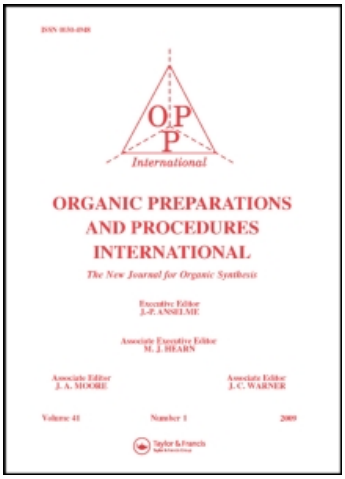

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ORGANIC SONOCHEMISTRY. A FACILE SYNTHESIS OF 1-METHYLISOQUINOLINE

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ORGANIC SONOCHEMISTRY. A FACILE SYNTHESIS OF 1-METHYLISOQUINOLINE

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(05/02/84)

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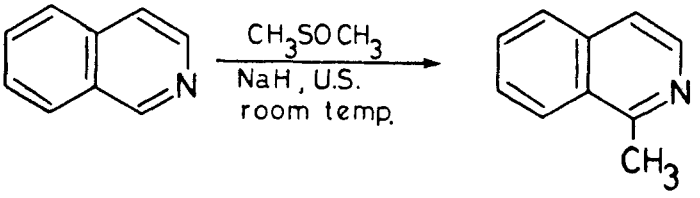
1-Methylisoquinoline has been prepared mainly by catalytic dehydrogenation of 1-methyl-3,4-dihydroisoquinoline obtained by Bischler-Napieralsky reaction,[1] or by alkylation of the isoquinoline Reissert compound.[2] As the latter procedure could not be performed by our group under phase-transfer catalysis,[3] we have adapted an interesting procedure



from Russell and Weiner[4] by generating methyl sulfinyl carbanion ("Corey base") under ultrasound[5] in the presence of isoquinoline. The method (72-

76%) produces slightly better yields than Russell's procedure,[6] but the speed and simplicity are superior to existing procedures.

EXPERIMENTAL SECTION

To a solution of 6.45 g (6 ml, 50 mmoles) of isoquinoline in 200 ml of dry DMSO,[7] 10.6 g (0.22 mole) of a 50% mineral oil dispersion of sodium hydride were added. The reaction mixture was irradiated with ultrasound in a flask protected from moisture with a calcium chloride tube, for two hrs at room temperature;[8] then the reaction mixture was poured over 600 ml of water. The resulting aqueous solution was extracted with ether in a continuous liquid-liquid extractor for 24 hrs. The ethereal layer (ca. 150 ml) was extracted with three 30 ml of 10% HCl, then the aqueous phase was basified to pH 8-9 with aqueous 10% NaOH and extracted again with three 50 ml of CH_2Cl_2. Finally, the dried organic extract was concentrated to ca. 20 ml, saturated with dry HCl and ethyl ether was added until complete precipitation. The hydrochloride of 1-methylisoquinoline was collected and crystallized from ethanol/ether to yield 5.15-5.64 g (72-76%),[9] mp. 208-209° (dec.), lit.[10] 200-205°.

Anal. Calcd for $C_{10}H_{10}ClN$: C, 66.85; H, 5.61; N, 7.79

Found: C, 66.68; H, 5.64; N, 7.94

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6. We have reproduced the process described in ref. 4 and the yields never exceeded 65-70%.

7. Commercial DMSO (max. 0.03 water) was used with same results.
8. Ultrasound irradiation was carried out by immersion of the reaction flask in a Branson Ultrasound Laboratory Cleaner (150w, 50-60 Hz); the temperature rose to 25-30° during the irradiation.
9. NMR analysis ($CDCl_3$, TMS) of the crude base showed a δ 9.4 signal corresponding to the H at carbon 1 of unreacted isoquinoline, which remained in concentrations of ~ 5% after the process; it disappeared after one crystallization.
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STEROIDAL ALLYLIC OXIDATION WITH CHROMIUM TRIOXIDE IN THE PRESENCE OF PYRAZOLE

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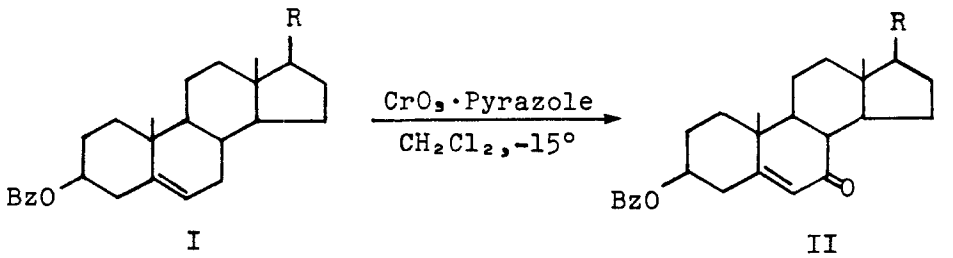
Synthetically useful changes in the properties and reactivity of chromium(VI) reagents have been brought about by the formation of amine complexes. Complexation of chromium trioxide with pyridine[1-3] or 3,5-dimethylpyrazole (DMP)[3,4] has been successfully used for the introduction of



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