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7-METHYLINDOLE

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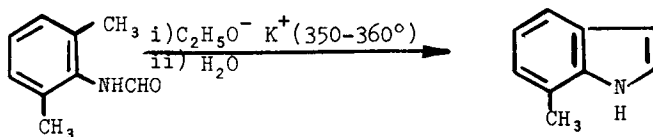
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7-METHYLINDOLE

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7-Methylindole is commercially available¹ but it is expensive. It may be conveniently prepared in reasonable yield by cyclization of 2,6-dimethylformanilide with potassium ethoxide by the Tyson method.^{2,3} The procedure described is essentially the same as that reported by Hoffmann, Ikan and Galun,⁴ in which potassium *t*-butoxide is used as the cyclizing reagent.

EXPERIMENTAL

2,6-Dimethylformanilide - A mixture of 2,6-dimethylaniline (30 g.) and formic acid (98%, 12 ml.) was heated under reflux (water bath) for 5 hr. The product was crystallized from benzene-light petroleum to give 34 g. (91%) of 2,6-dimethylformanilide as colourless needles, mp. 175°, lit.⁵ 176-177° (rapid heat).

7-Methylindole - 2,6-Dimethylformanilide (25.2 g.) was added to a solution of potassium ethoxide, prepared by dissolving potassium

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(9.8g) in absolute ethanol (110 ml.) under nitrogen. The excess of ethanol was removed by distillation under nitrogen, and the residue was heated under nitrogen at 350-360⁰ on a sand-bath for 30 min.⁶ The reaction mixture was allowed to cool in the stream of nitrogen, and then water (200 ml.) was added and the mixture was steam-distilled. The distillate was extracted with ether and the extract was washed successively with dilute 5% hydrochloric acid (to remove any 2,6-dimethylaniline), 5% sodium carbonate solution and water, and dried (Na₂SO₄). The solvent was removed and the residue was crystallised from water to give 11.0 g. (50%)⁷ of 7-methylindole as colourless plates, mp. 84-85⁰,⁷ lit.³ 85⁰.

Anal. Calculated for C₉H₉N: C,82.44; H,6.87; N,10.69%.

Found: C,82.32; H,6.96; N,10.72%.

The picrate was obtained as red needles from methanol, mp. 177⁰, lit.³ 177.5⁰.

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4. E. Hoffmann, R.Ikan and A.B. Galun, J. Heterocyclic Chem., 2, 298 (1965).
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6. A liquid, which is principally 2,6-dimethylaniline, distils.
7. Hoffmann et al., ⁴ report a yield of 42.5% and m.p. 82⁰ (from hexane).

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