

NITRATION OF IMIDAZO[4,5-c]-2-PYRIDONE

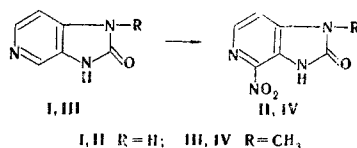
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Nitration of 1,3-dihydro-2H-imidazo[4,5-c]-2-pyridone (I) [1] with a mixture of sulfuric and nitric acids at 100–115° gives a mononitro derivative in almost quantitative yield. The PMR spectrum of this compound (obtained from a dimethyl sulfoxide solution with a Tesla BS-487B spectrometer with hexamethyl-disiloxane as the external standard) contains two doublets (δ 7.87 and 8.47 ppm, J_{HH} 5 Hz), the development of which is associated with the presence of two vicinal protons (in the 6 and 7 positions) of the pyridine ring (see [2]) in the nitro derivative; this is possible only in 4-nitro-1,3-dihydro-2H-imidazo[4,5-c]-2-pyridone (II).

1-Methyl-1,3-dihydro-2H-imidazo[4,5-c]-2-pyridone (IV), which is obtained by fusion of 3-amino-4-methylaminopyridine with urea at 170°C, is similarly nitrated to give 4-nitro-1-methyl-1,3-dihydro-2H-imidazo[4,5-c]-2-pyridone (III, δ 7.85 and 8.45 ppm, J_{HH} 5 Hz).

The direct introduction of a nitro group for imidazo[4,5-c]pyridine derivatives was accomplished for the first time in the experiment described above. No other nitro derivatives other than II and IV were detected in the nitration of I and III.



EXPERIMENTAL

4-Nitro-1,3-dihydro-2H-imidazo[4,5-c]-2-pyridone (II). A solution of 1 g of I in 4 ml of concentrated H₂SO₄ was mixed with 0.73 g of KNO₃ in 4 ml of H₂SO₄, and the mixture was heated at 110–115° for 2 h and poured over ice. The aqueous mixture was neutralized with ammonia to give prisms with mp > 400° (dimethylformamide) in 99% yield. Found: C 39.8; H 2.5%. C₆H₄N₄O₃. Calculated: C 40.0; H 2.2%.

4-Nitro-1-methyl-1,3-dihydro-2H-imidazo[4,5-c]-2-pyridone (IV). This compound was similarly obtained in 95% yield as colorless rods (from water or alcohol) with mp 309–310° (dec.). Found: C 43.6; H 3.3%. C₇H₆N₄O₃. Calculated: C 43.3; H 3.1%.

1-Methyl-1,3-dihydro-2H-imidazo[4,5-c]-2-pyridone (III). This compound was obtained in 80% yield by fusion of 3-amino-4-methylaminopyridine with urea at 170°. The colorless prisms (from dioxane) had mp. 272°. Found: C 56.6; H 4.7%. C₇H₇N₃O. Calculated: C 56.4; H 4.7%.

LITERATURE CITED

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2. G. Cleve, H. Gibian, G.-A. Hoyer, D. Rahtz, E. Schröder, and G. Schulz, *Ann.*, **747**, 158 (1971).

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