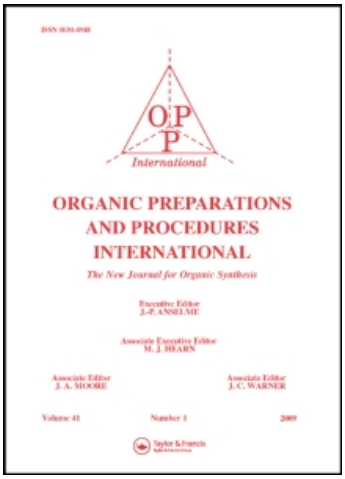

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3,3-DIMETHYLINDOLINE

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3,3-DIMETHYLINDOLINE

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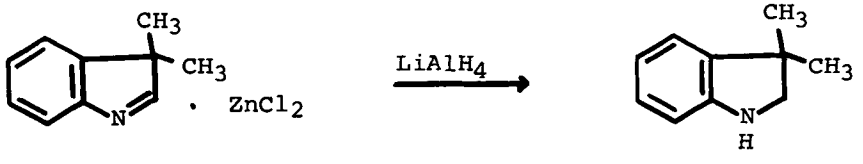
Adkins and Burks[2] have described the hydrogenation of 3,3-dimethylpseudoindole(I) to 3,3-dimethylindoline(II) at 500 psi over a copper chromite catalyst. Difficulties were experienced in attempts to repeat this work: two products were obtained in about equal amounts and resolved by gas chromatography. However, neither exhibited the expected -NH- band in the infrared and they were different from the desired indoline(II). The reduction of the zinc chloride salt of (I) to (II) can be effected conveniently, but in poor yield, using lithium aluminum hydride. No attempt has been made to carry out runs on a large scale. It seems clear that



(I) (II)

Adkins and Burks had indeed obtained II but the quality of their catalyst must have been superior to that prepared according to the procedure given by Vogel.[3]

Experimental

Dry nitrogen was bubbled through a solution of anhydrous zinc chloride (from 60 g. of the hydrate) in ethanol (100 ml.) and isobutyraldehyde phenylhydrazone (14.3 g.) was added. The solution was heated on a steam-bath for 20 min., cooled and acidified with cold 3 N hydrochloric acid to give a yellow precipitate. After several hours, the zinc chloride salt of 3,3-dimethylpseudoindole was filtered and recrystallized from absolute ethanol to give the product (3.5 g., 14.2%),m.p. 167-170° (lit.[4] m.p. 170-172°).

The salt (2.0 g.), dissolved in tetrahydrofuran (100 ml.), was treated with a slurry of lithium aluminum hydride (0.2 g.) in tetrahydrofuran (75 ml.). The mixture was boiled under reflux for 5 hours, cooled, and water (0.2 ml.), 10% aqueous sodium hydroxide (0.2 ml.), and more water (0.6 ml.) were added. The mixture was stirred, allowed to stand for ½ hr. at room temperature and the precipitate filtered. The solution was dried ($MgSO_4$) and the solvent evaporated to give a liquid. Distillation gave 3,3-dimethylindoline (0.2 g. 19.1%), b.p. 99-100°/16 mm. This was finally purified by preparative gas-liquid chromatography on an Apiezon L column and has m.p. 32-32.5° (lit.[2] m.p. 31-33°). I.R. spectrum (Nujol mull) (main peaks only): 3480(m), 1600(s), 1480(s), 1455(s), and 735 $cm.^{-1}$(s).

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