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By saturating II with hydrogen bromide in an atmosphere of nitrogen first in the cold and then at 100° C we obtained 1-bromo-2-(β bromopropyl)cyclohexane (III), the structure of which was confirmed by its IR spectrum. Bp 99-100° C (2 mm), n_2^{20} 1.5280, d_D^{20} 1.5251. Found, %: C 38.19; H 5.70; Br 56.34; MR_D 57.09. Calculated for C $_{eH_{16}Br_2}$, %: C 38.03; H 5.63; Br 56.23; MR_D 57.03. Yield 86.3%.

The reaction of III with a threefold excess of anhydrous Na₂S in dimethylformamide at 125° C gave a 54.5% yield of I in the form of a mixture of cis- and trans-isomers. Bp 89°-90° C (20 mm), $n_{\rm D}^{\rm 20}$ 1.5090, $d_4^{\rm 20}$ 0.9783. Found, %: C 57.24; H 10.35; MR_D 47.61. Cal-

culated for C_gH_{16}S, %: C 57.22; H 10.25; MR_D 47.43. The structure of I was confirmed by its IR spectrum.

REFERENCE

1. R. L. Letsinger, J. G. Traynham, and E. Babko, J. Am. Chem. Soc., 74, 339, 1952.

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NITRATION OF 3-METHYL-3H-IMIDAZO[4, 5-b]PYRIDINE

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We have effected the direct introduction of a nitro group into the molecule of an imidazo[4,5-b]pyridine for the first time. In contrast to benzimidazole, the nitration of which takes place readily even in the cold [1], 3-methyl-3H-imidazo[4,5-b]pyridine (I) undergoes nitration with a mixture of nitric and sulfuric acids only at 140-160° C. The reaction product [yield 50%, mp 220-221° C (ethanol). Found, %: C 46.92; H 3.34; N 31.34. Calculated for $C_7H_6N_4O_2$, %: C 47.19; H 3.39; N 31.45] proved to be identical with the compound obtained from 3-amino-2-methylamino-5-nitropyridine (II) and formic acid, which shows its structure to be 3-methyl-6-nitroimidazo[4,5-b]-pyridine (II).



No other nitro derivatives apart from II were detected. Compound III was obtained by the reduction with ammonium sulfide of the product of the interaction of 2-chloro-3,5-dinitropyridine with methylamine [2]. Mp 199-200° C (water). Found, %: C 42.94; H 4.83. Calculated for $C_6H_8N_4O_2$, %: C 42.86; H 4.79.

REFERENCES

L. S. Efros, ZhOKh, 22, 1008, 1952.
A. Hunger, I. Kebrle, A. Rossi, and K. N. Hoffman, US
Patent 3004978, 1960. C. A. 56, 4771, 1962.

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QUATERNIZATION OF 3-METHYL-3H-IMIDAZO[4, 5-b]PYRIDINE

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3-Methyl-3H-imidazo[4,5-b]pyridine (I), which has two basic centers [1], reacts with alkylating agents at the N¹ atom with the formation of monoquaternary salts. The structure of such compounds as N¹alkyl(aryl, aralkyl)-N³-methylimidazolium salts (II) has been shown by the oxidation of some of them (R = CH₃, X = I; R = CH₂C₆H₅, X = = Cl) with potassium ferrocyanide in alkali at a temperature not exceeding 10° C to N¹, N³-disubstituted imidazo[4,5-b]pyridin-2-ones (III) (R = CH₃, CH₂C₆H₅). The same substances have been obtained by the methylation with dimethyl sulfate and the benzylation with dimethylphenylbenzylammonium hydroxide of 3-methylimidazo[4, 5-6]pyridin-2-ones (IV) in an alkaline medium. Compound IV was synthesized by fusing 3-amino-2-methylaminopyridine with urea at 170° C.

