NOTES.

3-*Chlorophthalic Acid.* By J. C. SMITH.

3-NITROPHTHALIC anhydride (10 g., m. p. 163°) and phosphorus pentachloride (11 g.) were heated in a sealed tube for 9 hours at 190—200° (compare Bogert and Boroschek, *J. Amer. Chem. Soc.*, 1901, **23**, 751). The solid obtained was separated from volatile impurities by heating in a vacuum cautiously to the m. p.; from its filtered solution in benzene, ligroin precipitated colourless crystals, m. p. 122° (yield, 60%), and 126° after recrystallisation from benzene–ligroin. Hydrolysis of the anhydride, m. p. 122° or 126°, gave an acid, m. p. 200—202° (decomp.) (Found: C, 48·0; H, 2·5; equiv., 100·5. Calc.: C, 47·9; H, 2·5%; equiv., 100·2). Direct hydrolysis of the reaction products from the sealed tube by heating with water (vigorous reaction) gave 90% yields of 3-chlorophthalic acid, but one experiment in which impure 3-nitrophthalic anhydride (m. p. 156—159°) was used yielded a product, m. p. 185° (decomp.) (equiv., 103), unchanged by crystallisation. Addition of the acid, m. p. 202°, to this gave a mixture, m. p. 186° (Bogert and Boroschek's acid melted at 186°). Reaction with acetic anhydride yielded slightly yellow crystals, m. p. 124° and 126° after recrystallisation (equiv., 90·7. Calc., 91·2); hydrolysis gave the acid, m. p. 200—202°. 3-Chlorophthalic acid when pure is sparingly soluble in cold water. Its solubility in ether enables it to be separated from *o*-phthalic acid, m. p. 209° (decomp.).—THE DYSON PERRINS LABORATORY, OXFORD UNIVERSITY. [*Received, October* 20*th*, 1933.]

A Synthesis of Tricin. By K. C. GULATI and K. VENKATARAMAN.

BY a mixed m. p. determination and by colour reactions, synthetic 5 : 7 : 4′-trihydroxy-3′ : 5′-dimethoxyflavone (Gulati and Venkataraman, this vol., p. 942) has been identified, as already suggested (*Current Science*, 1933, **1**, 238), with tricin, the colouring matter of " Khapli " wheat, for a specimen of which we are greatly indebted to Dr. J. A. Anderson of the National Research Council of Canada.—FORMAN CHRISTIAN COLLEGE, LAHORE. [*Received, September* 19*th*, 1933.]

An Improved Preparation of Selenophen and Attempts to prepare Tellurophen. By FRANCIS A. MCMAHON, THOMAS G. PEARSON, and PERCY L. ROBINSON.

THE synthesis of thiophen and selenophen from the respective elements in acetylene (Briscoe and Peel, J., 1928, 1741; Peel and Robinson, *ibid.*, p. 2068) is facilitated by catalysts (compare Tschitschibabin, *J. Russ. Phys. Chem. Soc.*, 1915, **47**, 703): selenium with roasted bauxite in a furnace gave 25 g./24 hr., but selenium alone gave only 15 g.; from tellurium, no tellurophen was obtained. For both selenophen and tellurophen the best packing was roasted bauxite with aluminium selenide or telluride; at 400°, the former gave 20·25 g./24 hr. of a liquid (15% of selenophen and neither naphthalene nor other solid), the latter, 20 g./24 hr. of a liquid containing tellurium (at 600°, Te absent). 282 G. of the latter liquid prepared at 350° consisted of 61 g., b. p. $< 100°$; 62 g., b. p. 100—184°; and 159 g., b. p. $> 185°$. Tellurium was found only in the pale yellow middle fraction (*d* 0·98 g./c.c.), but was not segregated by fractionation; it was also present in the precipitates formed by this fraction with mercuric sulphate and acetate. Selenophen forms a white crystalline triphenylmethane derivative, m. p. 57° (Found : $CHPh_3$, 64·8; C_4H_4Se, 35·2. Calc. for $CHPh_3,C_4H_4Se$, 65·0 and 35·0%, respectively), from which selenophen may be recovered by heating. A corresponding compound appeared to be formed from the above distillate, and tellurium was certainly present in the product obtained from it by heating.

An attempt was made to obtain tellurophen by modifying Volhard and Erdmann's synthesis of thiophen (*Ber.*, 1885, **18**, 454), *viz.*, heating sodium succinate with phosphorus trisulphide. Foa (*Gazzetta*, 1909, **39**, 527) claims thus to have prepared selenophen, but we have obtained only traces of possibly selenophen from 30 g. of the succinate and 20 g. of the selenide, and no tellurophen with aluminium telluride alone and with red phosphorus. Paal and Tafel (*Ber.*, 1885, **18**, 456) got thiophen-α-carboxylic acid by heating mucic acid with barium sulphide. Aluminium selenide or telluride at 200°, however, did not give acids containing these elements, nor was selenophen or tellurophen formed from tetramethylene glycol and aluminium selenide or telluride.

The liquid obtained by heating aluminium telluride with bauxite in acetylene contains a tellurium compound, b. p. 120—150°, believed to be tellurophen, the probable b. p. of which is 136°.—UNIVERSITY OF DURHAM, ARMSTRONG COLLEGE, NEWCASTLE-UPON-TYNE. [*Received, August* 11*th*, 1933.]
