NOTE.

A Preparation of 8-Hydroxyquinoline. By F. E. KING and J. A. SHERRED.

8-METHOXYQUINOLINE, obtained by Skraup (*Monatsh.*, 1882, **3**, 544) from *o*-anisidine and glycerol in presence of 2-nitroanisole, has been prepared with arsenic pentoxide as oxidising agent. Its demethylation, previously effected by aniline hydrochloride under pressure (Riedel, D.R.-P. 583,705), in nearly quantitative yield, simply with boiling hydrobromic acid, is now described.

8-*Methoxyquinoline.*—Concentrated sulphuric acid (100 g.) was cautiously added to a mixture of redistilled *o*-anisidine (44·5 g.), arsenic pentoxide (51·5 g.), and glycerol (110 g.), and the moderate reaction initiated by heating under reflux. After 4 hours' boiling, the liquid was diluted with water to 1 l. and made slightly alkaline with concentrated aqueous sodium hydroxide. The precipitated oil was separated and distilled. The main fraction, b. p. 174—176°/29 mm. (yield 27%), was the pure methoxyquinoline, m. p. 45°, of which the picrate, crystallised from alcohol and dried in a vacuum, had m. p. 162° (Found : C, 49·4; H, 3·1. Calc. : C, 49·5; H, 3·1%). Fränkel and Grauer (*Ber.*, 1913, **46**, 2552) give m. p. 143° (decomp.) with discolouration at 130°.

8-*Hydroxyquinoline*.—A solution of the methoxyquinoline (5 g.) in hydrobromic acid (30—40 c.c.; *d* 1·5) was refluxed in glass-jointed apparatus for 3—4 hours. Crude 8-hydroxyquinoline could then be precipitated by solid sodium carbonate, or the acid first recovered by evaporation under diminished pressure. The phenolic base was liberated from the viscous hydrobromide by approximately the calculated amount of carbonate solution and distilled (yield, above 90%); b. p. 122°/0·1 mm., m. p. 77°. As recorded by Skraup (*loc. cit.*), the picrate crystallised in yellow prisms, sintering *ca.* 170°, m. p. 203—204° (Found : C, 48·1; H, 2·7. Calc. : C, 48·1; H, 2·7%).—DYSON PERRINS LABORATORY, OXFORD. [*Received, September 5th*, 1941.]