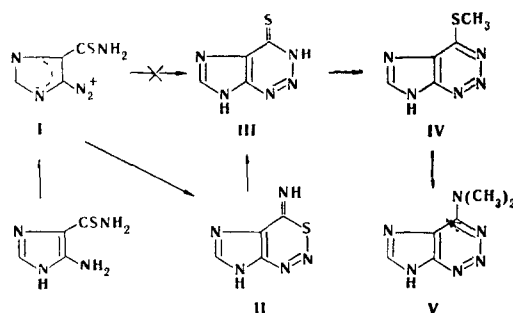


FEATURES OF THE INTRAMOLECULAR CYCLIZATION OF 4(5)-DIAZONIO-5(4)-THIOCARBAMOYLIMIDAZOLIDE

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UDC 547.785.5'872.3:543.422.4

In the diazotization of 4(5)-amino-5(4)-thiocarbamoylimidazole, a solution of 4(5)-diazonio-5(4)-thiocarbamoylimidazolid (I) was obtained which gave a coloration with the Bratton-Marshall reagent. At pH 5, a compound was isolated which consisted of the product of the intramolecular cyclization of (I), since its IR spectrum (in the solid state and in DMF) lacked a band in the $2200-2300\text{ cm}^{-1}$ region. By analogy with 4(5)-diazonio-5(4)-carbamoylimidazolid [1] it may be expected that (I) is converted into imidazo[4,5-d]-1,2,3-triazine-4-thione (III).



However, on reaction with NH_4OH the cyclization product gave a compound of the same composition but sharply differing in physical properties and the nature of its UV and IR spectra. On this basis, it may be concluded that (I) undergoes ring closure to form imidazo[4,5-d][1,2,3]thiadiazin-4-one imine (II) [mp 183°C (decomposes with explosion); λ_{max} , nm (log ϵ): 220 (4.18), 253 (4.11), 280 (3.63), 327 (3.59)] which in NH_4OH is converted into (III) [mp 190°C ; λ_{max} , nm (log ϵ): 237 (3.87), 315 (4.18)]. Methylation of the latter with methyl iodide in a methanolic solution of sodium methoxide gave 4-(methylthio)imidazo[4,5-d]-1,2,3-triazine (IV) [mp 230°C ; λ_{max} , nm (log ϵ): 234 (4.07), 290 (3.98); PMR spectrum (in DMSO-d_6): δ 2.9 ppm (3H), 8.9 ppm (1H)]. No formation of the corresponding N-methyl derivatives was observed. A confirmation of the structure of (IV) is the fact that the reaction of (IV) with dimethylamine led to the evolution of methyl mercaptan and to the formation of 4-dimethylaminoimidazo[4,5-d]1,2,3-triazine (V) [mp 225°C ; λ_{max} , nm (log ϵ): 249 (4.08), 316 (3.50)]. In addition, the UV spectra of (III), (IV), and (V) in 0.1 N HCl are similar and differ from that of (II). The results of elementary analysis for all the elements in (II-V) agreed well with the calculated figures.

LITERATURE CITED

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