NOTE.

The Preparation of m-*Nitrobenzenesulphonyl Chloride.* By HERBERT H. HODGSON and JOHN S. WHITEHURST.

THE directions for making this compound from nitrobenzene and chlorosulphonic acid given in D.R.-P. 89,997 (cf. Friedländer, 1894—1897, IV, pp. 38—39) are insufficient for obtaining consistent results on a laboratory scale, and the following more detailed description has been found satisfactory : Dry nitrobenzene (123 g., 1 g.-mol.) and chlorosulphonic acid (350 g., 3 g.-mols.) are stirred together at room temperature until thoroughly mixed (any temperature rise indicates water in the nitrobenzene), the temperature is then raised gradually to 100°, maintained there for 2 hours, and thereafter kept at 110—120° for 6 hours. The initial yellow colour has now changed to brown and the reaction is completed by a final heating at 125—130° for 2 hours, *i.e.*, a total heating period of 10 hours. After cooling, the solution is poured into a mixture of ice (1 kg.) and water (1 kg.), and the white precipitate collected, washed with water, and air-dried. Yield, 170 g. (76% of the theoretical); m. p. 54°. The crude *m*-nitrobenzenesulphonyl chloride is purified by dissolution in glacial acetic acid (500 c.c.), the filtered solution cooled below 15° (otherwise dilution with water precipitates the compound as a semi-fused mass), and water (200 c.c.) added cautiously with stirring; *m*-nitrobenzenesulphonyl chloride then separates in colourless prismatic needles, which are washed with 50% acetic acid and air-dried; m. p. 62° (Armstrong, Colgate, and Rodd, *Proc. Roy. Soc.*, 1914, [*A*], **90**, 111, give m. p. 61·5—62°). Yield of purified product, 124 g. (55%).

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