

ANALYTICAL CHEMISTRY.

On the Determination of Phosphorus in Iron and Iron Ores.—A. ZAMM.

Dissolve the iron in HNO_3 (1.20) using 12c.c. per grm. of iron, boil briskly to dryness and heat for 1 hour at 200°C . Then add 6c.c. of HCl (1.12) per grm. of iron, boil to dryness, add HCl , boil again to near dryness, and finally, add 1–2 volumes of water and filter off silica. Strong heating after action of HNO_3 ensures complete precipitation by molybdate.

Add an equal volume of the molybdate, prepared according to Eggertz' directions, to the solution of iron, which should not exceed 20c.c. per grm. of iron. If the proportion of phosphorus is up to several tenths of one per cent. add an excess of molybdate, equal to 2c.c. for each .001 grm. of P. Heat for 1 hour at 40° , remove the liquid with a syphon having a lateral opening near the closed end of the shorter limb (to prevent disturbance of the precipitate) wash on a very small filter with 1 % solution of HNO_3 (1.20) dry and weigh. The yellow precipitate contains 1.64% of P. The filter should have been previously dried at 120° and weighed or when great accuracy is not desired, 5 per cent. of the weight of the undried filter may be deducted for moisture. In absence of arsenic the precipitation may be completed in 1 hour at 50° . (*Chem. News*, **49**, 208.)

A. A. B.

New method of determining Carbon in Cast Iron. M. ZABOUDSKY.

The finely pulverized metal is mixed with a dry mixture of copper and sodium chlorides (prepared by evaporating to dryness a solution of copper sulphate with sodium chloride). About 14 grams. of the mixture, containing 4.8 grams. CuCl_2 , is taken for each gramme of iron. After thorough mixing (dry) in a mortar, water sufficient to form a paste is added, and the mass is triturated for about half an hour. The mass is then transferred to a beaker, the mortar rinsed into the beaker with ferric chloride solution (1 to 4), and 200 c.c. of the latter added. After gentle heating, hydrochloric acid is added. If properly managed at this point, only the carbonaceous material should remain undissolved; this should be filtered through asbestos, dried at 125° to 130°

and weighed. The residue is not pure carbon, as has been usually supposed, but contains hydrogen and oxygen in combination with it. The amount obtained must be multiplied by one of the following factors to obtain the actual amount of carbon present :

	Factor.
From Spiegel, not manganiferous	0.720
Ferromanganese	0.700
Spiegel, manganiferous	0.685
White pig	} 0.710
Pure gray pig	
Very pure gray pig with a little combined C	0.655
Cast steel of guns and muskets containing about 0.5% C.	0.660
Hard steel	0.675
Iron	0.690

The author finds the results more satisfactory than by the Eggertz iodine method. (*Bul. Soc. Chim.*, 41, 428.) E. W.

The estimation of Sulphurous Acid in its compounds.
 W. B. GILES and A. SHEAREE.

The authors incidentally note that crystallized sodium sulphite invariably contains 7H₂O, the 10 H₂O crystals having never been noticed by them. Fresenius' method of analysis, with iodine solution, was found to give results which were constant but invariably low. The difficulty in obtaining and keeping water free from dissolved air, also proved to be a strong objection to the process. The method which they propose consists in pouring upon the material (after weighing out), at once, *without dissolving*, an excess of $\frac{1}{10}$ normal iodine solution, effecting solution in this, and titrating back with thiosulphate and starch. In the case of fluids they may be sealed up in thin glass bulbs, which may be broken under the iodine solution. After a preliminary approximate test, a second is made, using only a slight excess of the $\frac{n}{10}$ I solution. Attention is called to the siphons of liquid SO₂, furnished for laboratory use by a manufacturer in England. (*Jour. Soc. Chem. Ind.*, 3, 197).

E. W.

Determination of Phosphoric Acid in Arable Soils.—By
 G. LECHATIER.

In two communications presented by M. de Gasparin, one in

1883, and another in January, 1884, (this journal, vol. VI., p. 137), the following process is given for the determination of phosphoric acid in arable soils.

The specimen of soil is treated by *aqua regia*, and the soluble portion obtained is precipitated by ammonia. The precipitate is collected, dried, strongly heated, pulverized and treated with cold nitric acid of $\frac{1}{10}$ strength. Phosphoric acid is dissolved and ferric oxide remains. The former is precipitated with ammonium molybdate.

The author has had to determine phosphoric acid in soils. The solutions of which in *aqua regia* contain variable quantities of lime, magnesia, potassa, soda ferric oxide, alumina, phosphoric acid and silica. Generally the quantities of lime and magnesia are very small. In these solutions, the successive addition of small quantities of ammonia, separate phosphoric acid as ferric and aluminic phosphates, while the liquid is still acid, and before calcic phosphate separates. The ferric and aluminic phosphates are not entirely soluble in nitric acid of $\frac{1}{10}$ strength.

The following experiments were made with acid solutions containing .041 gm. of phosphoric acid and varying proportions of ferric oxide.

Ferric oxide, mixed with .041 gm. phosphoric acid.	Phosphoric acid dissolved in weak nitric acid.
0.8	0.038
2.0	0.023
4.0	0.021

If the quantity of phosphoric acid is doubled, the quantity dissolved is only $\frac{3}{4}$ to $\frac{2}{3}$ of the quantity present. Nitric acid of $\frac{1}{10}$ strength was used. The remainder of the phosphoric acid was found in the calcined precipitate, showing that precipitation was complete, but solution defective. Similar results were obtained with alumina.

The author precipitates ferric oxide and phosphoric acid with milk of pure lime, so to have an excess of lime in the precipitate. This precipitate was strongly heated, reduced to a fine powder and digested at 60° C. in nitric acid (acid 2 c.c., water 200 c.c.) Phosphoric acid is then entirely dissolved, even when mixed with 80 times its weight of ferric oxide. If digestion

takes place in the cold, with an acid solution of $\frac{1}{40}$, only 0.036 gm. is dissolved out of 0.041 gm. of phosphoric acid.

A certain quantity of ferric oxide is dissolved, but phosphoric acid may be separated by molybdic acid even in presence of a large quantity of ferric oxide.—(*Comptes Rend.* 98, 817.)

P. C.

On the Examination of Tanning Extracts. F. SIMAND.

The author prefers the improved Löwenthal Method (*D. Pol. J.* 246, 41, 133) for technical determinations, but calls attention to the fact that hot water dissolves more tannin from these extracts than cold water, and insists on the necessity of specifying whether hot or cold water has been used, when results of an analysis are given. The results obtained with hot and cold water extracts of quebracho, oak, beech and chestnut wood, and with valonea, oak bark and sumac respectively, exhibit differences varying from 2.45 in oak wood, to 19.66 in sumac, per 100 of tannin. (*D. Pol. J.*, 251, 472.)

A. A. B.