Inorganic

Routine Analysis of Manganese Bronze. E. K. Babson and W. W. Johnson (*Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 292–293)—The method is claimed to give a complete analysis of manganese bronze with sufficient accuracy for control and inspection purposes in approx. 5 hr. The sample is dissolved in perchloric and nitric acids and a pptn. with hydrogen sulphide separates copper, lead and tin from iron, aluminium, manganese, nickel and zinc. Manganese is determined on a separate sample by existing methods (Scott, "*Standard Methods of Chemical Analysis*," 5th Ed., Vol. II, p. 1358, New York, D. Van Nostrand Co., 1939) and zinc is determined by difference.

Method—Weigh 1·000 g. of the bronze into a 400-ml. beaker, dissolve in 4 ml. of conc. nitric acid and 6 ml. of perchloric acid (70%), cover, and heat until fumes of perchloric acid are evolved for several min. Cool, dilute to 200 ml. with hot water and pass hydrogen sulphide for 20 min. Add paper pulp, coagulate the ppt. by heating the soln. to boiling, filter (Whatman No. 30) and wash the ppt. thoroughly with hot water.

Tin—Wrap the sulphide ppt. and filter paper in another filter paper, place in a clean porcelain crucible and ignite at 500° C. Cool. transfer the residue to a 150-ml. beaker and heat 10 ml. of conc. nitric acid in the crucible for several min. Pour the acid into the beaker and wash the crucible with 10 ml. of water, adding the washings to the acid. Heat until the black copper oxide dissolves, dilute to 50 ml. with hot water, add paper pulp and keep the soln. nearly at the boiling point for 30 min. Filter through a Whatman No. 42 paper containing paper pulp. Wash the ppt. 3 times, alternately with hot water and hot diluted nitric acid (1+4). Burn off at 500° C. in a weighed crucible, ignite at 900° C., cool and find the weight of SnO_2.

Copper and Lead—Dilute the filtrate from the tin ppt. to 200 ml. and add 2 ml. of diluted sulphuric acid (1+1) and a small amount of urea or sulphamic acid. Electrolyse to deposit the copper on a weighed

gauze cathode and the lead as PbO_2 on the weighed rotating gauze anode. With customary electrodes the process takes about 60 min. with a current of 2·5 amp. Reweigh the electrodes.

Iron—To the filtrate from the sulphide pptn. add a few glass beads to prevent bumping and boil the soln. vigorously for 30 min. to expel hydrogen sulphide. Cool in ice water, add 10 ml. of diluted sulphuric acid (1+1) and titrate with 0·05 *N* potassium permanganate until a faint pink colour persists for 10 to 15 sec.

Aluminium—Add 10 g. of ammonium chloride to the titrated iron soln., and add ammonia soln. (sp.gr. 0·90) until just alkaline to methyl red. Add paper pulp, boil, add 3 drops of ammonia soln. and leave for 5 min. Filter (Whatman No. 31) and wash 3 times with hot 10% ammonium nitrate soln. which has been made just ammoniacal to methyl red. Reserve the filtrate for the nickel determination. Dissolve the ppt. into the pptn. beaker with hot diluted hydrochloric acid (1+1). Add 2 g. of ammonium chloride to the soln. and reppt. the hydroxides as above. Filter, wash, burn off the paper at 500° C. in a weighed porcelain crucible, ignite at 900° C., cool and find the weight of $Fe_2O_3 + Al_2O_3$. Calculate the amount of aluminium.

Nickel—Add to the filtrate from the first ammonia pptn. 15 ml. of 1% alcoholic dimethylglyoxime soln. Warm until the ppt. coagulates, filter through a Whatman No. 41 paper, and wash 8 to 10 times with hot water. Wrap the filter in moist ashless filter paper, ignite in a weighed, covered porcelain crucible, first at 500° C. and then at 800° C., cool and weigh the NiO.

The precision and accuracy of the results of analyses of standard samples are good; possibly fortuitous compensation of errors leads to better results than might be expected. Presence of silicon in the sample appears to lead to high values for tin and aluminium. L. A. D.