CONVERSION OF NICOTYRINE METHIODINE TO 1-METHYL-7-FORMYLINDOLE

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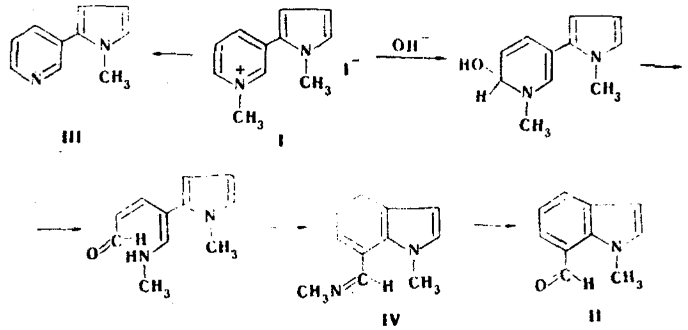
We have found that nicotyrine methiodide (I) is converted to 1-methyl-7-formylindole (II) when it is heated with a mixture (1:2) of methylamine bisulfite and 30% aqueous methylamine in a sealed ampul at 150°C for 60-80 h. The yield of product with mp 82-84°C is 25% after separation by means of preparative chromatography on silica gel (100/160 μ) in a benzene–ethyl acetate system (10:4). IR spectrum (in mineral oil): 1690 cm^{-1} (C=O). UV spectrum (in methanol), λ_{max} (log ε): 226 (4.38), 247 (4.34), and 340 nm (3.92). PMR spectrum (in CCl_4, with hexamethyldisiloxane as the internal standard): 3.30 (s, 1-CH_3), 5.65 (d, 3-H, J_{23} = 4 Hz), 6.15 (d, 2-H), 6.32 (dd, 5-H, $J_{45} = J_{56}$ = 8 Hz), 6.75 (dd, 4-H, J_{46} = 2 Hz), 6.95 (dd, 6-H), and 9.22 ppm (s, CHO). Mass spectrum, m/e: 159 (M^+), 158, 130, and 103. The results of elementary analysis for C, H, and N were in agreement with the calculated values.

The rearrangement also takes place under the influence of an aqueous alcohol solution of alkali, but the yield of aldehyde II is lower in this case. The initial product is evidently the pseudo base, which undergoes conversion to the open form; the latter undergoes ring closure with attack on the nucleophilic pyrrole ring to give the aldimine. Under the reaction conditions the aldimine is hydrolyzed to aldehyde II. Aldimine IV was detected in the reaction mixture by chromatographic mass spectroscopy.



Alternative attack of the nucleophilie on the CH_3–N bond leads to demethylation and the formation of nicotyrine (III), which was isolated in 43% yield.

The described conversion is yet another example of recyclization with the conversion of the pyridine ring to a benzene ring under the influence of a nucleophilic agent.

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