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SHORT COMMUNICATIONS

Oxidation of 1-Oxa-5-azaspiro[5.5]undecane with Cyclohexanone

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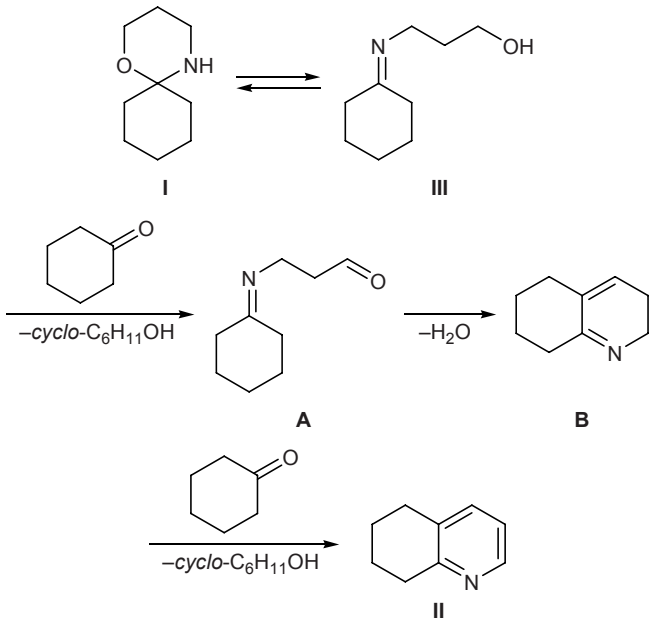
We previously reported that oxidation of five-membered cyclic N,O-acetals (oxazolidines) with carbonyl compounds leads to the formation of pyrroles [1]. In the present communication we describe the oxidation of 1-oxa-5-azaspiro[5.5]undecane (**I**) with cyclohexanone to produce 5,6,7,8-tetrahydroquinoline (**II**) as an example of synthesis of pyridine derivatives via oxidation of perhydro-1,3-oxazines having no substituent on the nitrogen atom with carbonyl compounds.



Presumably, as in the oxidation of oxazolidines with carbonyl compounds, initial oxidation of imino alcohol **III** (an open-chain tautomer of perhydro-1,3-oxazine **I**) gives imino aldehyde **A** which undergoes intramolecular condensation (like crotonization) to 2,3,5,6,7,8-hexahydroquinoline **B**, and the latter is oxidized with the second cyclohexanone molecule to quinoline **II**.

A mixture of 31 g (0.2 mol) of compound **I**, 39.2 g (0.4 mol) of cyclohexanone, and 4.48 g (0.08 mol) of powdered potassium hydroxide was heated for 5 h under reflux. The mixture was cooled, 25.2 ml (0.21 mol) of 36% hydrochloric acid was added, and the aqueous phase was separated, extracted with diethyl ether (3×50 ml), made alkaline by addition of potassium hydroxide, and extracted with benzene. The benzene extract was subjected to distillation to isolate 4.2 g (16%) of 5,6,7,8-tetrahydroquinoline **II** with bp 89–93°C (10 mm), d_4^{20} = 1.0314, n_D^{20} = 1.5428; published data [2]: bp 92–95°C (12 mm), d_4^{20} = 1.0304, n_D^{20} = 1.5435. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.74 m (4H, 6-H, 7-H), 2.68 t (2H, 5-H), 2.80 t (2H, 8-H, $J_{5,6}$ = 6.0, $J_{7,8}$ = 6.0), 7.0 d.d (1H, 3-H, $J_{3,4}$ = 8.0), 7.33 t (1H, 4-H), 8.24 d (1H, 2-H, $J_{2,3}$ = 5.0). Found, %: C 81.33; H 8.26; N 10.41. $C_9H_{11}N$. Calculated, %: C 81.16; H 8.32; N 10.52.

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