

Inorganic Analysis.

Analysis of Red Phosphorus. S. A. Tolkatschoff and M. A. Portnoff. (*Z. anal. Chem.*, 1930, **82**, 122–133.)—Improved methods are given for the determination of total phosphorus, phosphorus combined with oxygen, and yellow phosphorus. *Total phosphorus*: Complete oxidation to phosphoric acid was found to be readily accomplished by a mixture of bromine and nitric acid. About 0.25 gm. of phosphorus is covered with a water layer, 2 to 3 cm. deep in a 100 c.c. flask, which is then warmed; small portions of a saturated solution of bromine in

nitric acid are cautiously added, no white fumes should form above the liquid. When solution is complete (after about 20 minutes), the content of the flask is transferred to a basin, evaporated to small bulk, and the residue twice evaporated with 5 c.c. of bromine in nitric acid. The liquid is diluted, filtered, if necessary, from insoluble matter present in the sample, made ammoniacal, filtered if necessary, and precipitated with magnesia mixture. *Phosphorus combined with oxygen*: 20 grms. are weighed into a graduated 250 c.c. flask and shaken for 12 to 15 hours with 20 c.c. of 2 *N* sulphuric acid diluted with water. The volume is made up, the liquid filtered through linen, and 50 c.c. of filtrate evaporated twice with 5 c.c. portions of bromine in nitric acid. After dilution with hot water and filtration, the solution is precipitated with ammonium molybdate. *Yellow phosphorus*: 15 to 20 grms. are digested in a 100 c.c. graduated flask with carbon disulphide for 12 to 15 hours with occasional shaking. The solution, made up to the mark, is rapidly filtered in a carbon dioxide atmosphere, and 50 c.c. pipetted off. The pipette is emptied by a carbon dioxide supply into a flask containing bromine water; the flask is shaken, and more bromine added till its colour proves presence of an excess. The carbon disulphide is distilled off, and the residual liquid heated on the water bath with 5 c.c. of bromine in nitric acid. The liquid is then transferred to a basin, evaporated, and another evaporation with bromine in nitric acid made; the solution is filtered and the filtrate precipitated with magnesia mixture. Other impurities are determined, after solution of the phosphorus, by ordinary analytical methods. Moisture is determined in a vacuum desiccator; the weight becomes constant after about two days.

W. R. S.

Iodimetric Determination of Vanadium in Alloy Steels and Ferrovandium. W. Werz. (*Z. anal. Chem.*, 1930, **81**, 448-450.)—The drillings (1 to 3 grms.), in a 500 c.c. conical flask, are boiled with 50 c.c. of syrupy phosphoric acid, 175 c.c. of water, and 5 c.c. of nitric acid (1:1) till dissolved. The solution is oxidised with nitric acid, 0.5 to 1 gm. of ammonium persulphate is added, and the excess destroyed by 15 minutes' boiling. After cooling to about 70° C., any permanganic acid is destroyed with 5 to 10 c.c. of 1 per cent. oxalic acid solution. When quite cold, the solution is shaken with potassium iodide and titrated after 5 minutes with 0.05 *N* thiosulphate (1 c.c.=0.00255 gm. V). Starch is added towards the end; total volume, about 200 c.c. *Chrome steel* may give a dark residue of carbide, which is filtered off through glass wool before oxidation with persulphate. *Ferrovandium*: 1 gm. in a 500 c.c. flask is dissolved in 25 c.c. of nitric acid. The volume is adjusted, 50 c.c. transferred to a 500 c.c. conical flask, and treated with 75 c.c. of phosphoric acid, 100 c.c. of water, and persulphate as above. The method is claimed to be a rapid one.

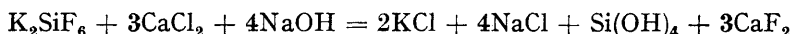
W. R. S.

Some Colour Reactions of Magnesium. I. M. Kolthoff. (*Mikrochemie Emich-Festschrift*, 1930, 180-190.)—The application of several dyestuffs for the detection of magnesium in strongly alkaline media is examined. In all cases, cobalt and nickel, and sometimes manganese, give the same test as magnesium, and, in the presence of these ions, special precautions must be taken.

Titan yellow is a delicate reagent for magnesium, and more specific than 1.2.5.8-oxyanthraquinone, which also reacts with beryllium, lanthanum and cadmium to give coloured compounds. For the titan yellow test, 10 c.c. of the solution are used, and 0.1–0.2 c.c. of a 0.1 per cent. solution of titan yellow G in water are added, and about 0.25 to 1 c.c. of 4 *N* sodium hydroxide solution. In the absence of magnesium the mixture is a brownish yellow colour; if 5 mgrms. per litre of magnesium are present the solution turns red; and for 1 mgrm. per litre, orange. The sensitivity* of the test is 0.2 mgrm. per litre, in a solution containing 2 μ gm. Small amounts of calcium salts intensify the colour. Zinc interferes with the test, unless potassium cyanide to the extent of about double the weight of the zinc present is added. Potassium cyanide also nullifies the interfering action of cobalt and nickel, but if manganese is present, it is better to remove it as sulphide. Beryllium does not interfere in the presence of sufficient sodium hydroxide, though it decreases the sensitivity of the test for magnesium. A mixture of Titan yellow and magnesium chloride can be used as a reagent for hydroxyl ions, the sensitiveness being dependent on the magnesium concentration. This reaction may also be used for spot tests on filter paper. Brilliant yellow (diaminostilbene-diphenylsulphonic acid) behaves similarly to titan yellow, but gives a less sensitive reaction. Using the same procedure as with titan yellow, the sensitivity for magnesium is 4 mgrm. per litre, and for cobalt and nickel, 2 mgrm. per litre. The reactions with *o-p*-dihydroxy-azo-*p*-nitrobenzene, Congo red, "la Motte purple," turmeric, benzopurpurin, and aniline yellow S, have also been examined. Aniline yellow S (Kahlbaum) gives a very sensitive reaction with lanthanum and copper in weakly acid medium (acetate buffer, pH about 5.0).

J. W. B.

Volumetric Determination of Silica in Silicates. N. A. Tananaeff and A. K. Babko. (*Z. anal. Chem.*, 1930, **82**, 145–150.)—The silicate (0.2 gm. SiO₂) is fused with potassium carbonate, the cake transferred to a platinum dish, and the crucible cleaned, with 20 c.c. of water. The liquid is treated with 20 c.c. of strong hydrochloric acid and 2 grms. of ammonium fluoride, and left to stand for 1 to 2 hours, with occasional stirring. The precipitated potassium fluosilicate is collected on paper in a paraffin-coated funnel, and washed 5 times with a saturated solution of potassium fluosilicate. Filter and precipitate are transferred to a conical flask with 20 c.c. of 4 *N* calcium chloride solution and water to make up about 100 c.c. The flask is warmed on a water bath, and the liquid titrated with 0.5 *N* sodium hydroxide after addition of methyl red:



The end-point is indicated by either the coagulation of the precipitate or the colour change. A blank determination is always required, and the volume deducted from that found in the assay. The determination requires 3 to 3½ hours. Deviations of 0.3 to 0.4 per cent. from the results obtained gravimetrically were observed.

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* Feigl (*Mikrochemie*, 1923, **1**, 1) defines "sensitivity" as the limit of dilution in which a test can be successfully carried out.

Determination of Lead Tetraethyl in Gasoline. L. J. Catlin and J. E. Starrett. (*Chemist-Analyst*, 1930, 19, No. 5, 5-6.)—A 10 per cent. solution of bromine in carbon tetrachloride is added slowly, with stirring, to 100 c.c. of sample to the extent of 10 c.c. for straight run spirit and 30 c.c. for cracked spirit. After a few seconds, 5 c.c. are added in excess, the lead bromide allowed to settle, filtered off and washed by decantation with 25 c.c. of carbon disulphide. The precipitate is then washed back into the flask with, and dissolved in, 30 c.c. of hot 15 per cent. nitric acid, 5 c.c. of concentrated sulphuric acid added, and the mixture heated till white fumes appear. The lead sulphate is filtered off, washed, dissolved in hot ammonium acetate solution and titrated while hot, with ammonium molybdate solution (8.6 grms. per litre; 1 c.c. = 0.01 grm. Pb). A yellow colour with a fresh 0.5 per cent. solution of tannin, used as outside-indicator, gives the end-point.

J. G.

Arsenic in Writing Materials. G. Kappeller. (*Z. Unters. Lebensm.*, 1930, 60, 213-215.)—Of 14 samples of violet carbon-paper, 5 were found to contain arsenic, 3 being of German origin (0.95 to 3.8 grms. of arsenic per 100 grms., or 16.1 to 59.6 mgrms. per sheet), while 2 were American (0.9 and 3.0 grms. of arsenic per 100 grms., or 19.8 and 45.4 mgrms. per sheet), corresponding with 1.9 to 7.6 and 1.8 to 6.0 grms. of arsenic per 100 grms. of colouring matter, respectively. Two (English) violet typewriter-ribbons contained 0.5 to 1.1 grms. of arsenic per 100 grms., or 5.8 and 15.4 mgrms. per metre, respectively. Violet pencils (4) and aniline ink-powders (2) were free from arsenic. The possibility of contamination through use of oxides of arsenic in the manufacture of (*e.g.*) fuchsin, methyl violet, Prussian blue, etc., is discussed.

J. G.