THE REACTION OF 2-AMINOMETHYLBENZIMIDAZOLE WITH NITROUS ACID

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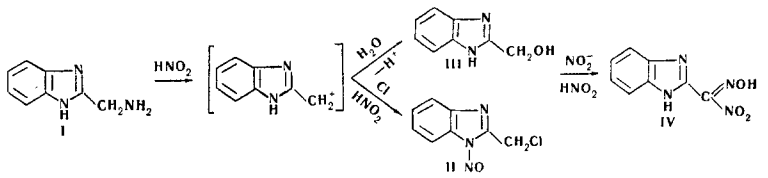
We have found that the reaction of 2-aminomethylbenzimidazole (I) with nitrous acid (1 mole) in HCl or HBr (3 moles) takes place with the liberation of nitrogen and the formation of 2-chloromethyl- and 2-bromomethylbenzimidazoles. The yields were 81 and 90%, respectively.

With an excess of nitrous acid (2 moles) the reaction takes place in a more complicated fashion. Three substances were isolated from the reaction mixture (see scheme):



2-Chloromethyl-1-nitrosobenzimidazole (II), yield 17%, decomp. p. 178° C (from methanol). Found, %: C 41.63; H 4.08; Cl 15.60. Calculated for $C_8H_6ClN_3O \cdot H_2O$, %: C 41.47; H 4.35; Cl 15.30. IR spectrum of II: ν_{N-NO} 1390 cm^{-1}. It is identical with the spectrum of the product of the nitrosation of 2-chloromethylbenzimidazole by a method similar to that of Ried and Urlass [1]. The presence of a N-nitroso group was shown by the Liebermann reaction and the Friess reagent.

2-Hydroxymethylbenzimidazole (III), yield 35%, mp 171–172° C (from water) [2].

Benzimidazole-2-carboxamide oxime (IV), yield 39%, decomp. p. 108° C. Found, %: C 42.22; H 3.07; N 26.94. Calculated for $C_8H_6N_4O_3$, %: C 46.60; H 2.93; N 27.17. The IR spectrum of IV and the compound obtained by the nitration of 2-formylbenzimidazole [3] were identical, containing: ν_{NO_2} 1330, 1560 cm^{-1}, $\nu_{C=N}$ 1620 cm^{-1}, and ν_{OH}—a broad band in the 3050 cm^{-1} region.

REFERENCES

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