

INORGANIC ANALYSIS.

Estimation of Sulphuric Acid in Presence of Iron. Determination of Pyrrhotite in Pyrites. O. Herting. *Zeits. angew. Chem.*, 1899, 274.)—Provided the instructions are carefully followed, Herting has always found Lunge's process quite accurate; and comparative experiments with Küster and Thiel's methods (ANALYST, this vol., p. 137) show that the latter are less rapid and convenient.

The author also calls attention to the frequent presence of pyrrhotite in American pyrites, which is not completely burnt in the usual furnaces, and refers to Cone's process (ANALYST, 1893, xxi., 223) for its estimation. F. H. L.

Estimation of Sulphuric Acid in Presence of Iron. G. Lunge. (*Zeits. anorg. Chem.*, 1898, xix., 454.)—This is a protest against the inference contained in Küster and Thiel's article (ANALYST, this vol., p. 137) that Lunge's process is tedious and inaccurate. It can be carried out completely in two or three hours; whereas the very removal of the iron in the methods suggested by the former authors occupies four hours. As regards accuracy, Jannasch himself has admitted that the Lunge process is trustworthy (*J. pr. Chem.* [2], xl., 236). F. H. L.

Determination of Tungsten in Steel. G. Auchy. (*Journ. Amer. Chem. Soc.*, 1899, xxi., 239.)—In the estimation of tungsten in steel the amount of acid in

the liquid from which the tungstic acid and silica are filtered should be as small as possible; and the preceding evaporation to dryness to render them insoluble should take place in hydrochloric (not nitric) acid solution. Evaporation of a nitric acid solution does not lead to a complete separation of the tungsten; but in presence of hydrochloric acid, provided an excess be not employed, only one evaporation to total dryness is necessary. The best method of procedure is to dissolve 4 grammes* of the steel in dilute nitric acid, evaporating until a scum appears on the surface, adding 30 or 40 c.c. of strong HCl, evaporating to dryness and heating on the hot plate, dissolving in 40 c.c. of strong HCl, evaporating again until a scum forms, taking up in 5 c.c. of HCl and 15 c.c. of water, heating, diluting with 25 or 30 c.c. of hot water and filtering.

At least in the case of steels low in phosphorus, the tungstic acid need not be removed before estimating the former; but the yellow molybdate precipitate must not be allowed to stand more than 2 or $2\frac{1}{2}$ hours. When neutralizing with ammonia, excess must be avoided, or tungstic acid will be thrown down and remain undissolved in the nitric acid subsequently introduced. The usual method of getting ferro-tungsten into solution depends on a preliminary roasting; the author has obtained unsatisfactory results when operating in this manner. He prefers to attack the alloy by heating it with strong nitric acid, adding strong hydrochloric acid by degrees, and then following the above scheme. A metallic residue is left consisting apparently of almost pure tungsten; but this is readily oxidized by ignition after it has been removed by filtration.

F. H. L.

Some Reactions of Chloric, Bromic, and Iodic Acids. D. Vitali. (*Giorn. Farm. Trieste*, 1899, iv., 39; through *Chem. Zeit. Rep.*, 1899, 99.)—When a solution of manganous sulphate acidified with sulphuric acid is treated with a bromate, a fine violet colour is produced, which is due to a mixture of basic manganese sulphate and free bromine. The salts of chloric and iodic acids yield no colours. This reaction may be used as a means of determining manganese; for if the violet liquid is boiled for some time, the metal is finally precipitated as dioxide.

Salts of iodic acid are decomposed in the cold by hydroxylamine sulphate, iodine being liberated; bromates only behave similarly on warming; chlorates remain unreduced. An analogous reaction occurs with compounds of phenylhydrazine, but the iodine, instead of being set free, combines with the phenyl group, producing a red precipitate which presumably consists of triiodophenol.

Hypophosphorous acid decomposes iodates in the cold, liberating iodine at first and yielding finally a mixture of a phosphate and hydriodic acid; bromates are attacked on warming; chlorates not at all.

F. H. L.

Titration of Persulphates. M. le Blanc and M. Eckardt. (*Zeits. Elektrochem.*, 1899, v., 355; through *Chem. Zeit. Rep.*, 1899, 62.)—Processes for determining the active oxygen in solutions of persulphates which depend on addition of

* If 3.9655 grammes be weighed out, the yield of WO_3 multiplied by 20 gives directly the percentage of tungsten in the steel.

ferrous ammonium sulphate, followed by titration with permanganate, are apt to give too low results, unless the temperature during the operation be raised. The employment of a large excess of iron salt serves the same purpose, but warming is simpler and more accurate.

F. H. L.

A New Indicator for Acidimetry. E. Riegler. (*Bulet. Soc. de Sciinte din Bucuresti*, 1899, vii., 453.)—This body consists of the diazo colour which is prepared by treating an alkaline solution of guaiacol with diazoparanitraniline. It is a brown substance, insoluble in water, and soluble in alcohol. 0.20 gramme dissolved in 100 c.c. of spirit forms an indicator exceeding phenolphthalein in sensitiveness, bright red in alkaline liquids, greenish-yellow with acids.

F. H. L.
