SYNTHESIS OF HEXAHYDROINDOLE

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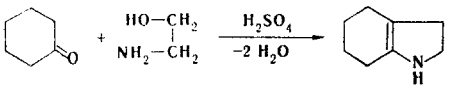
Hexahydroindole is formed in the condensation of 60 g (1 mole) of monoethanolamine with 98 g (1 mole) of cyclohexanone in the presence of 9.8 g (0.1 mole) of concentrated H_2SO_4 when the mixture is stirred and heated at 90°C for 6 h.



The organic layer was separated from the acid layer, washed with 10% sodium carbonate solution, dried with solid KOH or Na_2CO_3, and subjected to distillation, with selection of the fraction with bp 180-185°C, which was analyzed by gas–liquid chromatography (with an LKhM-8MD chromatograph and detection by thermal conductivity; the thermostat temperature was 150°C, the column was 3-m long and had a diameter of 3 mm, the stationary phase was Apiezon M on Celite-545, and the helium flow rate was 40 ml/min). The purity was 98-98.5%. The yield of 2,3,4,5,6,7-hexahydroindole, with n_D^{20} 1.4850 and d_4^{20} 0.9179, was 56%. IR spectrum (in KBr): 3400-3420 and 1490-1580 (NH), 1500-1600 (C=C), and 2850 cm^{-1} (C–H).

A multiplet of cyclohexane ring protons at 1.5 ppm (8H), a triplet at 3.03 ppm (3H, α-H and NH), and a triplet at 3.66 ppm (2H, 3-H) with $J_{2,3}$ = 10 Hz are observed in the PMR spectra (in hexamethyldisiloxane, 60 MHz). A dibromide (prisms with mp 61-62°C) is formed when the product is treated with bromine.

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